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Phyllosilicate deformation and its effect on shear zone formation in the upper and middle crust: an experimental study of the deformation mechanisms of mica-quartz assemblage

La déformation des phyllosilicates et son effet sur la formation des zones de cisaillement dans la croûte moyenne et supérieure :
une étude expérimentale des mécanismes de déformation de l'assemblage mica-quartz

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Abstract

In this study, we aimed to investigate the role of mica in strain accommodation and the development of shear zones in the upper to middle crust, examining scales from crystal defects to polycrystalline aggregates. Previous research has explored mica's involvement in shear zones but lacked detailed insights into its deformation processes in natural samples and experimental two-phase biotite-quartz systems. Existing studies primarily focused on single crystals, polyphased systems, or natural samples like schist, gneiss, and pegmatite, with limited attention to biotite in two-phase systems (Etheridge et al., 1973; Bell et al., 1986; Meike, 1989; Kronenberg et al., 1990; Mares and Kronenberg, 1993; Holyoke and Tullis, 2006a, b; Shea and Kronenberg, 1992, 1993; Mariani et al., 2006; Misra et al., 2012; Tullis and Wenk, 1994; Niemeijer and Spiers, 2005; Tokle et al., 2023). To address this gap, we employed a biotite-quartz two-phase system as an analogue for upper and mid-crustal rocks, thoroughly investigating deformation and reaction mechanisms. Our findings emphasize dissolution-precipitation as a prominent weakening process in biotite-rich systems, driven by solution-transfer diffusion creep localized at grain boundaries and facilitated by phyllosilicates. Concurrently, dynamic recrystallization was observed in quartz. Significant intracrystalline plastic deformation was not prevalent in biotite, except for localized kinking, while grain size reduction primarily occurred through dissolution-precipitation rather than basal slip or dynamic recrystallization. Increasing the proportion of mica in quartz aggregates accentuated dissolution-precipitation mechanisms, highlighting quartz's reactivity and substantial contribution to rock weakening. This effect was evident even with low fractions of mica, where quartz maintained its role as the interconnected, load-bearing framework mineral.

Furthermore, the chemical composition of mica influenced interactions at grain boundaries between mica and quartz. The dissolution of mica under stress altered the thickness and composition of the fluid-film at the mica-quartz interface, affecting solution pH, phase solubility, and consequently, dissolution-precipitation dynamics. These insights underscore the complex interplay of mineralogical reactions and deformation mechanisms in biotite-rich systems, shedding light on their role in shear zone evolution within the Earth's crust.

Résumé

Dans cette étude, nous avons investigué le rôle du mica dans l'accommodation des contraintes et le développement des zones de cisaillement dans la croûte supérieure à moyenne, en examinant les échelles allant des défauts cristallins aux agrégats polycristallins. Les recherches antérieures ont exploré l'implication du mica dans les zones de cisaillement, mais ont manqué de détails sur ses processus de déformation dans les échantillons naturels et les systèmes expérimentaux biotite-quartz à deux phases. Les études existantes se sont principalement concentrées sur les monocristaux, les systèmes polyphasés ou les échantillons naturels tels que schiste, gneiss et pegmatite, avec peu d'attention portée à la biotite dans les systèmes à deux phases (Etheridge et al., 1973 ; Bell et al., 1986 ; Meike, 1989 ; Kronenberg et al., 1990 ; Mares et Kronenberg, 1993 ; Holyoke et Tullis, 2006a, b ; Shea et Kronenberg, 1992, 1993 ; Mariani et al., 2006 ; Misra et al., 2012 ; Tullis et Wenk, 1994 ; Niemeijer et Spiers, 2005 ; Tokle et al., 2023).

Pour combler cette lacune, nous avons utilisé un système à deux phases biotite-quartz comme analogue des roches de la croûte supérieure et moyenne, étudiant en détail les mécanismes de déformation et de réaction. Nos résultats mettent en avant la dissolution-précipitation comme un processus d'affaiblissement prédominant dans les systèmes riches en biotite, entraîné par le fluage par transfert de solution localisé aux joints de grain et facilité par les phyllosilicates. Simultanément, de la recristallisation dynamique a été observée dans le quartz. Une déformation plastique intracristalline significative n'était pas courante dans la biotite, à l'exception de plissements localisés, tandis que la réduction de la taille des grains se produisait principalement par dissolution-précipitation plutôt que par glissement basal ou recristallisation dynamique. L'augmentation de la proportion de mica dans les agrégats de quartz a accentué les mécanismes de dissolution-précipitation, mettant en lumière la réactivité du quartz et sa contribution substantielle à l'affaiblissement des roches. Cet effet était visible même avec de faibles fractions de mica, où le quartz maintenait son rôle de minéral interconnecté et accommodant la contrainte. De plus, la composition chimique du mica influençait les interactions aux joints de grains entre le mica et le quartz. La dissolution du mica sous contrainte modifiait l'épaisseur et la composition du film fluide à l'interface mica-quartz, affectant le pH de la solution, la solubilité des

phases et, par conséquent, la dynamique de dissolution-précipitation. Ces observations soulignent l'interaction complexe des réactions minéralogiques et des mécanismes de déformation dans les systèmes riches en biotite, éclairant leur rôle dans l'évolution des zones de cisaillement au sein de la croûte terrestre.

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Résumé étendu

1. Introduction

Dans la croûte terrestre, les mécanismes de localisation des contraintes dans les zones de cisaillement ductiles influencent fortement la dynamique des chaînes de montagnes, la genèse des séismes et la formation des structures lithosphériques. Ces zones de cisaillement subissent régulièrement des transformations minéralogiques, identifiées comme des facteurs majeurs contribuant aux changements rhéologiques facilitant l'accommodation de la déformation dans les roches granitiques, représentatives de la composition de la croûte continentale (Shea & Kronenberg, 1993; Fitz Gerald & Stünitz, 1993; Wintsch et al., 1995; Tullis et al., 1996; Wibberley, 1999; Gueydan et al., 2003; Goncalves et al., 2012; Wehrens et al., 2016; Mansard et al., 2018; Airaghi et al., 2020a; Alaoui et al., 2023).

Le flux de fluides pendant la déformation joue un rôle essentiel dans le développement de ces zones de cisaillement, induisant des transformations minéralogiques telles que la croissance de phyllosilicates comme le mica (Ingles et al., 1999; Goncalves et al., 2012; Hentschel et al., 2019). Ainsi, leur étude peut fournir des informations importantes sur les processus nécessaires pour affaiblir suffisamment la roche afin d'accommoder les déformations de manière visqueuse/plastique. La croissance des phyllosilicates est l'une des réactions minéralogiques stimulées par les fluides observées le plus fréquemment dans les zones de cisaillement, influençant fortement la réponse mécanique des protolithes (Shea & Kronenberg, 1993; Holyoke & Tullis, 2006a, b).

À l'échelle d'affleurements, des études récentes sur divers massifs granitoïdes ont montré qu'une déformation pervasive (haute densité de zones de cisaillement) était observée uniquement dans les régions des granites présentant une forte teneur en phyllosilicates dans l'assemblage minéral. En effet, dans les domaines les plus contraints des zones de cisaillement, des phyllonites se forment, et les variations primaires dans la teneur en mica contrôlent l'intensité globale de la contrainte et la quantité de zones de cisaillement (Wehrens et al., 2017).

Par conséquent, la déformation macroscopique des roches contenant du mica implique un ensemble de processus distincts, de l'échelle des défauts cristallins à celle des agrégats, chacun pouvant contribuer à l'adoucissement et à la localisation des déformations. Ainsi, chaque processus doit être caractérisé pour

comprendre l'effet de la croissance des phyllosilicates dans le développement des zones de cisaillement (Holyoke & Tullis, 2006a, b; Airaghi et al., 2020b; Alaoui et al., 2023).

De nombreuses études sur la formation et l'évolution des zones de cisaillement crustales ont montré qu'il est difficile, dans les échantillons naturels, de séparer la contribution de chaque paramètre physique et chimique à la déformation et à l'affaiblissement des roches. Afin d'étudier la déformation des roches dans un cadre partiellement contrôlé, plusieurs appareils de déformation ont été développés, tels que l'appareil à gaz, le piston-cylindre et l'appareil à multi-enclume. Le principal avantage de l'appareil de Griggs réside dans sa capacité à simuler la déformation visqueuse du quartz sous des conditions de pression et de température élevées, permettant ainsi de comprendre les mécanismes de déformation et les propriétés mécaniques des roches (Griggs, 1967; Green et al., 1970; Byerlee, 1978; Kirby, 1985; Kohlstedt et al., 1995).

Cependant, malgré les avantages de l'approche expérimentale, il est difficile d'extrapoler les résultats aux zones de cisaillement naturelles en raison des différences de temps, de pression, de température et de taux de déformation pour la plasticité. Néanmoins, des similitudes microstructurales entre les échantillons expérimentaux et naturels suggèrent que des mécanismes de déformation similaires pourraient être impliqués (Kronenberg and Tullis, 1984; Linde et al., 1996).

En conclusion, l'étude expérimentale des assemblages quartz-mica permettra de comprendre comment la quantité de mica affecte les mécanismes de déformation, tout en examinant la nature des processus chimiques dans ces scénarios naturels. Ces résultats sont cruciaux pour quantifier l'affaiblissement des roches et comprendre comment l'assemblage de mica gouverne la distribution des zones de cisaillement localisées dans les roches naturelles (Wehrens et al., 2016; Airaghi et al., 2020a).

Ce projet vise à étudier le rôle des micas dans l'accommodation des contraintes et le développement des zones de cisaillement dans la croûte supérieure à moyenne, en analysant les mécanismes de déformation et de réaction à l'échelle microstructurale. Il utilise un système biphasé simple avec mica (biotite) et quartz comme minéraux clés. Les objectifs incluent l'identification des processus d'affaiblissement, la caractérisation des mécanismes de déformation intra-cristalline de la biotite, et l'étude de leur évolution

selon la proportion et la composition du mica. Les chapitres suivants couvrent la synthèse des connaissances actuelles, la méthodologie, l'analyse des propriétés mécaniques, l'exploration des nanostructures et une discussion générale basée sur une comparaison avec des échantillons naturels.

2. La déformation des phyllosilicates et des zones de cisaillement

La déformation des roches polyminérales est un domaine crucial de la géologie, influencé par une complexe interaction de mécanismes rhéologiques et chimiques. Les zones de cisaillement, souvent localisées dans les roches à faibles contraintes, exacerbent la déformation dans les volumes affectés sous une contrainte identique comparée à leur environnement (Fig. 1). Les modèles rhéologiques sont essentiels pour quantifier cette faiblesse, en établissant des relations entre la contrainte différentielle appliquée et le taux de déformation, fournissant ainsi des lois d'écoulement spécifiques pour les matériaux monominéraux. Pour les matériaux polyminéraux, des modèles de mélange ont été proposés, basés sur les lois d'écoulement des phases pures pour prédire le taux de déformation global de l'agrégat.

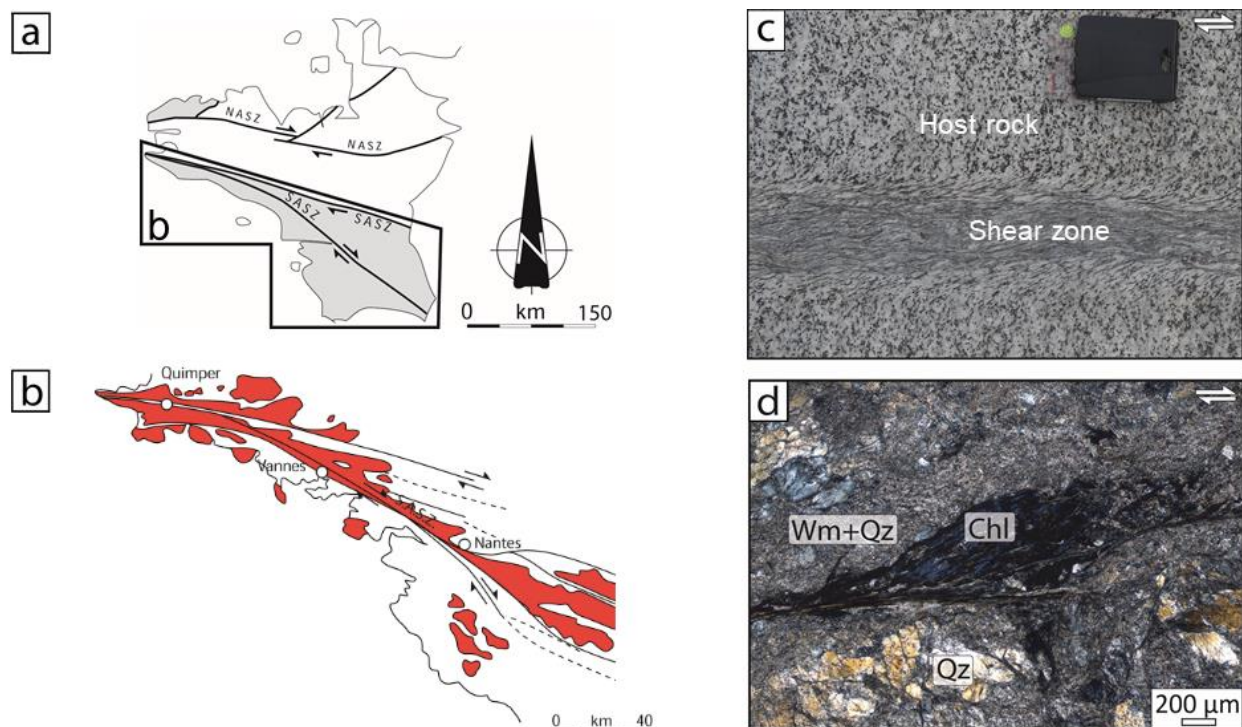


Figure 1 : Zones de cisaillement à différentes échelles. a) Localisation de la zone de cisaillement sud-armoricaine, b) Schéma structural de la zone de cisaillement sud-armoricaine (SASZ) avec les corps granitiques colorés en rouge (Augier et al., 2014 modifié d'après Berthé et al., 1979), c) Zone de cisaillement macroscopique située dans l'affleurement de Neves, d) Zone de cisaillement microscopique localisée dans une biotite chloritisée d'une mylonite du Massif de Bielsa (Alaoui et al., 2023). Chl: chlorite, Qz: quartz, Wm: mica blanc.

Les phyllosilicates, comme le mica, jouent un rôle critique dans la déformation des roches polyminérales (Fig. 1d). Leur présence influence considérablement les mécanismes de déformation, en particulier à travers des processus de solution-pressure et de fluage. Le mica (Fig. 2) agit souvent comme un site de faiblesse dans les agrégats, facilitant la déformation par des mécanismes tels que le glissement de dislocations et les interactions de solution de pression. Ces processus sont essentiels pour comprendre la déformation à long terme des roches et doivent être intégrés aux modèles rhéologiques pour prédire avec précision le comportement des roches polyminérales dans divers environnements géologiques.

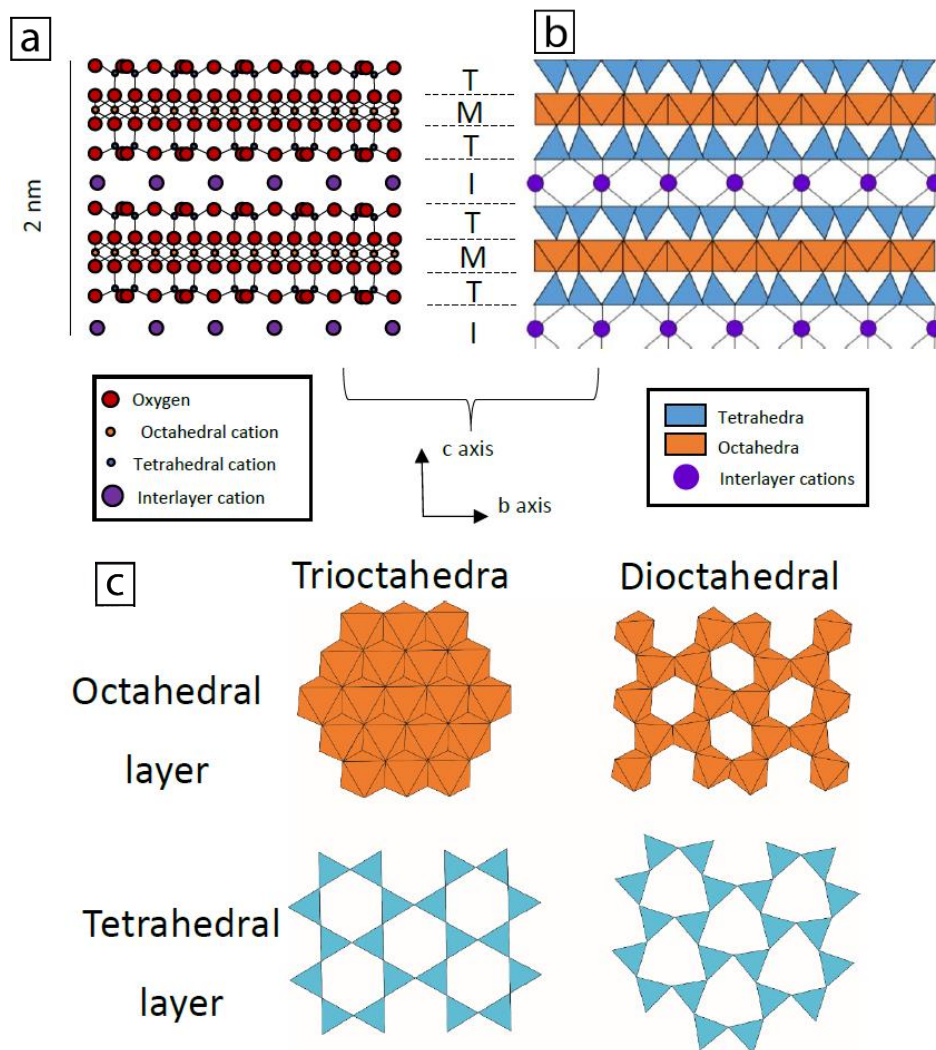


Figure 2 : Illustrations des structures cristallines du mica. a) Modèle boule et bâton d'une structure de mica trioctaédrique vu de la direction a. Les couches tétraédriques (T), octaédriques (M) et interfoliaires (I) sont mises en évidence à droite. b) Modèle en bloc du même réseau cristallin de mica vu également de la direction a, montrant l'arrangement des structures octaédriques et tétraédriques qui forment des couches TMT séparées par des interfoliaires. c) Diagrammes en bloc vus de la direction c des couches octaédriques et tétraédriques pour les micas trioctaédriques et dioctaédriques. Dans un mica dioctaédrique, un espace octaédrique sur trois est vacant, ce qui entraîne une répartition inégale des charges et déforme les formes des couches octaédriques et tétraédriques résultantes (Aslin, 2019).

Les modèles de mélange rhéologique, malgré leur valeur prédictive, présentent des limitations importantes. Ils ne capturent pas pleinement les interactions complexes entre les phyllosilicates et d'autres minéraux, ni les processus de dissolution-précipitation qui peuvent affaiblir considérablement les roches polyminérales sous contrainte. Ces modèles fournissent une estimation de la résistance maximale des agrégats, mais ne tiennent pas compte des effets d'affaiblissement induits par les réactions chimiques, limitant ainsi leur capacité à reproduire fidèlement le comportement des roches dans des conditions géologiques variées.

Malgré ces défis, les modèles rhéologiques restent cruciaux pour interpréter les données de terrain et expérimentales, fournissant un cadre théorique solide pour comprendre la déformation des roches à grande échelle. Cependant, pour répondre aux défis futurs, il est impératif de développer des modèles plus sophistiqués et intégratifs qui prennent en compte la diversité des interactions chimiques et physiques présentes dans les environnements géologiques réels. Cette évolution est essentielle pour améliorer la précision des modèles et pour approfondir notre compréhension des processus de déformation crustale à travers les âges géologiques.

En conclusion, l'étude des mécanismes de déformation dans les roches polyminérales est une composante fondamentale de la géologie moderne. Les modèles rhéologiques sont essentiels pour décrypter ces mécanismes complexes, en intégrant les effets des phyllosilicates et d'autres minéraux sur le comportement mécanique des roches. Leur développement futur devra prendre en compte les interactions chimiques et physiques détaillées présentes dans les systèmes géologiques réels, afin de fournir des outils plus précis pour prédire le comportement des roches sous différentes conditions géologiques et environnementales.

3. La dissolution-précipitation comme mécanisme d'affaiblissement dans les assemblages mica-quartz expérimentalement déformés (Chapitre IV)

L'étude porte sur le comportement mécanique et les microstructures d'agrégats de quartz et mica, variés en proportion de mica (phlogopite). Les résultats mécaniques montrent une variation significative de la résistance différentielle maximale en fonction de la proportion de mica. Les échantillons avec 10% et 20% de phlogopite présentent les résistances les plus élevées (1239 MPa et 943 MPa respectivement),

tandis que ceux avec 30% et 100% de phlogopite montrent les résistances les plus faibles (338 MPa et 432 MPa). Les taux de déformation varient peu malgré des conditions expérimentales identiques, mais les viscosités effectives calculées révèlent une tendance à la diminution de la résistance avec l'augmentation de la proportion de mica. Les microstructures analysées montrent que les échantillons riches en mica présentent des couches interconnectées de mica entourant des grains de quartz anguleux, tandis que les échantillons pauvres en mica montrent des couches interconnectées de quartz avec une matrice fine. La déformation intragranulaire du quartz, étudiée par EBSD (Fig. 3), montre des déformations croissantes avec la diminution de la proportion de mica, marquée par des angles de désorientation importants concentrés autour de l'axe [0001].

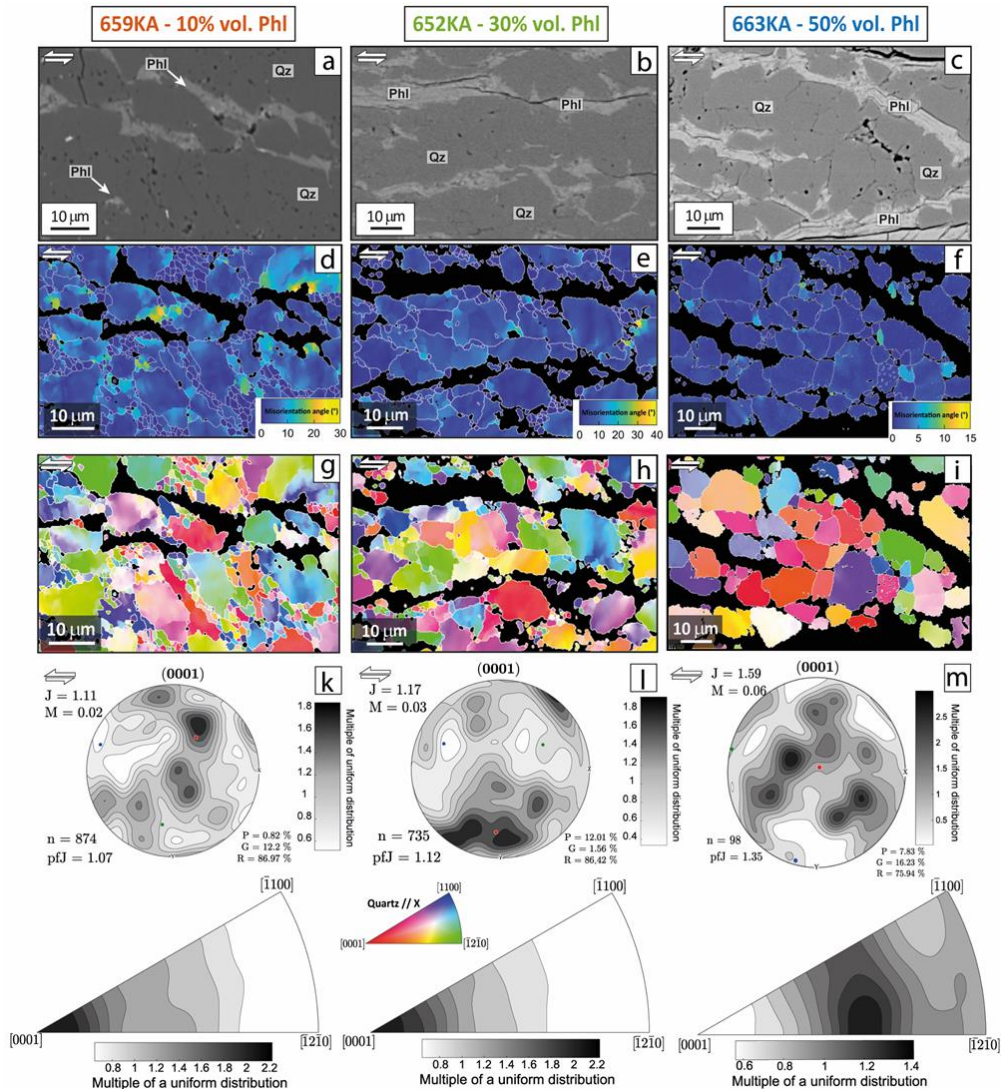


Figure 3 : Images BSE pour la cartographie EBSD de a) échantillons 10Phl, b) 30Phl, et c) 50Phl. Cartes d) à f) de désorientation intergranulaire correspondantes. Cartes g) à i) des orientations du quartz parallèles à X. Figures de pôles j) à l) montrant la CPO du quartz, en projections hémisphériques inférieures (point par grain). Inclus : indices J, M, nombre de grains (n), et distribution des axes de désorientation (2° à 10°). Tous les contours sont en multiples de la distribution uniforme (m.u.d).

La cathodoluminescence révèle deux types de grains de quartz (Qz1 et Qz2), avec Qz2 montrant une luminescence bleue due à une teneur plus élevée en aluminium, indiquant une dissolution-précipitation. L'analyse de la composition minérale confirme une augmentation de l'aluminium dans les grains de quartz recristallisés (Qz2) par rapport aux clastes de quartz hérités (Qz1).

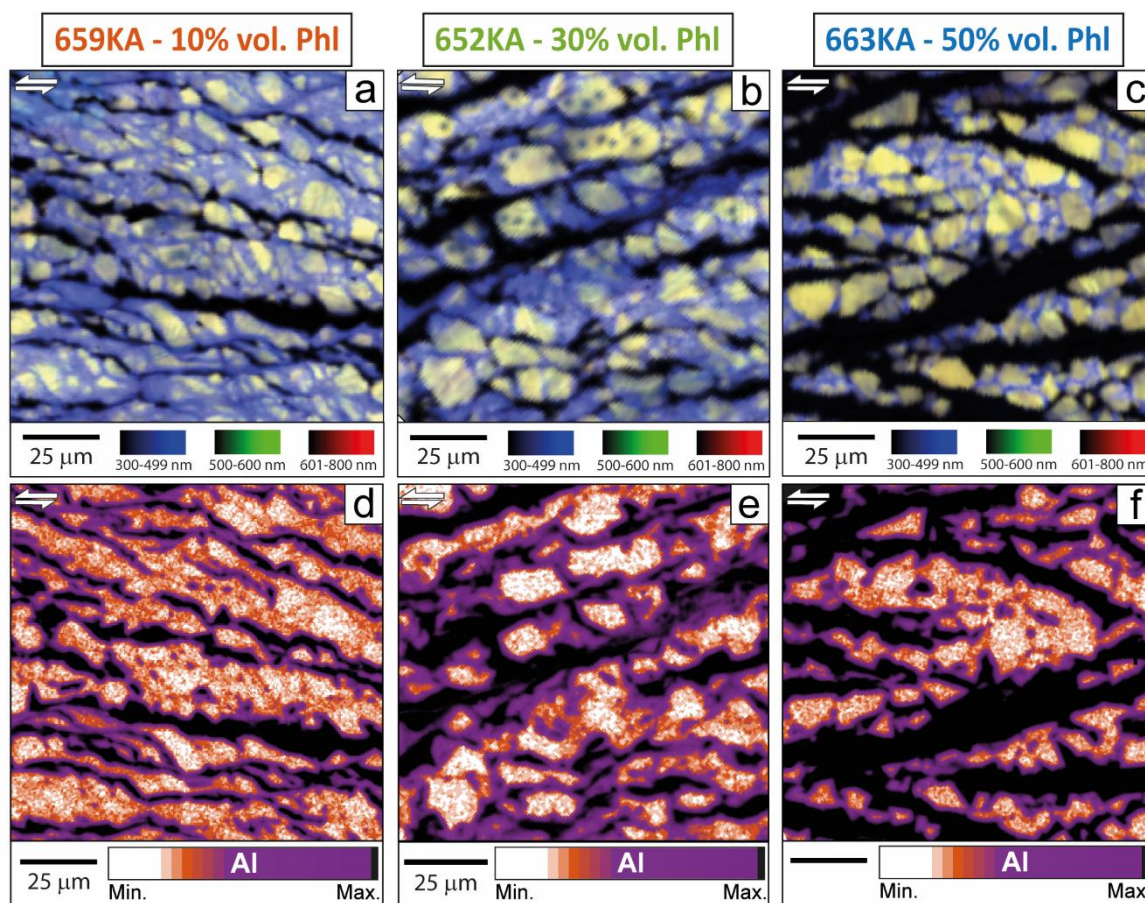


Figure 4: Cartes CL et carte élémentaire Al₂O₃ correspondante pour a), d) échantillon 10Phl, b), e) échantillon 30Phl, c), f) échantillon 50Phl.

Cela met en évidence l'impact du mica sur les mécanismes de déformation du quartz, montrant que la présence de mica favorise la dissolution-précipitation du quartz, affaiblissant ainsi l'assemblage (Fig. 5). Les observations de déformation du mica révèlent un potentiel mécanisme de déformation plastique par glissement basal, avec des grains de mica kinkés et plissés. La dissolution-précipitation est proposée comme le principal mécanisme de réduction de la taille des grains de mica.

En conclusion, l'étude souligne l'importance de la proportion de mica dans le comportement mécanique et la microstructure des agrégats quartz-mica, influençant à la fois la résistance mécanique et les mécanismes de déformation (Fig. 5). Ces résultats sont cruciaux pour comprendre les processus géologiques tels que la déformation crustale et la dynamique des matériaux dans des contextes tectoniques et métamorphiques.

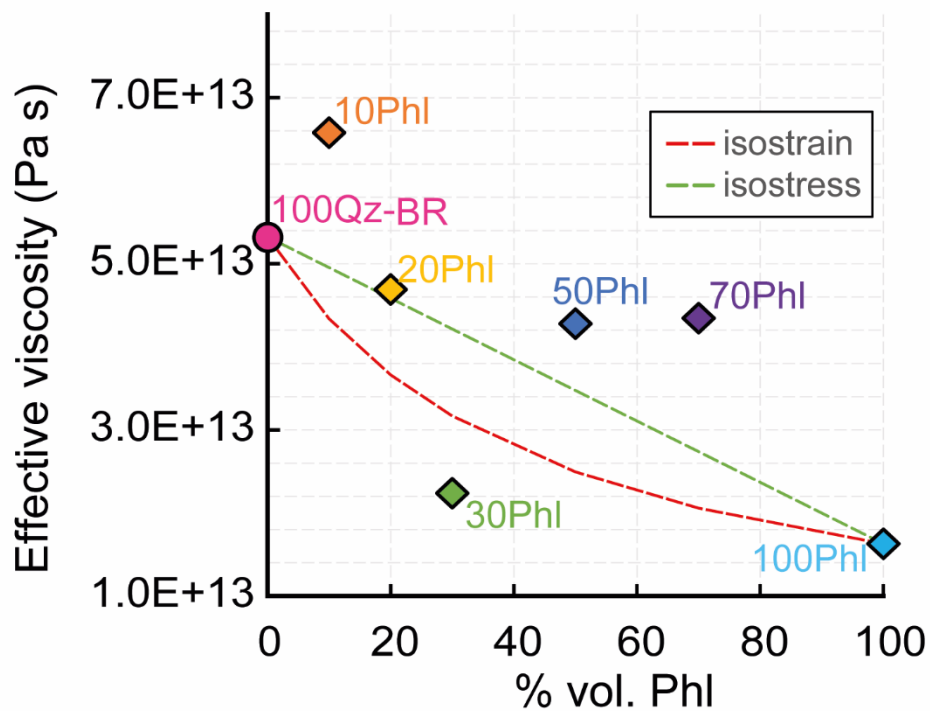


Figure 5 : Évolution de la proportion de viscosité-phlogopite, les lignes en pointillés correspondent aux modèles iso-déformation/iso-contrainte utilisant 100Qz-BR de Richter et al., 2018. La pression de confinement est de 1,0 GPa, la température est de 800°C et le taux de déformation est d'environ 10^{-5} s^{-1} . La taille des grains de quartz est de 10-20 μm dans toutes les expériences sauf dans 100Qz-BR (0-100 μm), et la taille des grains de phlogopite est de 63-125 μm . La teneur en H_2O est de 0,1% en poids dans toutes les expériences sauf dans 100Qz-BR (0,2% en poids).

4. Les nanostructures des assemblages mica-quartz et leur implication dans l'accommodation de la déformation à l'échelle micrométrique (Chapitre V)

L'étude explore les microstructures de déformation dans les agrégats quartz-phlogopite, mettant en lumière des mécanismes complexes à différentes échelles. Les résultats révèlent que les grains de phlogopite parent ($> 5 \mu\text{m}$) montrent principalement des plis, des torsions et des pinces parmi les grains de quartz, formant des apophyses. De nouveaux petits grains de mica ($< 5 \mu\text{m}$) apparaissent dans les interstices des limites de grains de quartz-quartz.

Dans les grains de quartz, les résultats EBSD montrent une différenciation entre les grains parents ($> 8 \mu\text{m}$) et les grains recristallisés ($< 8 \mu\text{m}$). Les grains parents montrent des angles de désorientation intragranulaires élevés ($10\text{-}30^\circ$), tandis que les grains recristallisés présentent des formes polygonales avec peu ou pas de désorientation intragranulaire. Les analyses CL hyperspectrales distinguent deux types de quartz avec différentes émissions lumineuses, suggérant de la dissolution-précipitation.

À l'échelle nanométrique, les grains de quartz parents montrent une densité élevée de défauts, tandis que les grains recristallisés sont plus petits (de $1 \mu\text{m}$ à 100 nm) et souvent associés en agrégats (Fig. 6). Les microstructures révèlent également la présence de couches amorphes de silice aux frontières de grains, résultant probablement de la dissolution-précipitation (Fig. 6).

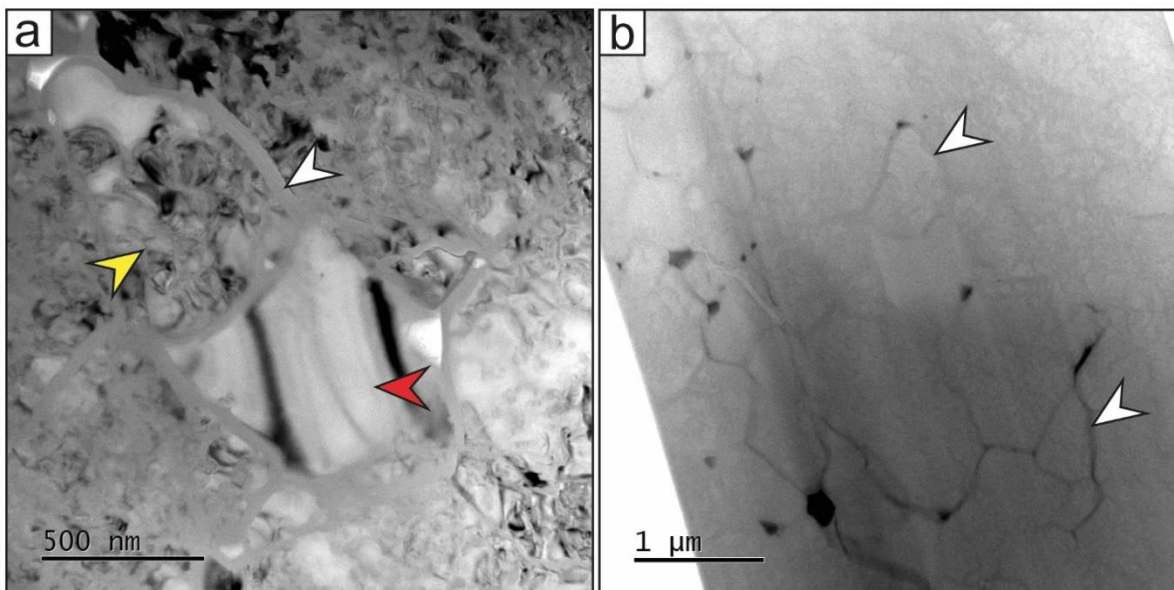


Figure 6 : a) Image en champ clair des grains de quartz recristallisés, avec une flèche blanche indiquant la limite de grain amorphe, une flèche rouge montrant le grain pauvre en défauts et une flèche jaune le grain riche en défauts. b) Image en champ sombre de l'échantillon 10Phl, avec des flèches blanches montrant le matériau amorphe.

Dans les zones riches en mica, les grains de mica présentent des déformations principalement par pliages et torsions, sans preuve de glissement basal (Fig. 7). Les défauts triangulaires observés le long des frontières des cristallites de mica sont associés aux joints de grains à faible angle et suggèrent une déformation accommodée par des mécanismes de type kink band et ripplocations (Fig. 7).

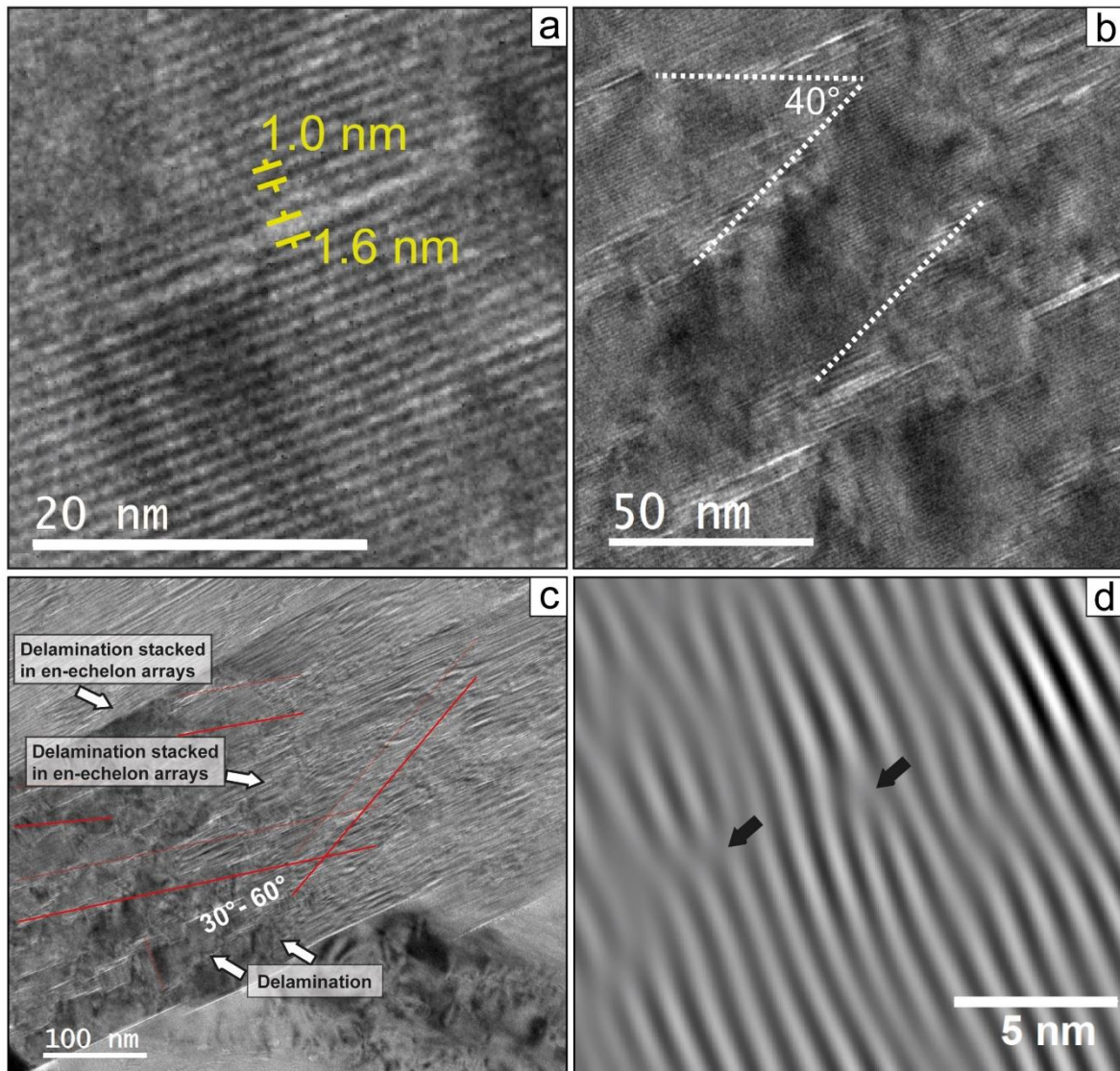


Figure 7 : Micrographies au microscope électronique en transmission en champ clair à haute résolution de la section mince 10Phl traitée par faisceau ionique, montrant les défauts intragranulaires dans la phlogopite. a) Site de dilatation avec un réseau cristallin passant de 1,0 nm à 1,6 nm (= défauts de délamination). b) Arrangement spatial des défauts de délamination (structures de type ripplocation). c) Structures d'expansion en forme de diamant, avec des angles aigus de 30° à 60°. Les lignes lumineuses correspondent aux structures de délamination. Les lignes rouges montrent les principales directions des délaminations alignées et, dans le zoom, la longueur de la structure d'expansion. d) Image filtrée par transformée de Fourier rapide (FFT) d'une image en champ clair à haute résolution avec des dislocations indiquées par des flèches noires montrant la distorsion des plans basaux.

Les analyses de composition révèlent des différences significatives entre les grains de phlogopite de différentes tailles, les petits grains montrant une richesse en fer et une faible teneur en magnésium et en aluminium par rapport aux grains plus grands. La variabilité de la composition chimique dans les grains de mica témoigne du rôle crucial de la dissolution-précipitation dans la réduction de la taille des grains de mica.

En conclusion, cette étude apporte des informations précieuses sur les processus de déformation dans les agrégats quartz-mica, mettant en évidence l'importance des interactions entre dissolution-précipitation, recristallisation dynamique et déformations intragranulaires dans la microstructure des minéraux sous contrainte, avec des implications significatives pour la compréhension des processus géologiques complexes.

5. L'effet de la composition des micas sur la résistance des assemblages mica-quartz (Chapitre VI)

Dans cette étude comparative, les principales observations révèlent des variations significatives de la résistance mécanique et du comportement de déformation en fonction du type de mica, en particulier la phlogopite fluorée (F-Phl) et la biotite (Bt).

Les échantillons contenant de la phlogopite fluorée (F-Phl) montrent systématiquement des valeurs plus élevées de contraintes maximales, variant de 1090 à 1341 MPa, avec une exception pour les échantillons à grains fins (0-63 μm) atteignant 878 MPa (Fig. 8). En revanche, ceux contenant de la biotite (Bt) affichent des contraintes maximales plus faibles, entre 320 et 392 MPa. Cette différence souligne une meilleure tenue structurale des assemblages contenant de la F-Phl (Fig. 8).

Les courbes de déformation révèlent des comportements distincts : les échantillons de Bt présentent un plateau de contrainte stable après un pic initial, tandis que ceux de F-Phl montrent une diminution rapide de la contrainte post-pic, indiquant une transition vers un état de faiblesse accrue (Fig. 8).

Les analyses microstructurales montrent une foliation initiale composée de couches de mica et de quartz dans tous les échantillons (Fig. 9). Cette foliation est altérée dans les échantillons déformés, où le quartz forme des domaines allongés et les grains de mica sont réduits en taille.

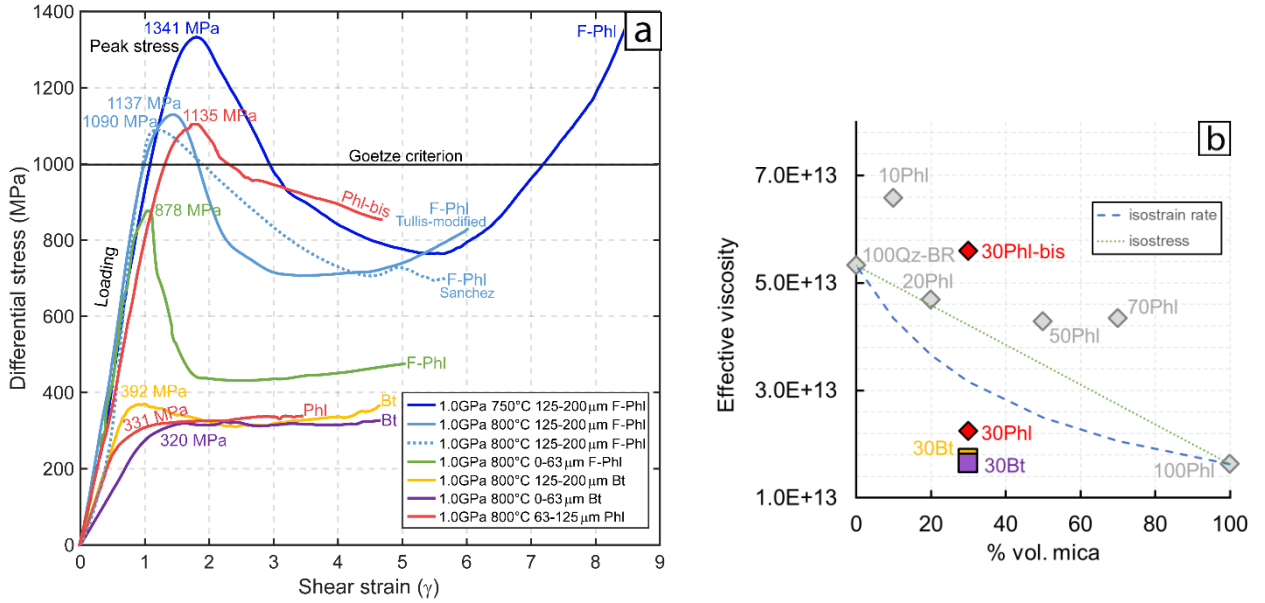


Figure 8 : Données mécaniques de la série d'expériences. a) Évolution contrainte-déformation pour les expériences d'assemblage mica-quartz, la courbe en pointillés correspond à une expérience réalisée sur l'appareil Sanchez Griggs. Toutes les expériences sont à 30% en volume de mica et 70% en volume de quartz. b) Évolution de la proportion de viscosité effective-mica, les lignes en pointillés correspondent aux modèles iso-taux de déformation/iso-contrainte utilisant 100Qz-BR de [Richter et al., 2018](#) pour $n = 3-5$ et calculées pour des contraintes différentielles à $\gamma=2,5$. La pression de confinement est de 1,0 GPa, les températures sont de 750°C et 800°C, et le taux de déformation est d'environ 10^{-5} s^{-1} . La taille des grains de quartz est de 10-20 μm pour toutes les expériences, la taille des grains de mica est indiquée pour chaque expérience sur le graphique. La teneur en H_2O est de 0,1% en poids.

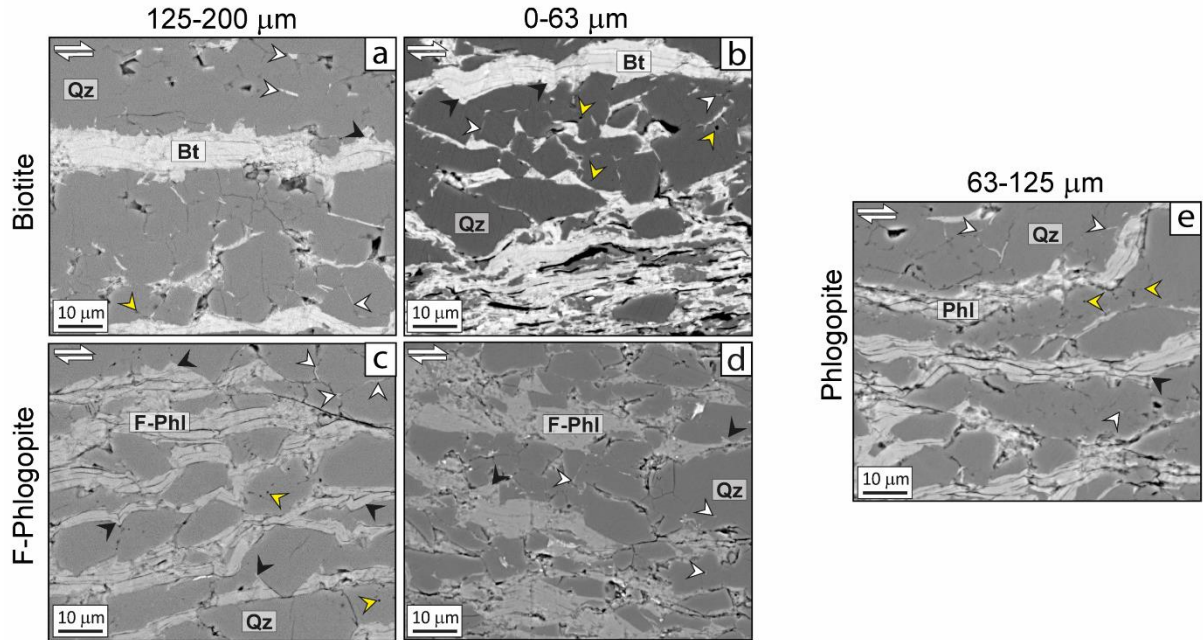


Figure 9 : Observations microstructurales des assemblages mica-quartz déformés sous un microscope électronique à balayage (SEM). Images à électrons rétrodiffusés (BSE) à grand grossissement montrant des grains de mica relictuels allongés, coudés et pincés entourés de clastes relictuels anguleux de quartz, avec quelques micropores pour : a) Gros grain de biotite, b) Petit grain de biotite, c) Gros grain de F-phlogopite, d) Petit grain de F-phlogopite, e) Grain intermédiaire de phlogopite. Flèches blanches : petits grains de mica (<5 μm). Flèches jaunes : micropores. Flèches noires : microstructures pincées et coudées du mica. Les abréviations des minéraux sont issues de [Whitney et Evans \(2010\)](#).

Les échantillons à phlogopite présentent des grains allongés parallèles à la foliation et des grains de quartz réticulés autour d'eux. De plus, de nouveaux grains de mica apparaissent aux interstices entre les grains de quartz, indiquant une restructuration cristalline due aux processus de dissolution-précipitation (Fig. 9).

Les images de cathodoluminescence (CL) révèlent que la luminescence du quartz varie selon le type de mica présent dans les assemblages. Les échantillons contenant de la F-Phl montrent une luminescence plus faible par rapport à ceux de Bt et Phl, malgré une même origine de quartz (Fig. 10).

Les analyses de composition chimique indiquent que les échantillons contenant de la F-Phl et de la Bt présentent des variations mineures dans la composition des micas. La F-Phl est généralement plus riche en Mg que la Bt, influençant ainsi la résistance mécanique et les processus de dissolution-précipitation du quartz.

En conclusion, la composition chimique des micas joue un rôle crucial dans le comportement mécanique et la microstructure des mélanges quartz-mica. La phlogopite fluorée (F-Phl) confère une résistance mécanique supérieure en raison de sa structure cristalline renforcée et de sa composition chimique spécifique.

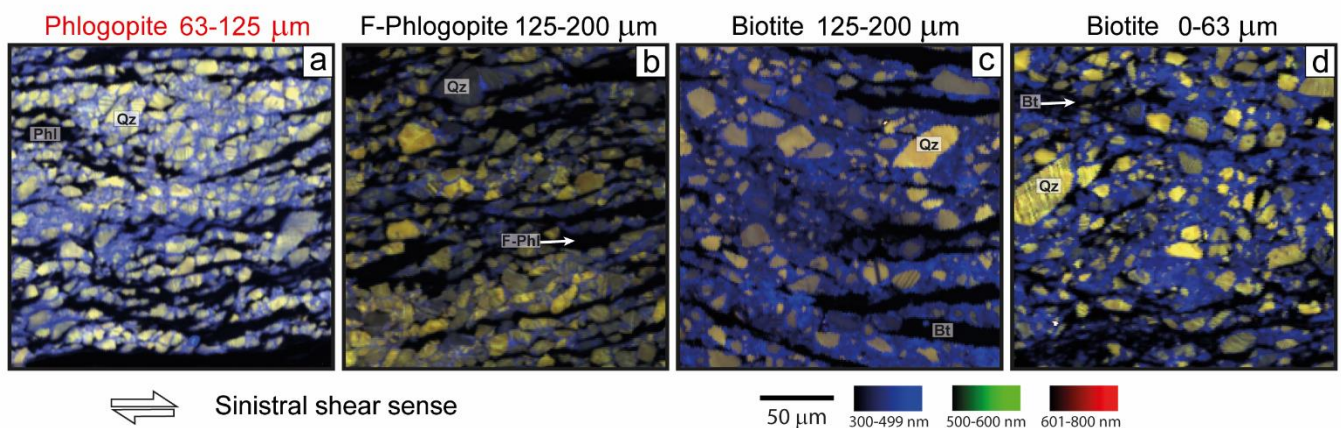


Figure 10 : Images de cathodoluminescence hyperspectrale (CL) sur des échantillons à 70% en volume de quartz (Qz) et 30% en volume de mica. a) Échantillon de phlogopite. b) Échantillon de gros grain de F-phlogopite. c) Échantillon de gros grain de biotite. d) Échantillon de petit grain de biotite.

6. Discussion, conclusion et perspectives (Chapitre VII)

La discussion explore les caractéristiques des zones de cisaillement naturelles dans la croûte supérieure à moyenne à travers une analyse comparative entre échantillons naturels et échantillons expérimentaux. Cette comparaison vise à mieux comprendre les mécanismes de déformation à différentes échelles, des microstructures cristallines aux agrégats polycristallins.

Les microstructures observées dans le mica des zones de cisaillement naturelles incluent des pliages, des kinks et une recristallisation, reflétant des processus de déformation intense. Les études expérimentales révèlent des similitudes marquées, notamment des processus de dissolution-précipitation qui réduisent la taille des grains de mica. Les mécanismes internes, tels que les ripplocations et les expansions de réseau, contribuent également à la déformation.

Le quartz, quant à lui, subit une déformation plastique dans les zones de cisaillement naturelles, avec une recristallisation dynamique par rotation de sous-grains. Les échantillons expérimentaux montrent des microstructures similaires malgré des conditions différentes. Les systèmes de glissement dominants sont discutés en fonction des conditions métamorphiques et de la présence de fluides.

La distribution des phases minérales dans les échantillons dépend non seulement des proportions mais aussi de la déformation et de la connectivité des minéraux. La connectivité du mica influence la force du CPO du quartz, impactant ainsi la résistance mécanique de la roche.

Les interfaces mica-quartz dans les zones de cisaillement montrent des preuves de dissolution-précipitation, affectant la microstructure du quartz et contribuant à la faiblesse mécanique de la roche.

En conclusion, l'étude met en évidence l'importance critique des processus de dissolution-précipitation dans l'affaiblissement des systèmes riches en mica. Les perspectives futures incluent la nécessité d'expériences supplémentaires pour valider les observations et d'analyses approfondies des microstructures à différentes échelles. Cette recherche approfondie améliorera notre compréhension des mécanismes de déformation dans la croûte terrestre et leur rôle dans la tectonique des plaques et la géodynamique.

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I.1. Scientific context and motivation

In the Earth crust, the mechanisms of strain localization in ductile shear zones strongly control the dynamics of mountain belts, the nucleation of earthquakes and the formation of lithospheric structures. Shear zones regularly exhibit mineralogical transformations, identified as a major factor contributing to rheological changes facilitating strain accommodation in granitic rocks, representative of the composition of the continental crust.

Fluid flow during deformation plays a pivotal role in the development of these shear zones, inducing mineralogical transformations such as the growth of phyllosilicates, as mica ([Ingles et al., 1999](#); [Goncalves et al., 2012](#); [Hentschel et al., 2019](#)). Therefore, their study may yield important information upon the processes necessary to weaken the rock sufficiently to accommodate strain in a viscous/plastic way. Phyllosilicates growth is one of the most common fluid-driven mineralogical reaction observed in shear zones and strongly influences the mechanical response of protoliths ([Shea & Kronenberg, 1993](#); [Fitz Gerald & Stünitz 1993](#); [Wintsch et al., 1995](#); [Tullis et al., 1996](#); [Wibberley, 1999](#); [Gueydan et al. 2003](#); [Goncalves et al., 2012](#); [Wehrens et al., 2016](#); [Mansard et al., 2018](#); [Airaghi et al., 2020a](#); [Alaoui et al., 2023](#)). The anisotropic structure of phyllosilicates makes them potentially weak when sheared parallel to their layers ([Kronenberg et al., 1990](#); [Ingles et al., 1999](#); [Wibberley & McCaig, 2000](#); [Gueydan et al., 2003](#); [Marsh et al., 2009](#)). Furthermore, the deformation of phyllosilicates is systematically associated with grain size reduction ([ten Grotenhuis et al., 2003](#)) playing a major role in mechanical softening by promoting grain-size sensitive creep ([Boullier & Gueguen, 1975](#); [Stünitz and Fitz Gerald 1993](#); [Raimbourg et al., 2008](#)). Mica in such cases ([Holyoke & Tullis, 2006a, b](#)) has a pronounced effect on the deformation and recrystallization of stronger phases such as quartz ([Tullis & Wenk, 1994](#); [Hunter et al., 2016](#)). The interconnection of weak phyllosilicates usually controls the degree of strain localization, especially the degree of mica grain connectivity ([Hunter et al., 2016](#)).

At an outcrop scale, recent studies on various granitoid massifs have shown that a pervasive deformation (high density of shear zones) was observed only in regions of the granites with a high content of phyllosilicates in the mineral assemblage. Indeed, in the most strained domains of the shear zones phyllonites are formed ([Hippertt, 1994](#)), and the primary variations in the mica content control the background strain intensity and the amount of shear zones ([Wehrens et al., 2017](#)). Furthermore, phyllosilicate growth reactions, which is more commonly found in the upper crust early in the shortening history and occurs not only in ultramylonites, ultracataclasites, and phyllonites but also more generally, cause a considerable weakening of the rock ([Bellahsen et al., 2019](#)). Pre-kinematic alteration, resulting in phyllosilicate growth reactions and phase mixing before shearing, is associated with the formation of mylonitic shear zones. ([Airaghi et al., 2020b](#); [Alaoui et al, 2023](#)).

Thus, the macroscopic deformation of rocks containing micas involves a set of distinct processes, from the scale of the crystal defect to the aggregate, each of which can contribute to softening and localization of the deformation. Therefore, each has to be characterized in order to understand the effect of phyllosilicate growth in the development of shear zones. However, many fundamental questions remain concerning the deformation processes that operate in mica and in mica-bearing rocks as: (i) which dominant processes is associated to rock weakening? (ii) How does mica participate to rock weakening? At present, relatively little is known about the mechanical strength of mica and about deformation mechanisms in mica-bearing assemblages. As most deformation experiments on mica and mica-bearing rocks have been performed in the friction regime ([Moore et al., 1989](#); [den Hartog et al., 2013](#)), the viscous or plastic realm, that defines the behavior of the mid-lower crust, remains relatively unexplored. If some experimental studies exist for white mica-bearing mineral assemblages ([Shea and Kronenberg, 1993](#); [Holyoke and Tullis, 2006](#); [Tokle et al., 2023](#)), this is particularly true for biotite-bearing assemblages. Therefore, the systematic study of the mechanical and reactional behavior of biotite-rich systems may provide evidence upon the impact the role of mica in strain accommodation of crustal rocks and in the development of upper and mid crustal shear zones.

I.2. Why an experimental deformation approach using the Griggs apparatus?

The numerous studies dealing with nucleation, propagation and evolution of crustal shear zones have demonstrate that, in natural samples, it is difficult to separate the contribution of each physical and chemical parameter to rock deformation and weakening.

The parameters in question are temperature, pressure, fluid content, strain rate, grain size and phase proportions, experimentally we can control pressure, temperature, bulk composition, strain rate, water-added content however we do not have a precise control over fluid throughout the experiment. It is important to note that inside the capsule, there is a temperature gradient but, in our case (shear), the sample thickness is so little (~1 mm) that this effect is very limited ([Baïssset, 2023](#)).

Furthermore, deformation durations vary largely in the geological scale (milliseconds to millions of years), and accessing the samples directly after deformation is rarely feasible. In order to investigate the impact of various parameters on rock deformation in a partially controlled setting, several deformation apparatuses have been developed such as gas apparatus, piston cylinder apparatus, and multi-anvil apparatus. Despite quartz typically exhibiting viscous behavior in natural rocks under low-grade conditions, experimental deformation at high pressures and temperatures is necessary to induce crystal plasticity. Hence, Griggs apparatuses, pioneered in the 1960s ([Griggs, 1967](#); [Green et al., 1970](#)), are employed to investigate viscous deformation in quartz. Indeed, to understand deformation mechanisms and explore mechanical properties of rocks, several experimental deformation studies have been performed ([Byerlee, 1978](#); [Kirby, 1985](#); [Kohlstedt et al., 1995](#)). Which constrained a link between material strength and its deformation, highlighting two types of rheological behavior controlling rock deformation, brittle and viscous behavior. Nevertheless, by simplifying rheology to a single-phase model, one fails to consider that lithospheric layers predominantly consist of polyphase aggregates ([Handy, 1990](#); [Handy et al., 1999](#)), thus overlooking potential interactions within these multiphase aggregates. The rheological properties of two-phase aggregates have been examined in a large amount of studies ([Handy, 1990](#); [Tullis and Wenk, 1994](#); [Dimanov and Dresen, 2005](#); [Takeda and Griera, 2006](#); [Dimanov et al., 2007](#)). It is widely acknowledged that the mechanical characteristics of polyphase

aggregates can be significantly influenced by various factors, including phase proportions, their contrasting rheological properties, grain size, spatial distribution, and geometric configuration. This suggests that our current understanding of the lithosphere could undergo substantial revision if we account for the intricate interactions among mineral phases.

I.3. A challenging extrapolation to natural samples

Even if an experimental approach is a proper alternative to study shear zones in a more restricted and controlled setting, it is not straightforward to extrapolate results to natural shear zones. Certainly, the main difference being the time scale and pressure, temperature, strain rate conditions for plasticity. Concerning pressure and temperature, it is different with nature as we can always increase them more in comparison to the structural level in natural rocks that we want to simulate, to promote plasticity and reactions. However, it is not different with nature in the sense that the ranges used retain a geological meaning, which is not at all the case for strain rate (excluding co-seismic deformation). Another difference compared to natural rocks is that the volumes we deform are necessarily very small, which forces us to have a small starting grain size too.

Generally, conditions observed with experiments at $\sim 600\text{--}1200^\circ\text{C}$ and strain rate of 10^{-4} to 10^{-7} s^{-1} while in nature $\sim 300\text{--}700^\circ\text{C}$ 10^{-12} to 10^{-15} s^{-1} . Yet, similar orders of strain rate in nature as in experiments indicating plastic regime exist ([Linde et al., 1996](#)).

Therefore, it is fairly accepted that similar microstructures between nature and experiments can be assumed to have similar deformation mechanisms. It is imperative to assess external variables such as the pressure-dependent nature of water fugacity as stress levels are greatly influenced by the accuracy and precision of the equipment used ([Kronenberg and Tullis, 1984](#)). These extrapolations are contingent upon identifying the rate-limiting step of the prevailing deformation mechanism, however, such extrapolations are restricted to a singular mechanism, which is rarely encountered in deformed rocks. Instead, multiple mechanisms often interact concurrently.

For instance, 10% vol. of hydrated phases are shown to be adequate at the mineral aggregate scale in natural shear zones of the lower crust to encourage the dissolution-precipitation of anhydrous phases (Graziani et al., 2020). In granitic shear zones of the upper crust, variations of 20% in mica content (comprising over 25% of the assemblage) are noted between regions in which quartz dynamically recrystallizes and regions in which it displays widespread dissolution-precipitation with no intragrain plastic deformation (Alaoui et al., 2023). Through a comparison of the microstructures, deformation, and reaction mechanisms in both quartz and mica, the results of simplified quartz-mica assemblages will and can be directly applied to quartz-mica-rich layers of naturally deformed crustal rocks. Our experimental findings will make it possible to determine how much the quantity of mica in the mineral assemblage affects deformation mechanisms, and comprehend the nature of chemical processes in these natural scenarios. On a larger scale, quantifying the weakening of rocks resulting from chemical interactions will be essential to comprehending the circumstances and processes by which the assemblage of mica governs the distribution of localized shear zones in natural rocks (Wehrens et al., 2016; Airaghi et al., 2020a). Alternatively, mica may produce stronger rocks or mechanical hardening during mica growth (Wibberly, 1999). Analogous two-phase assemblages consisting of hydrated and anhydrous mineral phases could serve as a starting point for comparing results with findings reported in other polymineralic rocks.

I.4. Manuscript outline

The aim of this project is to investigate the role of micas in strain accommodation and development of shear zones within the upper to middle crust, from the scale of crystal defects to the polycrystalline aggregate. The study will employ a simple two-phase system, so that deformation and reaction mechanisms can be studied in detail. The most representative and important minerals for such a system are mica (i.e. biotite) and quartz. In details, the aims of the PhD are to:

- identify the weakening processes in biotite-rich systems;
- characterize the different intra-crystalline deformation mechanisms in biotite;

- identify how these processes evolve when changing the proportion and the composition of mica in the mineral assemblage.

The project combines detailed microstructural and petrological study of the mica-quartz samples experimentally deformed under different temperature, pressure and strain rate conditions in the Griggs apparatus. The other chapters of this manuscript are composed as follow:

- **Chapter II** presents the state of the art in our knowledge on phyllosilicates deformation and shear zones development explaining why we have an interest on mica and mica-bearing rocks in the crust. In addition, the evolution in the knowledge on experimental and natural samples are described. A synthesis of mica effects on quartz, through the review of existing literature, including all types of deformation processes from solid-state dynamic to dissolution-precipitation is provided as well as an overview on rheological models of polymineralic and monomineralic aggregates at the end of this chapter.
- **Chapter III** describes materials and methods, regarding the approach and aims, sample preparation, experimental and analytical procedures that have been used in this study.
- **Chapter IV** examines the mechanical properties of mica-bearing assemblages in order to understand the mechanical properties of the mica-bearing shear zones present in deformed upper to mid crustal rocks, and therefore the role of mica in rocks strength and underlying processes. For that understanding, the mechanical strength of quartz and phlogopite mixtures and underlying deformation processes is investigated for variable proportions of the two phases.
- **Chapter V** explores the effect of nanostructures on strain accommodation at micrometric scale in experimentally deformed phlogopite-quartz assemblages from chapter IV. The purpose is to better understand the different deformation processes controlling rheology and microstructures produced at grain boundaries.
- **Chapter VI** investigates the role of composition (using different biotite) on the formation of shear zones, mechanical behavior and strain accommodation. It is presented as a comparative

study on experimentally deformed mica-quartz assemblages with different type of biotite-rich systems.

- **Chapter VII** presents a general discussion confronting natural shear zone samples with our experimental samples on the basis of 36 papers selected for comparison, a general conclusion and perspectives of following research work.

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II.1. Shear zones formation, strain localization and weakening processes in the continental crust

On Earth, the lithosphere consists of two layers (1) a crust (continental or oceanic) and (2) the upper mantle, and its deformation is localized at plate boundaries. While the upper crust is commonly associated with brittle deformation characterized by faults, the middle-lower crust (beyond the brittle-ductile transition) is dominated by plastic or viscous deformation and is where shear zones mainly develop. Shear zones are zones of strain localization that can accommodate tens to hundreds of kilometers of relative displacement. In several orogenic systems (Variscan, Caledonian, Himalayan) crustal scale shear zones are formed both in compressive and strike-slip tectonic settings (Jégouzo, 1980; Fauconnier, et al., 2014; Cottle et al., 2015). As the continental crust is mainly composed of granitoid rocks, various studies focused on mechanisms of deformation of granites to investigate the geometry, and microstructures of crustal shear zones as well as their rheology (Stünitz and Fitz Gerald, 1993; Ingles et al., 1999; Oliot et al., 2014; Wehrens et al., 2017). Nevertheless, significant uncertainties persist regarding the processes underlying rock weakening and the localization of strain in the continental crust.

Crustal shear zones are often observed as localized tabular-shaped zones of high strain (Ramsay and Graham, 1970) that, from the scale of the mineral aggregate to the lithospheric scale, develop in an anastomosed network and separating less deformed units. This strain gradient is often marked by the rotation of markers or the development of a new fabric (Fig. 1; Fossen and Cavalcante, 2017). A shear zone is indeed distinguished by the presence of a foliation induced by strain, exhibiting a sigmoidal-shaped geometry that reflects a symmetrical decrease in strain from the center to the boundary of the shear zone (Ramsay and Graham, 1970; Choukroune and Gapais, 1983; Marquer, 1990; Fousseis et al., 2006; Ceccato et al., 2022). Protomylonites, mylonites, and ultramylonites form in shear zones as strain increases (Fig. 1; Tsurumi et al., 2003; Baratoux et al., 2005; Goncalves et al., 2012; Linckens et al., 2015; Farkašovský et al., 2016; Mansard et al., 2018; Airaghi et al., 2020b; Ceccato et al., 2022; Alaoui et al., 2023).

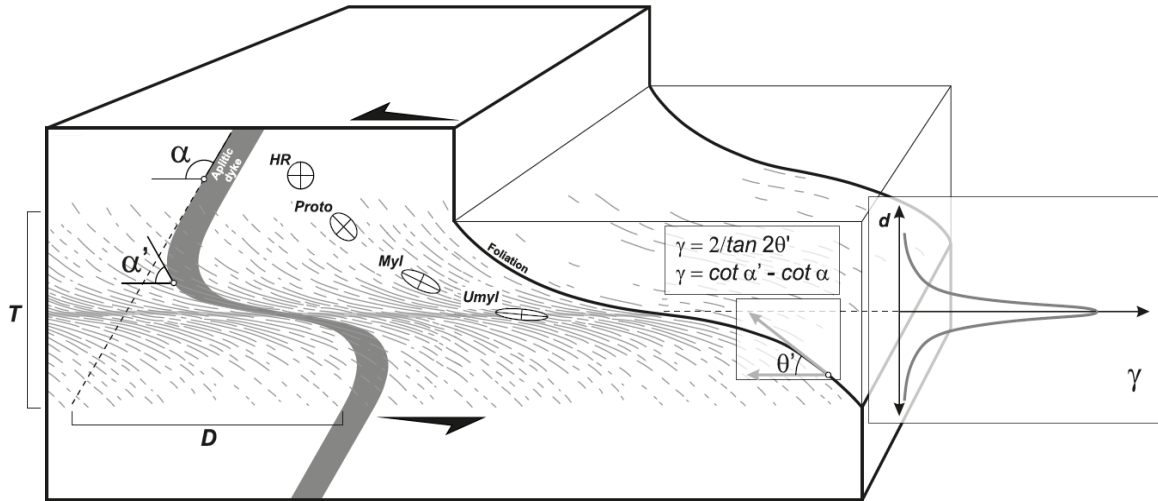


Figure 1 : Schematic representation of the geometrical model of a small-scale shear zone, showing the transition, developed along the strain gradient identified by the sigmoidal foliation (marked by grey dashed curves), from the undeformed granitoid host rock (HR), to protomylonite (Proto), mylonite (Myl) and ultramylonite (Umyl). The model presents the geometrical parameters (α , α' , θ') and equations adopted to calculate shear strain γ . The angle θ' represents the acute angle between the foliation and the shear plane – also corresponding to the angle between the shear plane and the orientation of the principal extensional axis of the strain ellipsoid (Ramsay and Graham, 1970; Ramsay, 1980). The angles α and α' represent the angle between the shear plane and the orientation of a passive strain marker (an aplitic dyke in dark grey) calculated as the angular distance between the shear plane and the tangent at the measuring points (white dots). The d- γ diagram on the right-hand side schematically represents the variation of the shear strain γ (reported on the horizontal axis) as a function of the distance d from the shear zone centre (reported on the vertical axis to orient the diagram in the same way as the shear zone in the figure). Redrawn by Ceccato et al. (2022) and modified after Ramsay (1980). T: thickness, d: distance from the shear zone center.

In the continental crust, shear zones operate at all scale from the mineral to the lithosphere (Fig. 2) and are largely represented by granitic rocks composed essentially of feldspar and quartz. However, phyllosilicates as micas, which are less abundant than quartz and feldspar, are systematically associated with shear zones (Goncalves et al., 2012; Wehrens et al., 2016; Airaghi et al., 2020a; Alaoui et al., 2023). In the upper and middle crust shear zone formation occurs in greenschist-facies conditions, where mainly white mica is stable (along with chlorite) and biotite may appear as a mineral phase inherited from the protolith. Conversely, in the middle crust, shear zone formation occurs in amphibolite-facies conditions, where biotite can recrystallize along with white mica during shearing (Goncalves et al., 2012). Since the major components of the continental crust are quartz and feldspar, a large amount of work focused on the mechanisms of deformation of these mineral phases and how they lead to rock weakening and strain localization. The overall strength of the rock is largely influenced by the viscous behavior of quartz and feldspar, which varies depending on water content and stress levels (Stünitz and

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Tullis, 2001; Stipp et al., 2002; Menegon et al., 2008; Kilian et al., 2011; Palazzin et al., 2018; Ceccato et al., 2022). Yet, rocks in the continental crust are polymineralic. Within a shear zones it is common to observe monomineralic aggregate layers alternating with (Fig. 3; Berthé et al., 1979; Dell'Angelo and Tullis, 1996; Holyoke and Tullis, 2006b), polyphase aggregates (Fliervoet et al., 1997; Keller et al., 2004; Marti et al., 2018; Mansard et al., 2020; Lee et al. 2021).

However, the mechanisms of rock weakening in polymineralic rocks are still far to be established (Stünitz and Fitz Gerald, 1993; Fliervoet et al., 1997; Fossen and Cavalcante, 2017; Ceccato et al., 2022). Strain softening is necessary for localizing the deformation, implying that the deformation can advance with a diminishing stress level while maintaining a constant strain rate (Handy, 1989; Means, 1995). Various textural and mineralogical alterations are associated with this shift in deformation mechanisms from high-stress to low-stress.

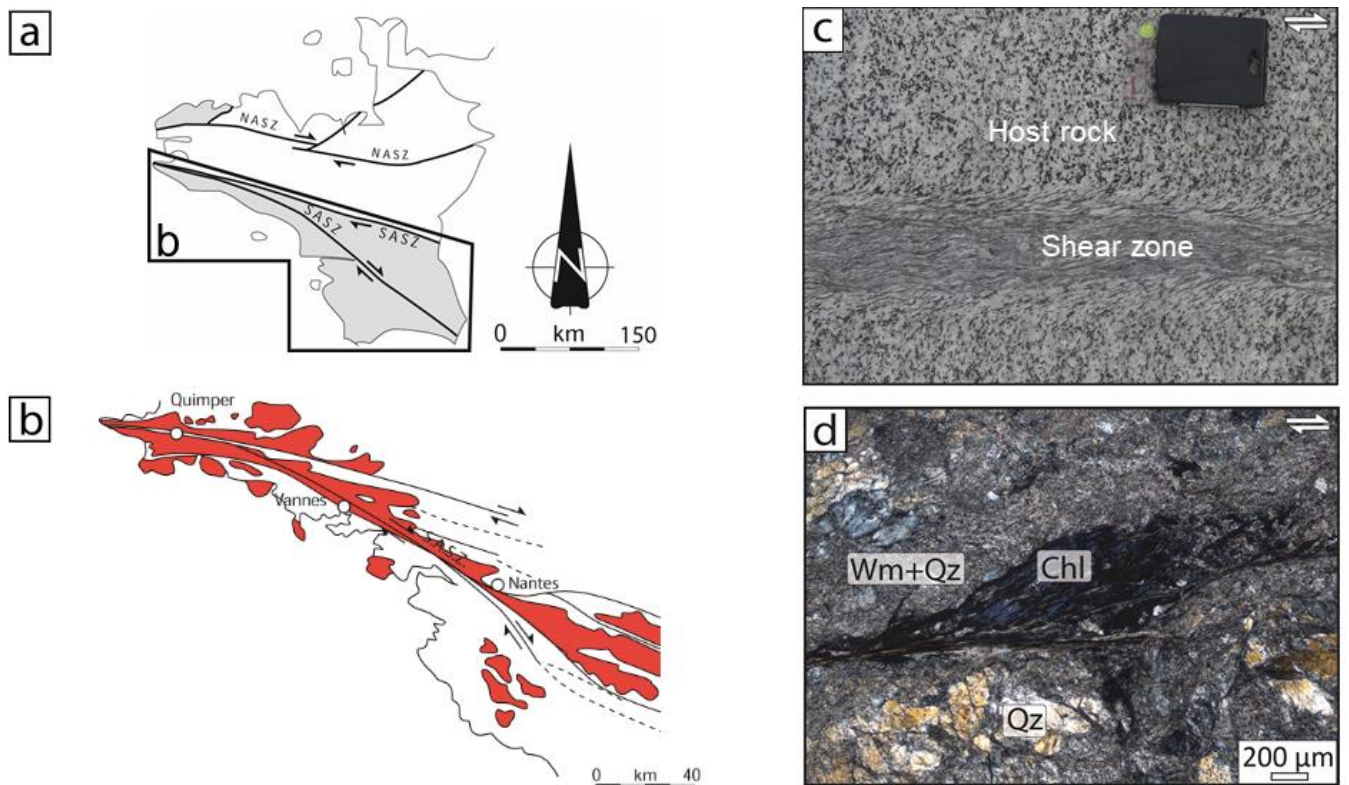


Figure 2: Shear zones at different scales. a) Location of the south Armorican shear zone, b) Structural sketch of the south Armorican shear zone (SASZ) with granitic bodies colored in red (Augier et al., 2014 modified from Berthé et al., 1979), c) Macroscopic shear zone located in the Neves outcrop, d) Microscopic shear zone localized in a chloritized biotite from a mylonite of the Bielsa Massif (Alaoui et al., 2023). Chl: chlorite, Qz: quartz, Wm: white mica.

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One mechanism for shear zone development and widening is the crystallization of new minerals or grains produced by metamorphic or metasomatic reactions, at the cost of load-bearing framework deforming by crystal plasticity (Handy, 1990, 1994; Goncalves et al., 2012; Oliot et al., 2014). In the ductile crust, minerals deform plastically however in the last decades a growing number of studies highlighted the pivotal role of metamorphic and metasomatic reactions in strain accommodation by the means of grain size reduction, phase mixing and substitution of mechanically strong phases by weak phases. Indeed, metamorphic reactions promote grains size reduction and phase mixing, parameters that have been demonstrated to favor strain accommodation (Stünitz and Tullis, 2001; Gueydan et al., 2003; Holyoke and Tullis, 2006). Grain size reduction might enhance a transition from grain size insensitive to grain size sensitive deformation mechanism resulting in weakening (Rutter and Brodie, 1988; Kruse and Stünitz, 1999; Newman et al., 1999). Another major mechanism of strain accommodation in the ductile crust is the plastic deformation of minerals that may also lead to the grain size reduction due to dynamic recrystallization.

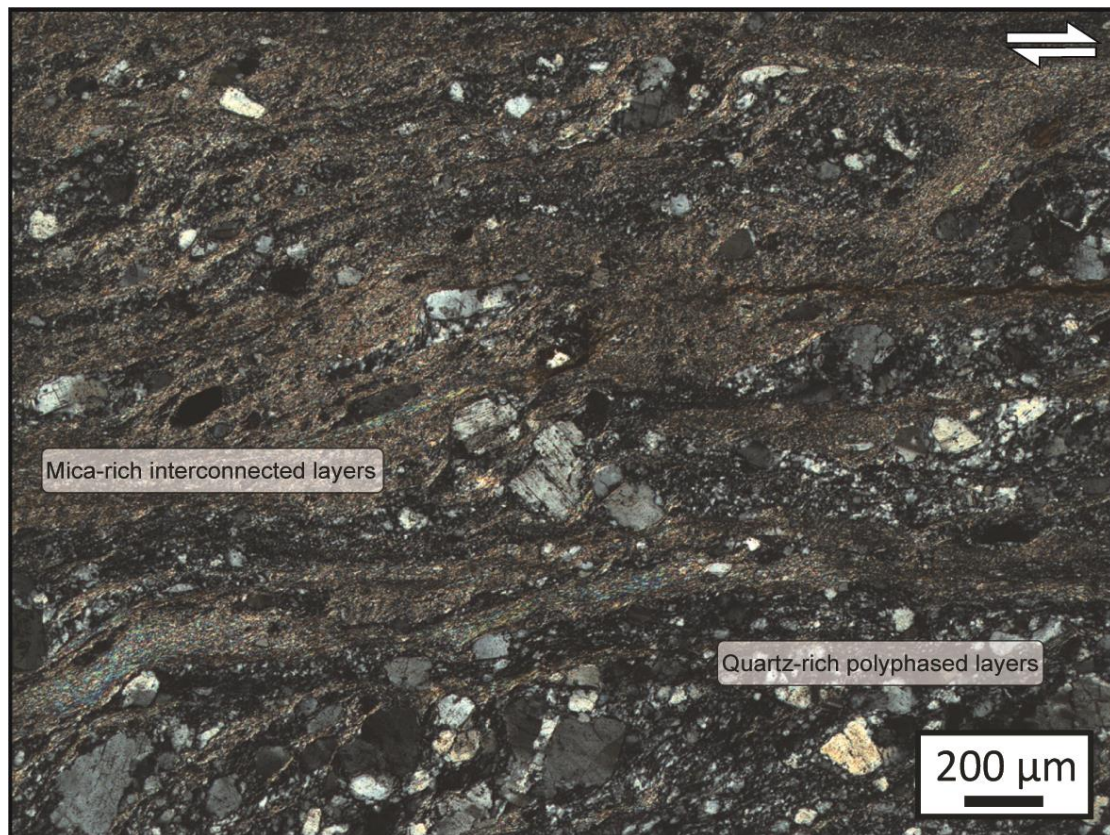


Figure 3 : Mylonite from the south Armorican shear zone located in Questembert.

II.1. Shear zone formation, strain localization and weakening processes in the continental crust

This mechanism happens when deformation conditions take place in the grain-size insensitive dislocation creep, during plastic flow (Etheridge and Wilkie, 1979; Behrmann, 1985; Hirth and Tullis, 1992; De Bresser et al., 2001; Passchier and Trouw, 2005). Processes related to dislocation creep are manifested as deformation lamellae, deformation bands, undulose extinction, the development of recovery-related structures like subgrain boundaries, and dynamic recrystallization in general (Passchier and Trouw, 2005). Subgrain boundary is produced by dislocation arranging themselves in a plane separating to domains of a crystal. Dynamic recrystallization is defined as deformation-induced reorganization of material with a change in shape, grain size and orientation in the same mineral, with or without chemical change (Poirier and Guillopé, 1979; Urai et al, 1986; Hirth and Tullis, 1992).

As grain size reduction is promoted by metamorphic reactions, the nucleation of new grains with different composition is another main mechanism, and it can influence the viscosity (Kruse and Stünitz, 1999a; Kenkmann and Dresen, 2002; Précigout and Stünitz, 2016). Indeed, the dislocation density of a sample can be reduced by strain-free nucleated grains, and grain insensitive dislocation creep could shift to grain sensitive diffusion creep and grain boundary sliding (Poirier, 1985; Behrmann and Mainprice, 1987; Drury and Urai, 1990; Fliervoet et al., 1997; Platt, 2015). During dominant diffusion creep, cavitation creep can occur when local grain sliding cannot be accommodated fast enough by crystal-plastic or diffusional mass transport processes (Meyersm and Ashworth, 1982; Benson et al., 2001; Fusseis et al., 2009; Précigout and Stünitz, 2016; Gilgannon et al., 2017; Précigout et al., 2017; 2019). Both metamorphic/metasomatic reactions and grain size reduction often lead to phase mixing, the latter may cause rock weakening by hindering grain growth due to pinning of grain boundaries (Fig. 4), this process is called Zener pinning (Etheridge and Wilkie, 1979; Olgaard, 1990; Evans et al., 2001; Herwegh et al., 2011; Gilgannon et al., 2017).

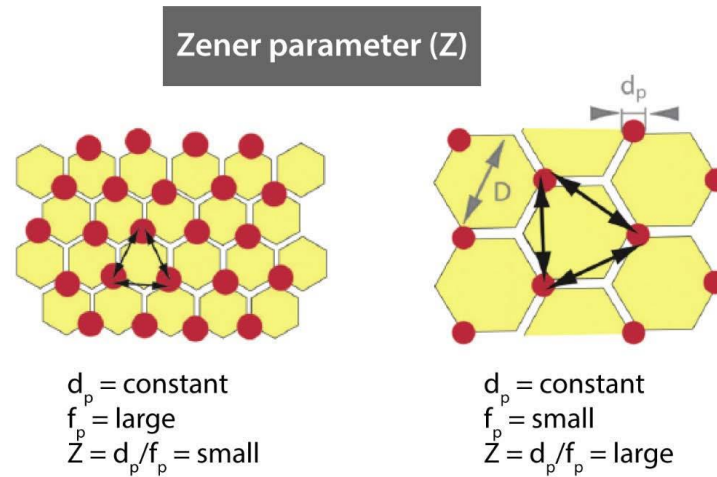


Figure 4 : Effect of the Zener parameter (Z) (from [Herwegh et al., 2011](#)). d_p = size of the second phase; f_p = volume fraction of the second phase.

In polymineralic rocks as the ones of the continental crust, the formation of interconnected weak layers, as the ones made of phyllosilicates ([Berthé et al., 1979](#); [Tullis and Wenk, 1994](#); [Holyoke and Tullis, 2006b](#)) or of small-grain aggregates ([Fitz Gerald and Stünitz, 1993](#); [Oliot et al., 2010, 2014](#)), greatly enhance strain localization ([Holyoke and Tullis, 2006b](#); [Menegon et al., 2008](#); [Montési, 2013](#)). Phyllosilicate are ubiquitous minerals constituting the continental crust frequently observed in fault and shear zones ([Boulton et al., 2012](#); [Hunter et al., 2016](#)), with clay-bearing fault gouges in fault zones ([Rutter et al., 1986](#); [Zoback et al., 1987](#); [Faulkner et al., 2003](#)), and mica-rich phyllonites and mylonites in shear zones ([Bell and Etheridge, 1973](#); [Sibson, 1977](#); [Behrmann, 1985](#); [Imber et al., 2001](#)). The development of interconnected weak layers is more likely to happen when the proportion of weaker phase is high, due to reduced spacing between them ([Bons and den Brok, 2000](#)). Shape variation, spatial distribution and proportion of weak and strong phases hence influence directly the strength of the material ([Dell'Angelo and Tullis, 1996](#); [Handy et al., 1999](#); [Takeda and Griera, 2006](#); [Gerbi, 2012](#); [Montési, 2013](#); [Gueydan et al., 2014](#)). Particularly when newly formed minerals in shear zones are weaker than the host rock assemblage as phyllosilicates ([Mitra, 1978](#); [White et al., 1980](#); [Tullis and Wenk, 1994](#)). Indeed, mica can pervasively substitute strong phases, as feldspar ([Fitz Gerald and Stünitz, 1993](#); [Park et al., 2006](#); [Menegon et al., 2008](#); [Mansard et al., 2018](#); [Airaghi et al., 2020b](#); [Alaoui et al., 2023](#)). Micas are recognized as weak due to their crystal structure, low shear strength, and deformation mechanisms. Mica sheet-like structure with weak van der Waals bonds between layers facilitates easy

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cleavage and sliding, reducing overall strength (Kronenberg et al., 1990; Shea & Kronenberg, 1993). Mica deforms through frictional sliding and flexural slip, mechanisms that accommodate strain more easily than those in stronger minerals like quartz or feldspar (Holyoke & Tullis, 2006). Additionally, mica-rich rocks often show lower mechanical strength and higher ductility, as observed in both field studies and laboratory experiments (Fitz Gerald & Stünitz, 1993; Menegon et al., 2008). Phyllosilicate growth has been recognized to be one of the major fluid-induced softening reaction contributing to the fault and shear zones development both in greenschist and amphibolite facies (Bell and Etheridge, 1973; McCaig et al., 1984; Wintsch et al., 1995; Wibberley, 1999; Goncalves et al., 2016). Indeed, the phyllosilicates growth decreases bulk rock strength and phyllosilicates abundance governs shear zones location and bulk rock strain intensity (Gueydan et al., 2003; Wehrens et al., 2016; Airaghi et al., 2020a; Graziani et al., 2020; Alaoui et al., 2023). Therefore, phyllosilicates are generally considered weaker than other silicates and to accommodate strain (Shea and Kronenberg, 1992; Wintsch et al., 1995; Holyoke and Tullis, 2006b; Mariani et al., 2006; Hirth and Guillot, 2013; Hunter et al., 2016; Hirauchi et al 2023). Also, phyllosilicates have a pervasive influence on strain accommodation mechanisms of other silicates as for example quartz (see the following section for a detailed description).

However, research focusing on quartz, feldspar and olivine prevailed over that on mica deformation because of their weakness and the difficulty to carry out experimental studies with strongly anisotropic minerals such as phyllosilicates. Indeed, phyllosilicate crystal orientation relative to principal stress axes strongly affect the strength (Kronenberg et al., 1990; Shea and Kronenberg, 1993), as well as deformation mechanisms mentioned in the following section.

Therefore, in order to understand the behavior of the lithosphere-scale structures over geological timescale, it is crucial to identify the processes responsible for strain localization. More specifically, it is essential to explore the evolution of the active softening mechanisms with increasing strain to better constrain how shear zones form and to model the evolution of the strength of the continental crust. Yet, in actual rheological models of the continental crust phyllosilicates are mostly neglected in the sense that the rheology of the phyllosilicate-rich rocks is treated as the one of hydrated quartzites.

Consequently, in order to better constrain behavior and properties of fault and shear zones it is pivotal to better understand the behavior of phyllosilicates in deforming rocks.

II.2. Deformation processes in mica

Mica minerals are distinguished by their unique sheet-like crystal structure and exceptional cleavage properties, which stem from their internal bonding. The structure consists of stacked layers of silicate tetrahedra and octahedral sheets, creating a robust yet flexible framework.

Each layer in the mica structure comprises two silicate tetrahedral sheets with silicon atoms surrounded by oxygen atoms (Fig. 5). These tetrahedral sheets sandwich an octahedral sheet made up of aluminum or magnesium hydroxide. The tetrahedral sheets are interconnected through shared oxygen atoms, forming a stable two-dimensional network. The octahedral layer, positioned between the tetrahedral sheets, provides additional structural integrity. In muscovite, this layer is primarily composed of aluminum, while in biotite, it includes magnesium and iron.

The tetrahedral sheets (T), shown in blue, and the octahedral sheets (O), depicted in orange, are stacked in a sequence (Fig. 5). The interlayer cations (I), represented as purple spheres, reside between the tetrahedral sheets of adjacent T-O-T units. These cations, such as potassium in muscovite or magnesium and iron in biotite, act as binding agents, maintaining the structure's stability through weak ionic bonds. Trioctahedral layers, typical of biotite, have all octahedral sites occupied by cations, while dioctahedral layers, characteristic of muscovite, have only two-thirds of the octahedral sites occupied (Fig. 5). This differentiation is crucial in distinguishing various types of mica.

The perfect basal cleavage of mica is due to the weak ionic bonds between the interlayer cations and the adjacent T-O-T units. When stress is applied, these bonds break easily, allowing the mineral to split into thin, flexible sheets. This intricate arrangement and the weak bonding between layers explain mica's characteristic cleavage and flexibility, making it valuable in various industrial applications.

Therefore, various types of mica can behave differently due to their structure, composition and stability fields (Fig. 5; [Wilson and Bell, 1979](#); [Bell et al., 1986](#); [Kronenberg et al., 1990](#); [Christoffersen and Kronenberg, 1993](#); [Mares and Kronenberg, 1993](#)), further questions arise from the difference in the

stability conditions of muscovite and biotite highlighting the necessity of further experimental testing of crystal-chemical effects on rheology (Kronenberg et al., 1990; Shea and Kronenberg, 1992, 1993; Mares and Kronenberg, 1993; Dahl and Dorais, 1996). In fact, on a grain scale, few experimental studies have investigated the behavior of mica in the viscous realm (Shea and Kronenberg, 1992, 1993; Christoffersen and Kronenberg, 1993; Holyoke and Tullis, 2006a, b). As a result, the intra-crystalline mechanisms of deformation are still poorly constrained, and the processes responsible for substantial grain size reduction in mica are not understood at all, except for nucleation of phases with different chemical composition (Wilson and Bell, 1979), constituting recrystallization or metamorphic reactions.

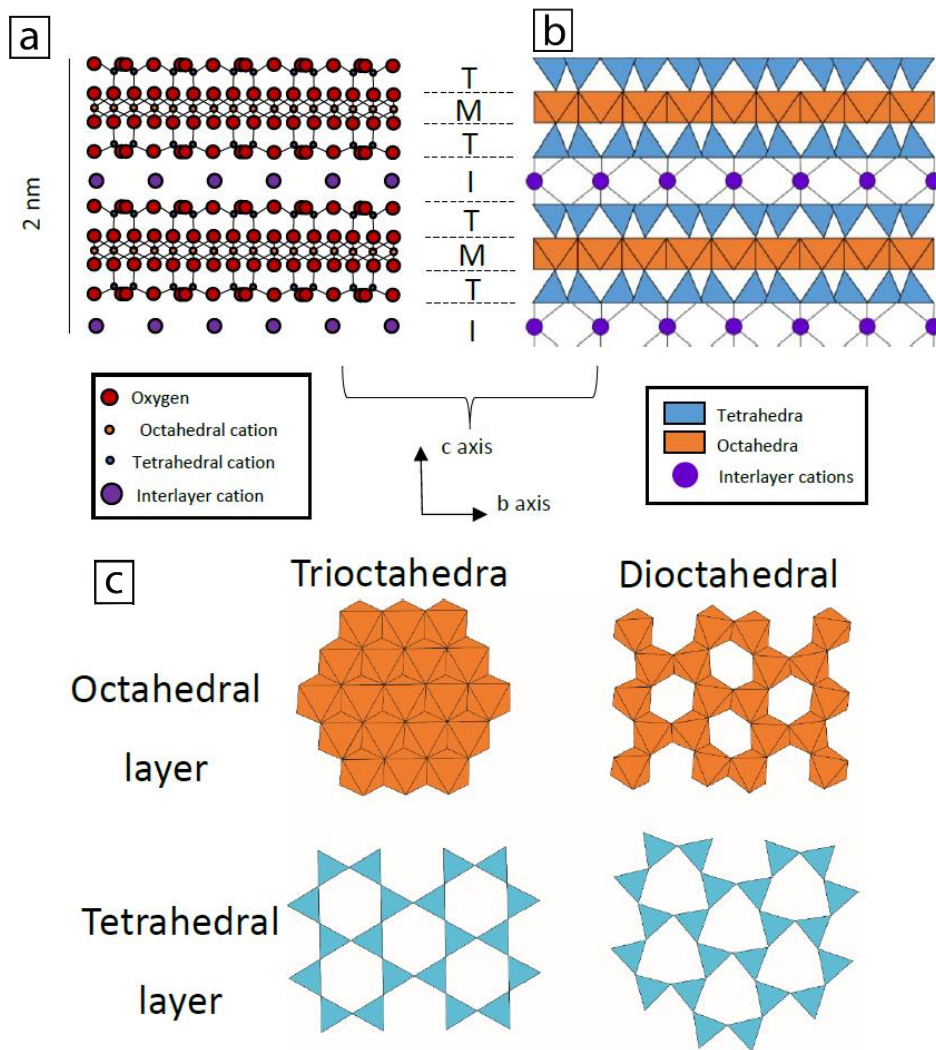


Figure 5 : Illustrations of mica crystal structures. a) Ball and stick model of a trioctahedral mica structure viewed from the a direction. Tetrahedral (T), octahedral (M) and interlayer (I) layers are highlighted to the right. b) Block model of the same mica lattice also viewed from the a direction showing the arrangement of octahedral and tetrahedral structures, which form TMT layers separated by interlayers. c) Block diagrams viewed from the c direction of octahedral and tetrahedral layers for both trioctahedral and dioctahedral micas. In a dioctahedral mica, one in every three octahedral spaces is vacant, the unevenly distributed charges distort the shapes of the resulting octahedral and tetrahedral layers (Aslin, 2019).

White micas in mylonites often have a strong crystallographic fabric called mica fish microstructures, which manifest in various distinctive shapes, such as lenticular fish, rhomboidal fish, and fish with either low or high aspect ratios (Fig. 6, [ten Grotenhuis et al., 2003](#)). Mica fish serve as valuable indicators of shear sense and often delineate the S and C surfaces in S/C mylonitic fabrics, with their basal planes contorted to conform to this fabric orientation. Additionally, mica fish can exhibit elongated, slender trails of very fine-grained mica emanating from their edges, extending into the fine-grained matrix parallel to the direction of shear ([ten Grotenhuis et al., 2003](#)). Mica fish microstructures develop through a combination of crystal plastic deformation involving slip on (001) and rigid rotation (Fig. 6). The mica grain may undergo deformation via slip on (001), resulting in a distorted rhomboidal shape during, after, or as an alternative to this process ([ten Grotenhuis et al., 2003](#)).

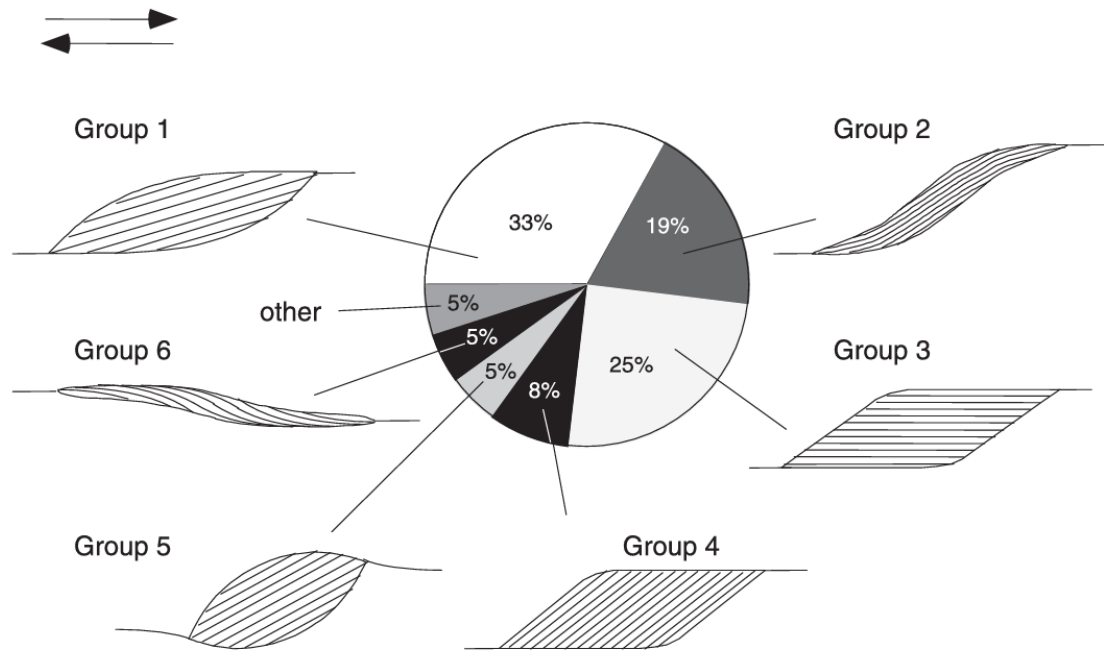


Figure 6 : Schematic drawings and pie chart showing the different morphological types of mica fish (after [ten Grotenhuis et al., 2003](#)). The groups are as follows: Group 1 (33%): Lenticular mica fish; Group 2 (19%): Lenticular fish with points inclined in the direction of the foliation; Group 3 (25%): Rhomboidal shaped fish with (001) planes parallel to the longest side of the fish.; Group 4 (8%): Rhomboidal shaped fish with (001) planes parallel to the shortest side of the fish; Group 5 (5%): Fish with a small aspect ratio and curved tails; Group 6 (5%): Mica fish with a high aspect ratio and inverted stair-stepping; Other (5%): Various other forms not fitting into the above categories. Incorrect interpretation of these structures, without considering their geological context, could lead to erroneous shear sense determination.

Generally, mica has been recognized to deform plastically by basal sliding along the cleavage (001) plane, when σ_1 is oriented at a low angle to the latter (Fig. 7, Bell et al., 1986; Bell and Cuff 1989; Kronenberg et al., 1990; Mares and Kronenberg, 1993; Meike 1989). Nevertheless, evidences for basal as basal dislocation (dislocation in (001) glide plane), were seen only by Meike (1989) and Kronenberg et al. (1990). Due to their layered crystal structures, phyllosilicates restrict dislocation glide primarily to the basal plane, offering no opportunity for dislocation glide along other planes (Kronenberg et al., 1990). However, deformation results in the development of high angle kink bands (Dewey, 1965) and strain cannot be accommodated by slip on (001) plane, when σ_1 is oriented parallel to the latter (Kronenberg, et al., 1990).

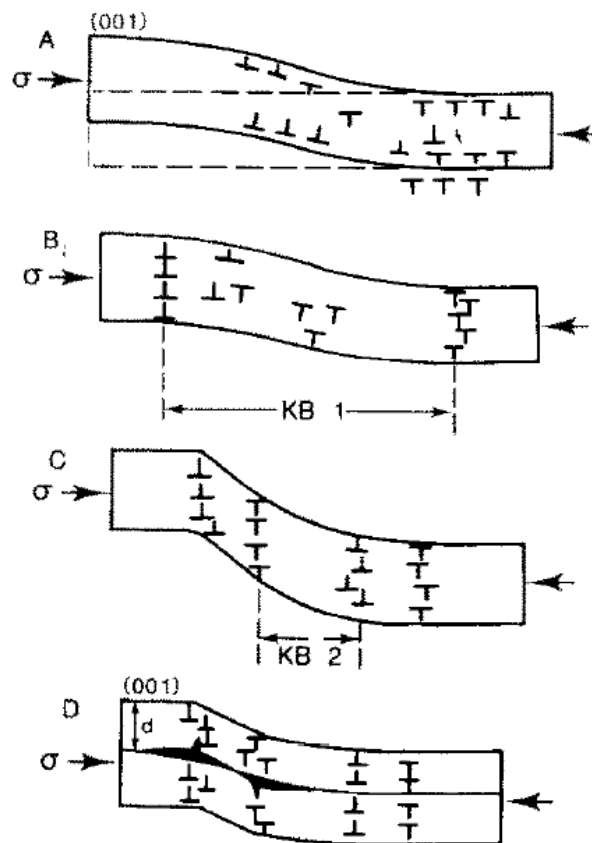


Figure 7: Model to explain the formation of kinks (after Bell et al., 1986). A: Elastic bending of a crystal along the (001) slip plane. B: Increased stress leads to an increase in dislocation density and the formation of dislocation walls, resulting in the formation of the first kink (KB1). C: The formation of new dislocation walls continues with further shortening, creating a new generation of kink (KB2) within the disoriented lattice region (within KB1). D: Expansion of the (001) slip planes at the borders of the kinks. d represents the width, which remains constant.

Kink band development is a classical deformation mechanism observed in both white mica and biotite (Fig. 7, Dewey, 1965; Bell et al., 1986; Bell and Cuff 1989; Meike 1989; Kronenberg et al., 1990; Mares and Kronenberg, 1993). Indeed, the formation of tight intracrystalline folds known as kinks result of basal plane perpendicular strain in mica. When two folds run parallel to each other, they form what is referred to as kink band boundaries, surrounding a kink band (Fig. 7, 8a). Kinking is common in phyllosilicates as it requires pronounced anisotropy, however it can be also observed in other minerals. Kink bands intertwine to create intricate interconnecting networks facilitating basal plane-parallel shortening in larger mica grains. The development of kink bands consists of nucleation, longitudinal propagation and lateral growth (Weiss, 1980). Etheridge et al., 1973 showed that lenticular regions of lattice, where basal planes misoriented with respect to those of their host grains by the angle of bending (ω), are the initial core of kink bands (Fig. 7, 8b). Longitudinal propagation takes place at the boundaries of these lenticular regions following high elastic strain concentrated in the flanking undeformed lattice. Finally, the kink band boundaries will grow laterally reaching the undeformed lattice and widening the kink band (Weiss, 1980). Afterwards, a new generation of kink band will nucleate following the same development. In detail, the exact process through which kink bands develop remains unclear, primarily due to the challenge of explaining such sharp bending and deformation parallel to the c-axis solely through basal dislocation glide.

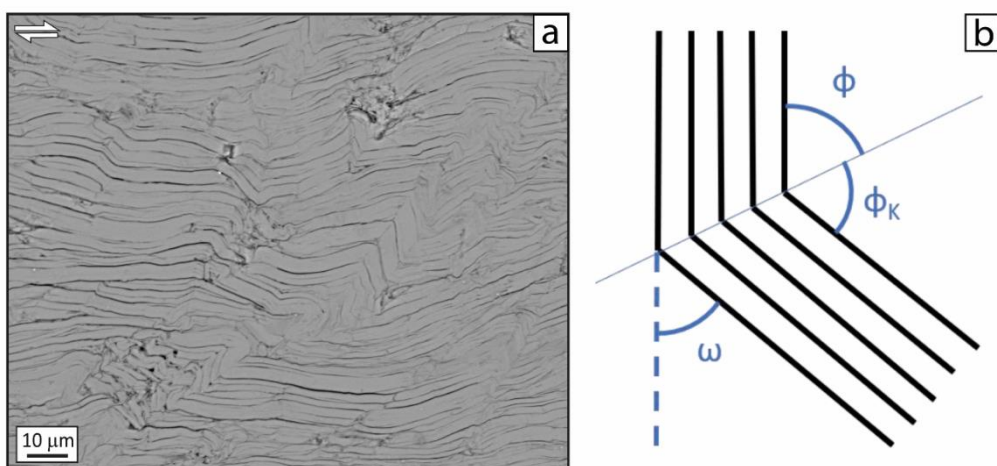


Figure 8 : a) Plane polarized light optical micrograph of a kink band within a biotite grain. The sharp fold axes are the kink band boundaries, the orientation of the basal planes is highlighted by dark micro-cleavages. b) Conceptual diagram illustrating the components of a kink band (Aslin, 2019 adapted from Etheridge et al. 1973).

The most detailed models propose intricate arrangements of dislocation walls and stacking faults concentrated within a limited region (Etheridge et al., 1973; Bell et al., 1986). However, even these models acknowledge the necessity of another mechanism to account for c-axis strain and address the asymmetry observed in kink bands. The latter is defined by $|\phi - \phi_K|$, where ϕ is the angle between (001) in the area adjacent to the kink band and the axial plane of the kink band boundary and ϕ_K is the angle between (001) in the area within the kink band and the axial plane of the kink band boundary (Fig. 7, 8b; Etheridge et al., 1973). Though they are more prevalent in micas subjected to higher temperatures and compressed along [010], asymmetric kink bands are observed under various conditions (Etheridge et al., 1973). This asymmetry leads to an expansion in the basal plane spacing within the kink band, a phenomenon that cannot be accounted for solely by basal dislocation (Fig. 7).

Recently, ripplocations a new type of defect and accompanying deformation mechanism, have been proposed in materials science and could elucidate intracrystalline c-axis parallel strain in layered materials (Kushima et al., 2015; Gruber et al., 2016; Barsoum and Tucker, 2017; Griggs et al., 2017; Freiberg et al., 2018; Barsoum et al., 2019). These ripplocations manifest as atomic-scale ripples with the combined characteristics of both a ripple and a basal dislocation, and they are applicable to micas (Fig. 9, Aslin et al., 2019). Indeed, ripplocation have been proposed for biotite (Aslin et al., 2019; Airaghi et al., 2020b), but have not yet been observed in white mica. Ripplocations in biotite ensure a propagation of dilatancy of the interlayer across the c-axis. This deformation process has also been recognized as possible pathway, via porosity, for fluid circulation that triggers further transformation during shear zone formation (Aslin et al., 2019; Airaghi et al., 2020b).

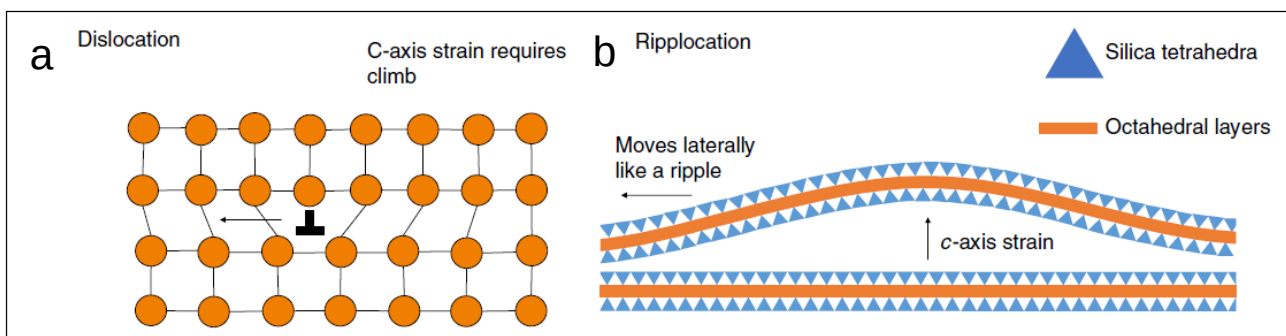


Figure 9 : Comparison between a dislocation (a) and a ripplocation in a generic phyllosilicate lattice (b). Both enable the movement of one plane of atoms over another. However, while dislocation climb is required to deform out of this plane in (a), ripplocation intrinsically contain an element of c-axis parallel strain due to bending and more crucially are attracted to each other rather than repelled (after Aslin et al., 2019).

However, other processes than mica fish, basal slip, dislocation glide and kinking, such as oriented growth can account for plastic deformation of micas ([Stallard and Shelley, 2005a](#)). The heterogeneous chemical composition often observed in matrix micas suggests that micas are susceptible to deform by dissolution (pressure-solution; [Airaghi et al., 2017a, b](#), [Hentschel et al., 2019](#)). Experimental observation of pressure-solution in phyllosilicates is lacking, but it is speculated to occur in natural settings, such as during the development of crenulation cleavage and metamorphic differentiation ([Starkey, 2002](#)). Microstructures of biotite and muscovite from a deformed pegmatite were characterized as irregular grain boundaries, serrated fracture planes, shape variations, and notable differences in strains in individually folded grains, all which indicate of dissolution processes ([Wilson and Bell, 1979](#)).

Finally, other deformation mechanisms for mica would be grain size reduction and recrystallization but the presence of conventional dynamic recrystallization mechanisms in micas is a subject of debate. As micas possess a pronounced mechanical anisotropy and are prone to kinking, it sets them apart from materials like quartz and metals. Conversely to quartz, where typical microstructures such as core and mantle structures and chessboard grains are commonly used to identify dynamic recrystallization, low-angle subgrain boundaries (detailed in the following subsection) are rarely observed in micas. Consequently, these typical markers of dynamic recrystallization are not readily apparent in micas, but new small grains around large, inherited ones, are often present. Various mechanisms for grain size reduction in micas have been proposed, including bulging recrystallization at high-angle boundaries ([Etheridge and Hobbs, 1974](#)), segmentation through the generation of a series of slight misorientations parallel and subnormal to (001) ([Wilson and Bell, 1979](#); [Bell and Wilson, 1981](#)), and intracrystalline folding and cataclasis ([Goodwin and Wenk, 1990](#); [ten Grotenhuis et al., 2003](#)).

II.3. Deformation processes in quartz

As for mica, several factors can play a role on deformation processes of quartz such as temperature, pressure, strain rate, displacement rate, stress, water content and chemical composition of the system. Through the last decades, a large number of studies have focused on deformation processes in quartz in both natural rocks ([Kronenberg et al., 1990, 2020](#); [Nakashima et al., 1995](#); [Menegon et al., 2008](#); [Stipp et al., 2002](#); [Kilian et al., 2011](#); [Finch et al., 2016](#); [Kilian et al., 2016](#); [Ceccato et al., 2022](#)) and in

experimentally deformed single crystals or polymineralic material, in both brittle and viscous conditions (Carter et al., 1964; Hobbs, 1968; Jaoul et al., 1984; Kronenberg and Tullis, 1984; Mainprice and Paterson, 1984; Hirth and Tullis, 1992; den Brok et al., 1994; Gleason and Tullis, 1995; Post and Tullis, 1998; Palazzin et al., 2018; Richter et al., 2018; Fukuda et al., 2018, 2019; ; Nègre et al., 2021; Ceccato et al., 2022; Ghosh et al., 2022). In the viscous realm, the plastic deformation is described as the dominant process. The presence of a strong crystallographic preferred orientation (CPO) in quartz, a monoclinic fabric, and a large grain size, among others, are associated with dislocation creep (Baker and Wenk, 1972; Hobbs, 1985; Schmid et al., 1987; Stipp et al., 2002; Kilian et al., 2011). Conversely, a small grain size, a weak or absent CPO, and low fabric anisotropy are typically associated with diffusion creep (Boullier and Gueguen, 1975; Stünitz and Fitzgerald, 1993; Fliervoet et al., 1997; Mehl and Hirth, 2006; Kanagawa et al., 2008; Kilian et al., 2011). However, transitional microstructures exist because processes from both creep regimes can contribute to the final microstructure.

Three distinct dynamic recrystallization mechanisms are known (Fig. 10): bulging, subgrain rotation and grain boundary migration recrystallization (Bailey and Hirsch, 1962; Poirier and Nicolas, 1975; White, 1977; Poirier and Guillopé, 1979; Drury et al, 1985; Poirier, 1985; Urai et al. 1986; Wu and

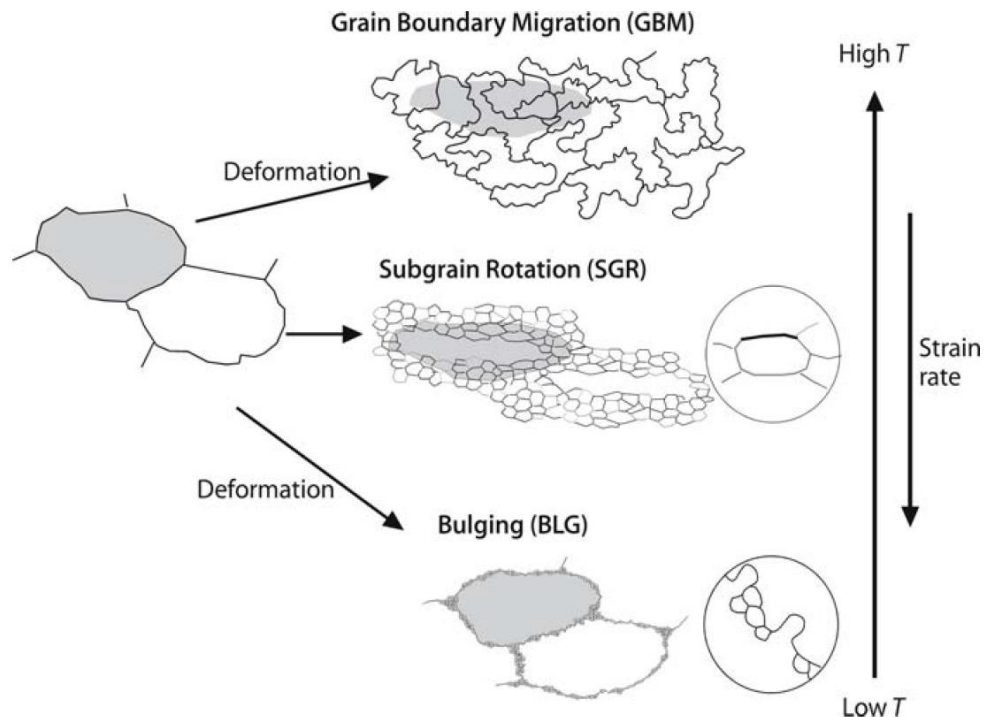


Figure 10 : The three main types of dynamic recrystallization in a polycrystal. The substance of one of two large grains that recrystallize is shown by grey shading, before and during recrystallization (Passchier and Trouw, 2005)

Groshong 1991a; Hirth and Tullis 1992; Dunlap, 1997; Stipp et al. 2002). Bulging (BLG) recrystallization happens when the grain boundary bulges into the crystal with high dislocation density forming new small crystals (Means 1981; Tungatt and Humphreys 1984; Urai et al. 1986; Passchier and Trouw, 2005; Stipp et al. 2002). Evidences for BLG are very strong bimodal grain size with many small grains mainly along old grain boundaries with optical micrographs, and grains with different dislocation density with TEM imaging (Fig. 10, 11; Passchier and Trouw, 2005).

If dislocations are relatively free to climb from one lattice plane to another, dislocations might add continuously to subgrain boundaries. This leads to an increase of the angle between the crystal lattice on both sides of the subgrain boundary, until the subgrain can no longer be part of the same grain. Therefore, a newly formed grain developed by subgrain rotation (SGR) recrystallization (Passchier and Trouw, 2005).

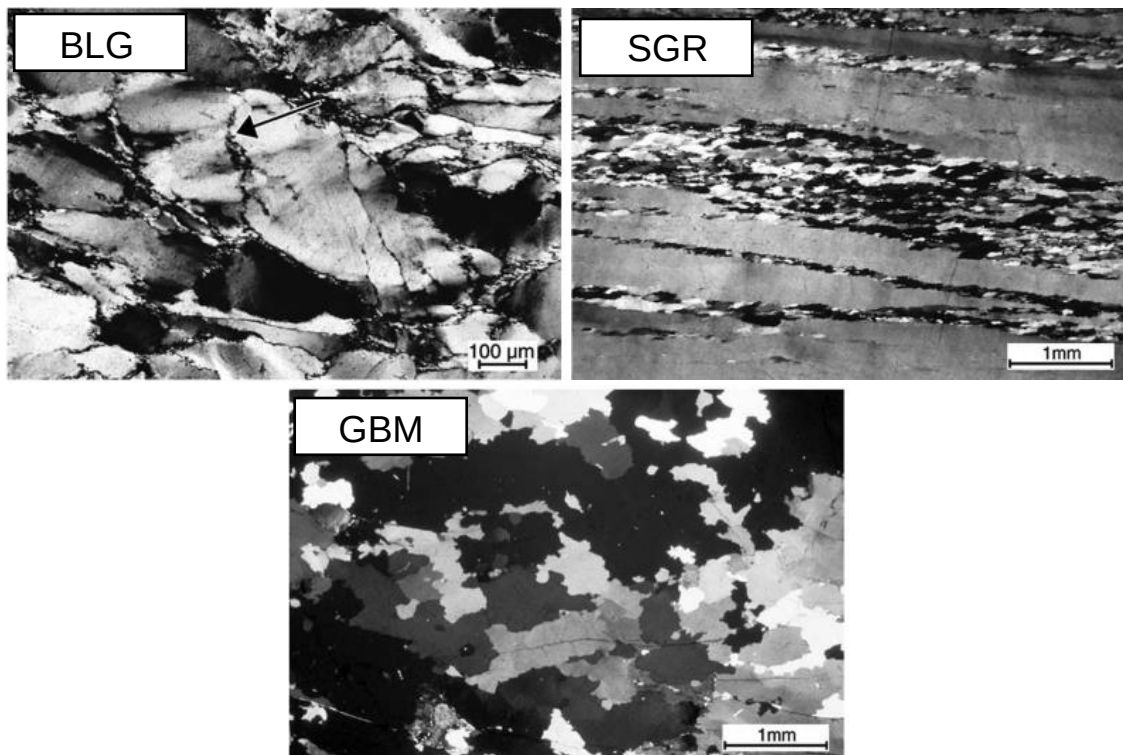


Figure 11 : Micrographs showing examples of the three main types of dynamic recrystallization (after Stipp et al., 2002).

SGR is evidenced by gradual transitions between undulose-subgrains-grains and orientated families of grains, often exhibiting core-and-mantle structures in optical microscopy (Fig. 10, 11), also when all grains have the same dislocation density (TEM) (Passchier and Trouw, 1996). When BLG shifts to SGR, there is a switch from bulging, mantle subgrain formation and recrystallization along the grain boundaries to dominant subgrain formation and recrystallization (Passchier and Trouw, 1996). Grain boundary migration (GBM) recrystallization is defined by grain boundary mobility enhanced to an extent where grain boundaries can traverse entire crystals, eliminating dislocations and potentially subgrain boundaries in the process (Guillopé and Poirier 1979; Urai et al. 1986; Passchier and Trouw, 1996; Stipp et al. 2002). Grain boundary migration is temperature dependent, and indicates no new grain but only migration of former boundaries. Sharp boundaries with old grains, bulges, pinning, left-over grains structures witness GBM in optical micrographs, while in TEM imaging GBM is evidenced by strong variation in dislocation density from grain to grain (Fig. 10, 11; Passchier and Trouw, 1996). SGR transitions to GBM when there is a switch from core subgrain formation and recrystallization to dominant fast grain boundary migration sweeping entire grains grain (Passchier and Trouw, 1996).

Hirth and Tullis (1992) experiments highlighted three dislocation creep regimes for quartz aggregates, which are temperature, strain rate, and water content dependent during deformation (Fig. 12). The shift between each regime happens gradually and depends on the rates of grain boundary migration, dislocation climb, and dislocation production. Regime 1 is defined by the absence of fracturing, strain-induced grain boundary migration, no climb-accommodated recovery and progressive strain weakening. Conversely, regime 2 is characterized by the absence of fracturing, climb-accommodated recovery, subordinate grain boundary migration and no progressive strain weakening. In Hirth and Tullis (1992) experiments BLG corresponds to regime 1, SGR to regime 2 and GBM to regime 3.

Overall, past studies focused mainly on dislocation creep of quartz to understand the rheology of quartz as [Rutter and Brodie \(2004a, b\)](#) pointed out that, unrealistically small grain sizes below 1 μm are required for dominant diffusion creep in nature. Nonetheless, diffusion creep-related processes involve the formation of grain boundary alignment, phase mixing, and structures related to dissolution-precipitation ([Drury and Humphreys, 1988](#); [Kruse and Stünitz, 1998](#); [Wintsch et al., 2002](#)). While these criteria are often observed in metals or phase mixtures, reports of diffusion creep in quartz are rare ([Behrmann, 1985](#)). Grain boundary sliding (GBS) in quartz occurs when grains move past each other along their boundaries, facilitated by high temperatures, low to moderate stress, small grain sizes, and the presence of fluids ([Hirth & Tullis, 1992](#)). Microstructural evidence such as grain boundary migration and dynamic recrystallization observed in both naturally deformed rocks and experimental studies ([Stipp et al., 2002](#); [Fusseis et al., 2009](#); [Kilian et al., 2011](#)) supports the significant role of GBS in quartz deformation. Theoretical models suggest that GBS, combined with other mechanisms like diffusion creep, enhances the ductility of quartz-rich rocks ([Rutter, 1983](#)).

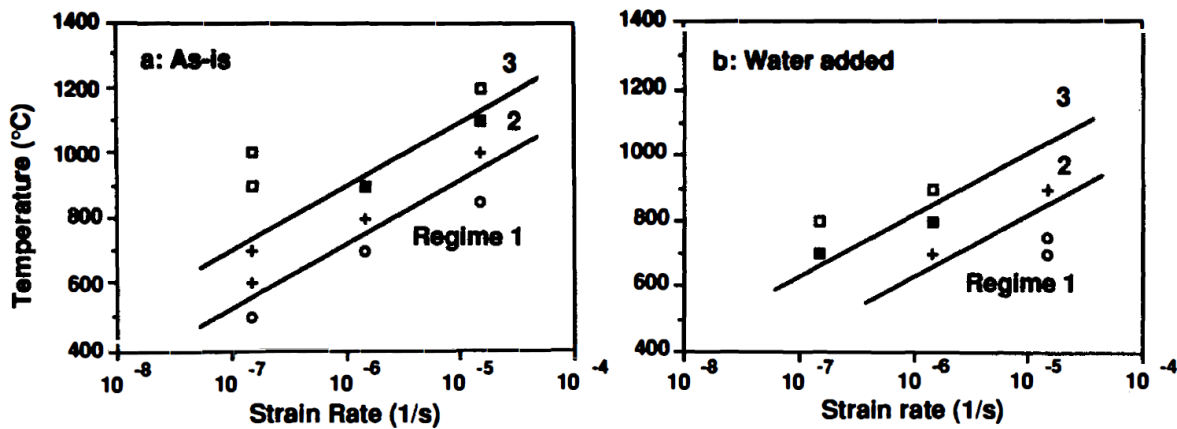


Figure 12 : Experimental conditions (strain rate and temperature) for the three dislocation creep regimes defined for quartz aggregates, a) deformed “as-is” or b) with water added ([Hirth and Tullis, 1992](#)).

II.4. Dissolution-precipitation as film-fluid diffusion at grain boundaries

Dissolution-precipitation as film-fluid diffusion at grain boundaries plays a pivotal role in the deformation and metamorphism of polymineralic rocks, particularly through pressure solution creep. This process initiates with the dissolution of minerals at stressed points along grain boundaries, where fluid films then facilitate the diffusion of dissolved ions towards lower stress areas, leading to their precipitation (Gratier et al., 1999; Gratier et al., 2009; Hellmann et al., 2002; Den Brok, 1998; Anzalone et al., 2006; Renard et al., 2001).

The geometry of the fluid film varies depending on factors such as mineral composition and stress conditions. It can exist as continuous films that cover grain boundaries uniformly, or as island-channels where fluid accumulates in isolated pockets connected by narrow channels (Fig. 13). In some cases, fluid films accumulate predominantly at triple junctions where three grains meet, influencing local ion transport and precipitation dynamics (Den Brok, 1998; Gratier et al., 1999).

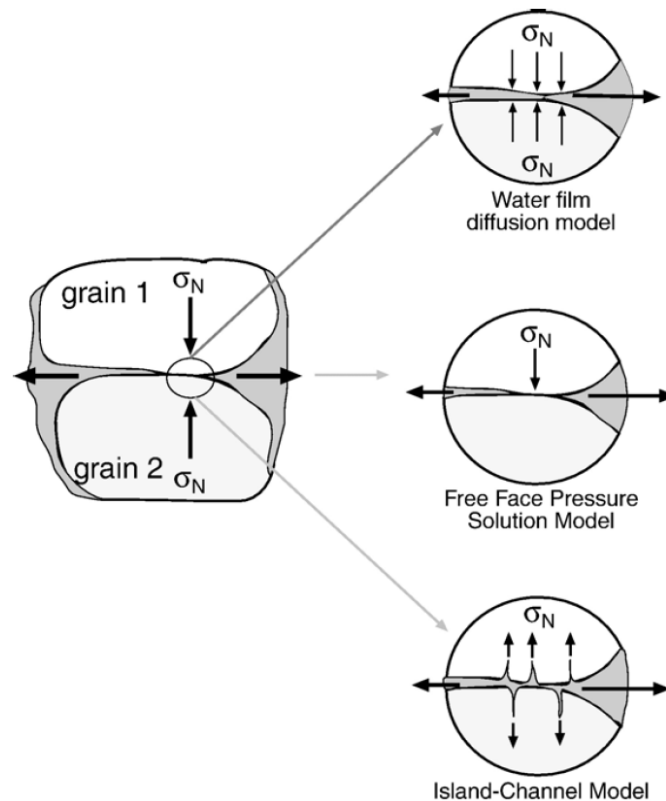


Figure 13 : Proposed models of pressure solution (Anzalone et al., 2006 adapted from den Brok, 1998 and Renard et al., 1999). Vertical arrows refer to normal pressures or crack propagation directions. Horizontal arrows refer to material transport.

II.4. Dissolution-precipitation as film-fluid diffusion at grain boundaries

Chemically, the fluid film is typically an aqueous solution containing ions such as silica (SiO_2), sodium (Na^+), potassium (K^+), calcium (Ca^{2+}), magnesium (Mg^{2+}), chloride (Cl^-), and carbonate (CO_3^{2-}). The composition of this fluid significantly influences the rates of dissolution and precipitation reactions along grain boundaries, altering the mechanical behavior of the rock (Fig. 14, Gratier et al., 1999; Anzalone et al., 2006; Meyer et al., 2006; Greene et al., 2009).

Diffusion rates of these dissolved ions within the fluid film are crucial parameters, varying with temperature, pressure, and the specific chemistry of the solution. For instance, silica ions typically exhibit diffusion coefficients in the range of 10^{-10} to 10^{-9} m^2/s , influencing the kinetics of dissolution and precipitation processes (Farver and Yund, 2000; Renard et al., 2001; Gratier et al., 2009).

Understanding the complex interplay of geometry, chemistry, and diffusion rates in fluid films at grain boundaries is essential for developing accurate models of rock deformation and metamorphism. These insights not only advance our understanding of geological processes but also contribute to predicting the behavior of polymineralic rocks under various natural conditions.

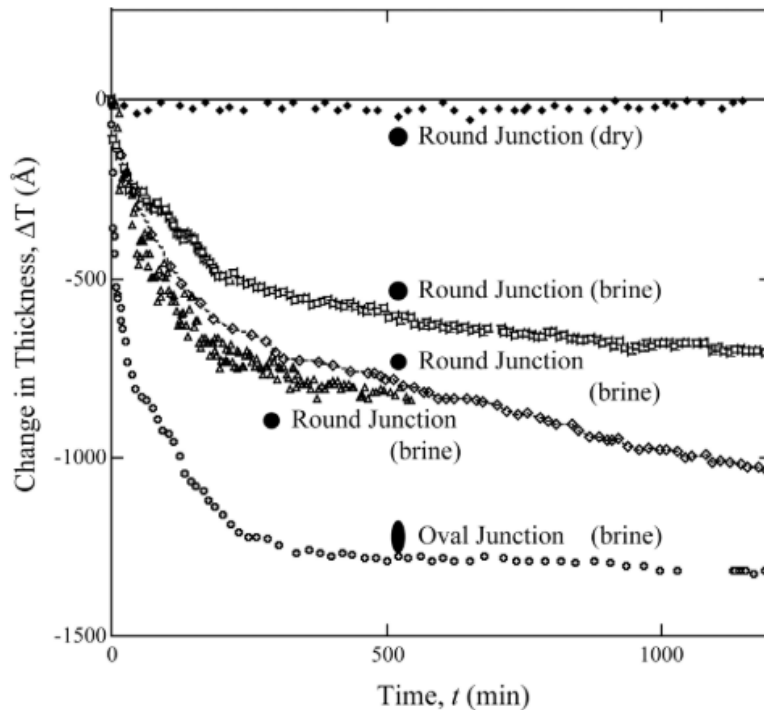


Figure 14 : Five representative data sets from nine SFA experiments (after Meyer et al., 2006) showing the change in Z-cut quartz thickness over time when in contact with muscovite mica at 2-3 atm pressure. The bottom four curves (open symbols) are from experiments in acidic brine, while the top curve (solid symbols) is from dry conditions. The shape and relative size of each junction are indicated above each plot, with contact areas approximately 1.0 ± 0.5 mm^2

II.5. Effect of mica on quartz microstructures

The presence of mica and the interconnection of mica grains play a key role in the deformation and recrystallization of anhydrous, nominally stronger phases as quartz (Tullis and Wenk, 1994; Hunter et al., 2016; Holyoke and Tullis, 2006b; Alaoui et al., 2023; Tokle et al., 2023). The increase in mica content causes decrease in the sample strength (Tullis and Wenk, 1994; Cacciari and Futai 2019). At grain scale, the growth of mica and the increase in the interconnection of mica grains inhibits quartz recrystallization at its boundaries through the Zener-pinning effect and results in a weakening of the crystal preferred orientation (CPO) of quartz (Song and Ree, 2007; Little et al., 2015; Hunter et al., 2016; Cross et al., 2017; Alaoui et al., 2023).

In a biminerale and polyminerale rocks, the rheology of the rock during pressure solution may be drastically reduced relative to its monomineralic equivalents, and the importance of pressure-solution is underestimated (Wheeler, 1992). In fact, phyllosilicates affect film fluid diffusion at grain boundaries and enhance dissolution-precipitation processes in stronger phases, particularly in quartz (Becker, 1995; Schwarz and Stockhert, 1996; Farver and Yund, 2000; Rutter and Wanten, 2000; Renard et al., 2001; Anzalone et al., 2006; Meyer et al., 2006; Gratier et al., 2013). Niemeijer (2018) assigns structural observations to mixed creep regimes with frictional sliding in muscovite (present at quartz grain boundaries) coupled with pressure-solution mechanisms in quartz grains. These processes, influenced by phyllosilicates and pressure solution, contribute to promoting velocity-weakening behavior (Niemeijer, 2018). Experiments on quartz-mica aggregates showed a decrease in the pressure-solution rate of quartz when muscovite is present, indicating that muscovite accommodates a significant proportion of increasing strain (Niemeijer and Spiers, 2005, 2007). This type of micro-rheological inter-phase transfer influences strain localization as a function of changes induced by dissolution-precipitation under stress. Hence, in an assemblage made of quartz and mica, weakening is partially linked to the activation of quartz pressure-solution by the presence of mica (Fig. 14, Rutter and Wanten, 2000; Alcantar et al., 2003; Anzalone et al., 2006; Meyer et al., 2006; Greene et al., 2009). The presence of mica in quartz mylonites has a significant enhancing effect on the solution-transfer diffusion creep in

quartz aggregates compared to mica-free rock (Renard et al., 1997; Farver and Yund 2000; Anzalone et al., 2006; Meyer et al., 2006; Greene et al., 2009).

Therefore, there is an importance of chemical aspects of the deformation mechanisms. Indeed, Various chemical components diffuse in response to stress, potentially mingling and interacting as they move through the network of grain boundaries. (Wheeler, 1992).

Wheeler (1992) predicts the possible important effect of pressure solution on rock strength and highlights that this is neglected in rheological models. In actual rheological models for crustal rocks, effects of mica transport, recrystallization and replacement properties, pressure-solution and other chemical interactions are not considered nor included. Generally, the rheology of mica-bearing rocks in large-scale thermomechanical models is addressed either by employing comparatively weak flow laws of other minerals, by integrating single-phase laws arbitrarily (Maggi et al., 2014), or by implementing strong and subjective kinematic assumptions, such as uniform strain rate (Gueydan et al., 2003).

II.6. Modelling of the rheology of monomineralic and polymineralic aggregates

Deformation tends to occur more in weaker areas of rock. This strain localization further weakens the affected rock volume, causing it to deform faster under the same stress compared to its surroundings. Rheological modelling is a way to quantify that weakening. Experimental deformation allows establishing/quantifying the relationship between the applied differential stress and the strain rate. Hence, flow law can be retrieved, at a specific pressure, temperature, for specific deformation mechanisms. This is usually performed for monomineralic material, while for polymineralic materials mixing models have been proposed. They are based on monomineralic flow laws of pure phases. In these cases, the bulk strain rate of the aggregate is defined as the sum of strain rates of each pure phase, considering the activation of various deformation mechanisms to establish an aggregate flow law (Tullis et al., 1991, Dimanov and Dresen, 2005, Platt, 2015, Ceccato et al., 2020). These composite flow laws for polyphase aggregates assume that the total strain rate is the sum of the dislocation creep strain rate and the diffusion creep strain rate.

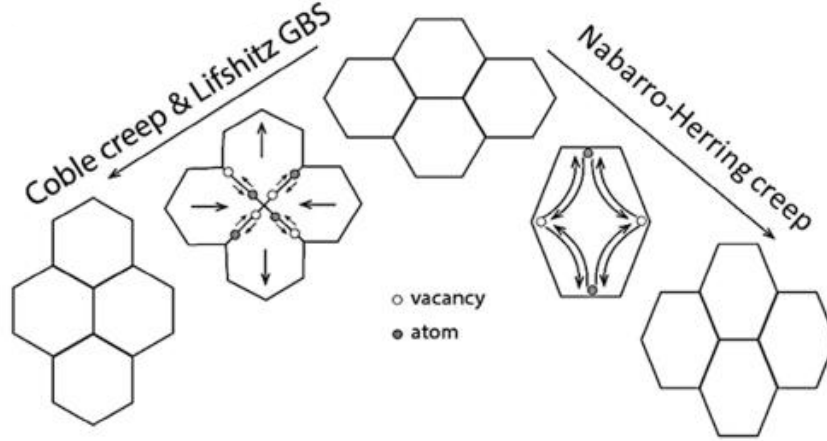


Figure 15 : Simplified sketch explaining Coble creep and Nabarro-Herring creep (Hobbs et al., 2019).

Other mixing models used flow laws for independent processes as dislocation/diffusion creep for quartz and feldspar (Hirth, et al., 2001; Rybacki et al., 2006; Tokle et al., 2019), or thin-film pressure solution for quartz (Fig. 13; den Brok, 1998). The processes of grain boundary sliding (GBS) and dissolution-diffusion-precipitation mechanisms are crucial for understanding creep laws in quartz-rich rocks. According to the work of Gratier et al. (1999), dissolution occurs at points of high stress, followed by the diffusion of dissolved ions through the intergranular fluid, and finally, precipitation in low-stress areas, facilitating the relative movement of grains (Gratier et al., 2009). These dissolution-diffusion-precipitation processes, also known as pressure solution creep, are essential for modeling viscous deformation in rocks and must be incorporated into creep laws to explain the evolution of rock structure and strength over geological timescales.

Flow law is generally expressed as: (1) for dislocation creep $\dot{\epsilon} = \sigma^n A e^{\frac{-Q}{RT}}$ and (2) for diffusion creep $\dot{\epsilon} = \frac{\sigma^n}{d^p} A e^{\frac{-Q}{RT}}$ with $\dot{\epsilon}$ is the strain rate, σ the differential stress, Q is the activation energy, R is the gas constant, T is the temperature, n is the stress exponent, d is the grain size and p is the grain size exponent. It is known that for $n = 1$ and $p = 2$ or 3 indicate, respectively, lattice diffusion (Nabarro-Herring) creep (Nabarro, 1948; Herring, 1950) or grain boundary diffusion (Coble) creep (Fig. 15; Coble, 1963). Whereas for dislocation creep process $n \geq 3$ and $p = 0$ in general (Ghosh et al., 2022). Usually, for dominant diffusion creep $n = 1$, for dominant dislocation GBS $n = 2$ and for dominant dislocation creep $n = 3$, nevertheless a dominant regime never excludes other processes. Additionally, water fugacity was

II.6. Modelling of the rheology of monomiralic and polymineralic aggregates

introduced to the flow law to incorporate the effect of water on the creep rates at a certain pressure-temperature (P-T) condition (Kohlstedt et al., 1995). Therefore, the flow law for the dislocation creep can also be expressed as: $\dot{\epsilon} = f_{H_2O}^r \sigma^n A e^{\frac{-Q}{RT}}$ where f_{H_2O} is water fugacity and r is water fugacity exponent. Flow laws of two-phased assemblages are generally constructed by averaging the rheological properties on pure end-members. In order to quantify the viscosity of two-phase mixtures, some rheological mixing models using end-member flow laws for each phase were developed (Tullis et al., 1991; Dimanov and Dresen, 2005; Huet et al., 2014; Platt, 2015; Ceccato et al., 2018, 2020; Rast and Ruh, 2021; Tokle et al., 2023).

These mixing models are based on the Reuss-Voigt (also known as Taylor-Sachs) models (Fig. 16) where the rheology of a two-phase aggregate is determined by averaging the rheology of its pure phases. It is anticipated to fall within two end-member scenarios (Fig. 16, 17): one where the same stress is applied to the different phases (iso-stress bound), and another where the same strain rate is applied to the different phases (iso-strain rate bound). In the iso-strain rate case, which defines the upper strength bound for a two-phase aggregate, the strong phase must deform at the applied strain rate, there is a poor effect of adding the weak phase (Fig. 16, 17).

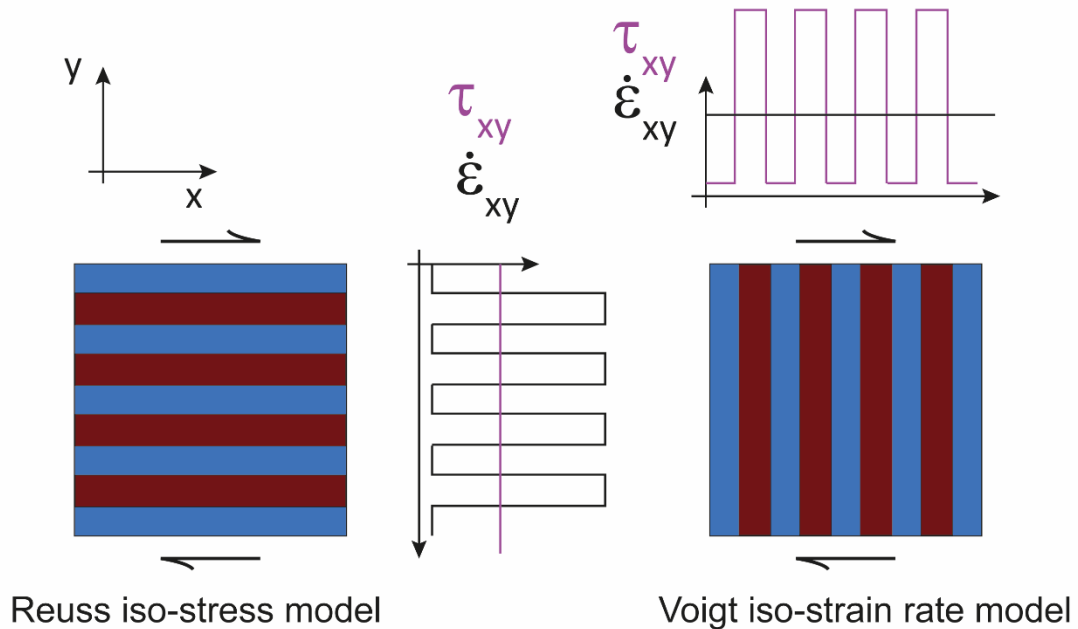


Figure 16 : Simplified sketch of Reuss-Voigt bounds models. Reuss iso-stress bounds with the same stress (τ) applied to phase A and B. As a consequence, the A and B respond to the stress by different strain rate (ϵ). Voigt iso-strain rate bound with the same strain rate applied to phase A and B and the behavior of the aggregate is controlled by the stronger phase.

II.6. Modelling of the rheology of monomiralic and polymineralic aggregates

For the iso-stress case, characterizing a lower strength bound, there is more partitioning of the strain rate into the weaker phase indicating a high weakening effect of adding the weak phase (Fig. 16, 17). Rheological mixing models reproduce field observations and experimental data, nonetheless in an averaging way inducing various limitations (Huet et al., 2014; Platt, 2015; Ceccato et al., 2020). In fact, mixing models hardly reproduce the experiment data over the full range of phase volume proportion (Dimanov and Dresen, 2005) and have no room left to the effects of chemical interactions on rock weakening. Indeed, these models do not include the reaction-induced weakening effects and therefore provide an estimate for maximum strength (Ceccato et al., 2020). In order to apply rheological mixing models, it is necessary to obtain flow law parameters for each end-member phase.

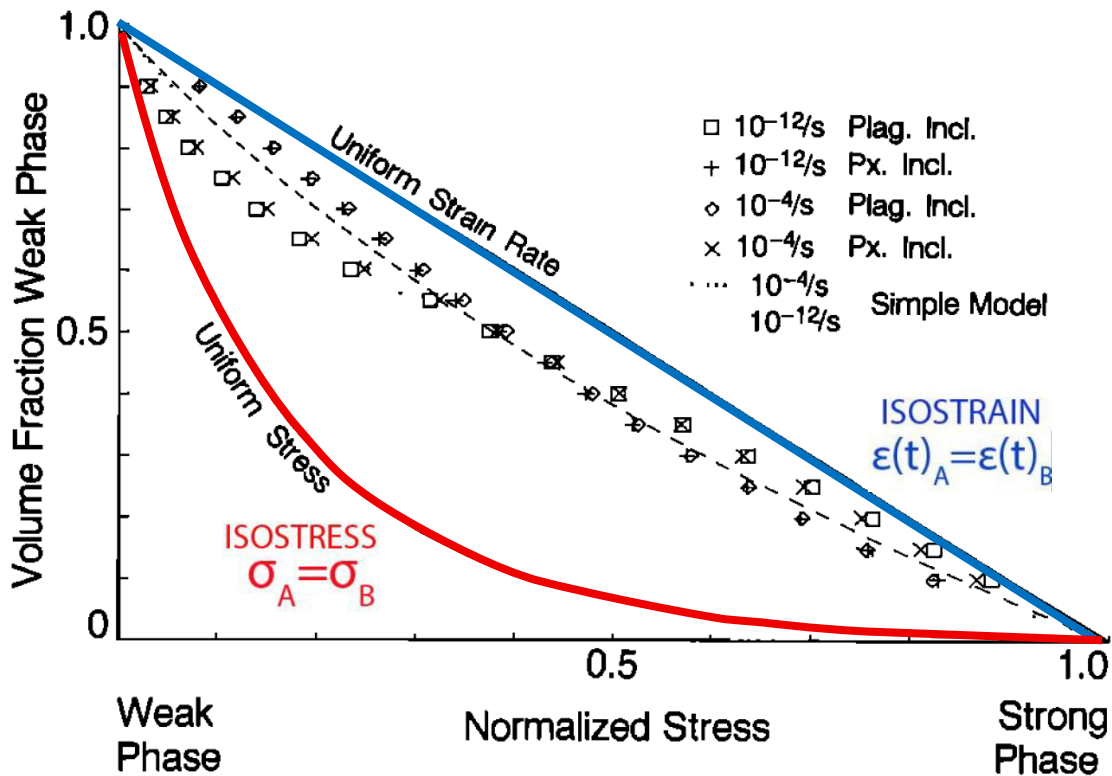


Figure 17 : Example of a rheological model for an aggregate made of phase A and B (plagioclase-pyroxene), obtained by combining the mechanical behavior of the pure phases (Tullis et al., 1991). The rheology of the aggregate lies between the Reuss-Voigt bounds (red and blue curves). Red curve: the same stress (σ) applies to phase A and B. As a consequence, the A and B respond to the stress by different strain rate ($\epsilon(t)$). Blue curve: the same strain rate applies to phase A and B and the behavior of the aggregate is controlled by the stronger phase

II.6. Modelling of the rheology of monomineralic and polymineralic aggregates

For quartz-mica assemblages, a quartz flow law is based on deformation experiments of polycrystalline quartz aggregates ([Tokle et al., 2019](#); [Ghosh et al., 2022](#)), whereas for muscovite a flow law is based on single crystal deformation experiments where the single crystal was oriented to facilitate basal slip ([Mares and Kronenberg, 1993](#); [Tokle et al., 2023](#)). However, the stress exponent n is not constrained for a biotite flow law. Another limitation to these mixing models is that as they are derived from mechanisms modelled by the end-member flow laws, they do not directly incorporate potential intergranular processes. Indeed, there is no room left for grain boundary sliding, dissolution-precipitation processes and interaction between phases which are often promoted in multiphase aggregates. Yet, it has been proposed in theory, that we may expect due to these interactions, weaker behavior from mixtures than for the weakest pure phase ([Wheeler, 1992](#)). Indeed, these classical models applied to two-phase aggregates are described as a mechanical mixture of both pure phases, and do not consider interphase processes promoted in these aggregates ([Pongrac et al., 2022](#); [Alaoui et al., 2023](#)).

Chapter III – Materials and methods

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III.1. Aims and approach

In order to study the mechanical behavior of mica + quartz, two-phases assemblages, we carried out deformation experiments combined with detailed microstructural and petrological studies (Fig. 1). The experimental approach makes it possible to control a multitude of parameters, such as pressure, temperature, strain rate, phase proportion or water content, allowing to investigate deformation and chemical mechanisms in detail for each mineral phase. Deformation experiments were conducted on synthetic samples formed by mica and quartz, at conditions prone to induce ductile deformation in both minerals involved without melting. The deformation experiments were carried out in non-coaxial shear deformation geometry, in order to attain strain large enough for steady-state microstructures to be achieved and extrapolation to natural conditions to be possible. Equivalent hydrostatic experiments were performed for comparison. The micas used were chosen with a composition such as to avoid melting during deformation.

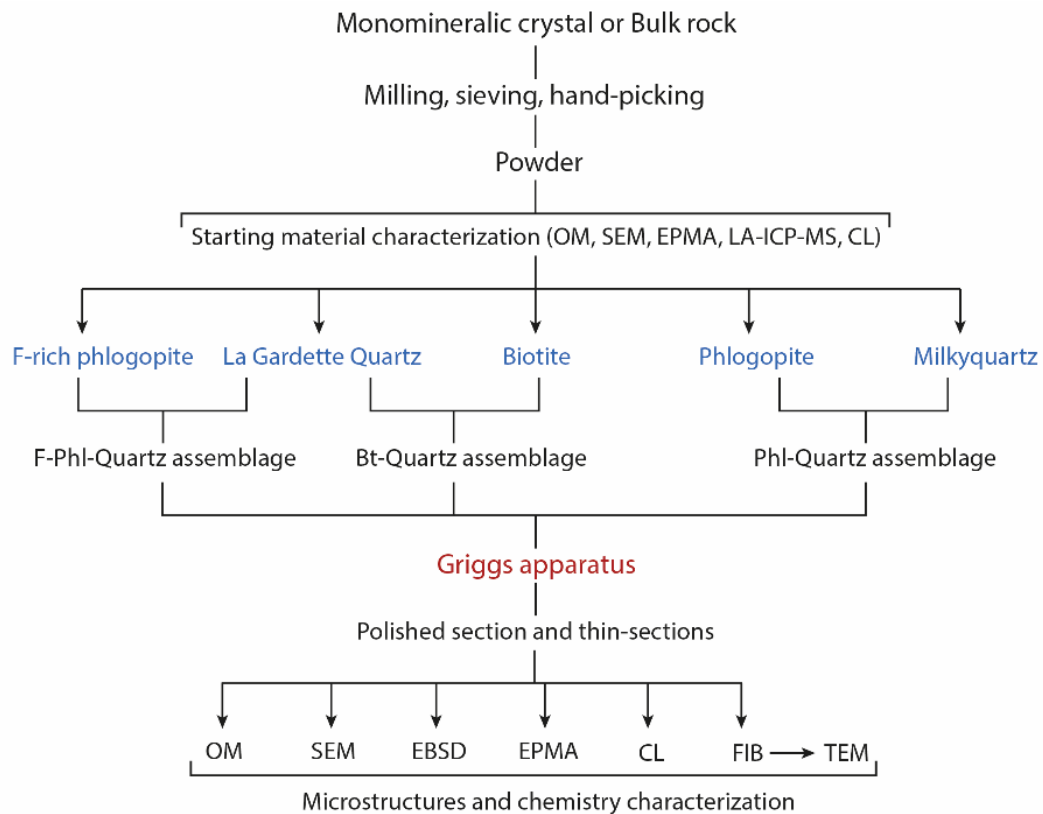


Figure 1 : Diagram summarizing the scientific analytical approach. Starting material is in blue, experimental part in red. OM: optical microscopy, SEM: scanning electron microscopy, EPMA: electron probe micro analyzer, LA-ICP-MS: laser ablation inductively coupled plasma mass spectrometry, CL: cathodoluminescence, EBSD: electron back-scattered diffraction, FIB: focused ion beam, TEM: transmission electron microscopy.

III.1. Aims and approach

Quartz crystal used were chosen to be as close as possibly to the typical quartz of upper and middle crust shear zones. First step was sample preparation with starting material characterization (Fig. 1), then deformation experiments (exploratory ones first in order to constrain experimental conditions, well-constrained ones, second) and microstructural and compositional study of mineral phases of these experiments. In order to study the microstructures resulting from weakening or strain localization, the samples were analyzed using SEM, TEM and the EBSD. Compositional changes were analyzed in order to detect the role of chemical processes as solution transfers, with the EPMA, TEM and CL. All these methods are presented below.

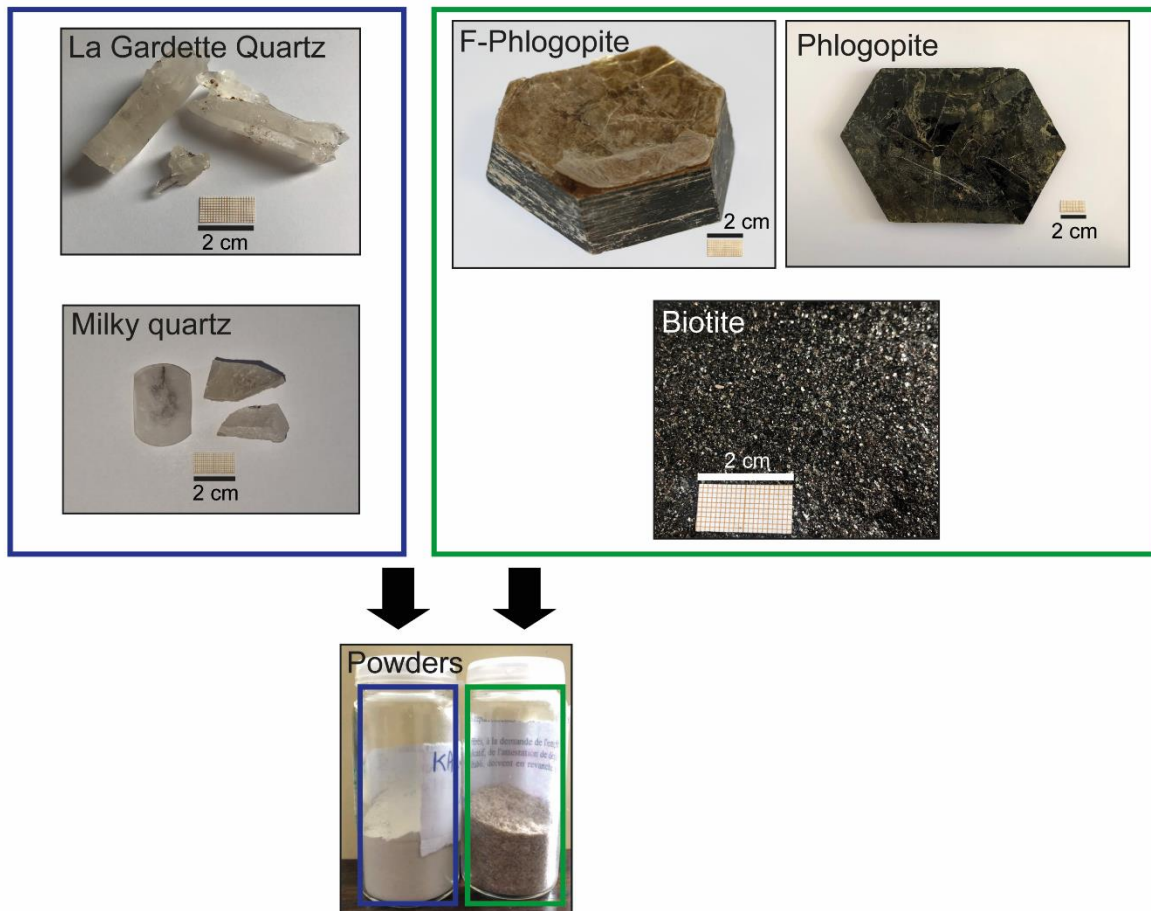


Figure 2 : Starting material from bulk to powder. Blue rectangles for quartz and green rectangles for mica.

III.1. Aims and approach

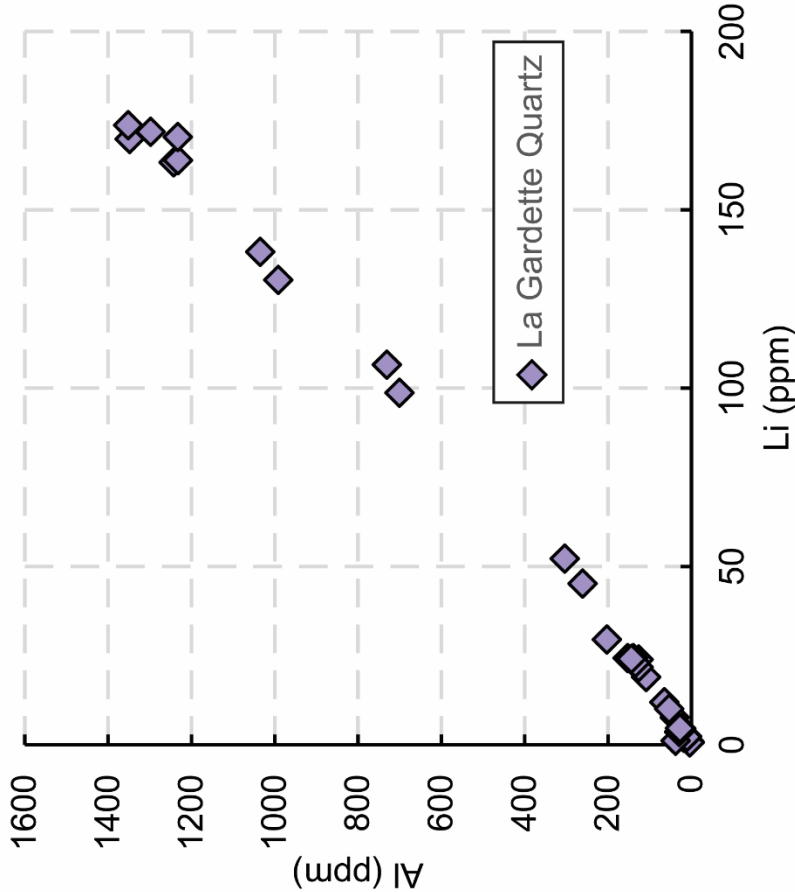


Figure 3 : Li-Al binary plot in ppm of La Gardette Quartz using LA-ICP-MS.

Sample	Li		Al	
	ppm	Sigma Error	ppm	Sigma Error
GF	163.31	5.36	1242.85	39.86
	106.58	3.51	730.96	23.46
	169.86	5.59	1348.24	43.29
	173.68	5.72	1351.62	43.42
	163.89	5.40	1231.92	39.60
	171.85	5.67	1297.7	41.75
	130.3	4.30	991.29	31.93
	138.17	4.57	1034.83	33.37
	45.17	1.50	260.86	8.43
	52.21	1.74	303.55	9.82
	1.607	0.069	11.04	0.37
	0.942	0.067	4.75	0.21
GT	0.825	0.070	5.04	0.22
	0.798	0.048	4.18	0.16
	0.657	0.053	3.77	0.17
	1.927	0.083	17.7	0.60
	2.501	0.097	23.95	0.80
	2.297	0.098	9.92	0.35
	2.18	0.10	10.77	0.39
	2.237	0.095	10.83	0.38
	18.96	0.70	108.23	3.67
	24.29	0.87	152.58	5.13
	23.88	0.84	126.18	4.23
	21.74	0.76	126.53	4.25
GA	1.113	0.081	37.53	1.30
	3.58	0.14	29.17	1.00
	24.31	0.86	139.08	4.72
	29.53	1.10	202.74	6.96
	98.69	3.51	700.54	23.88
	170.41	6.09	1232.66	42.16
	7.67	0.28	39.75	1.37
	4.59	0.18	24.43	0.86
	4.48	0.17	24.28	0.86
	11.92	0.44	64.48	2.25
	10.09	0.38	53.17	1.86
	4.62	0.18	27.3	0.97

Table 1 : Trace element compositions in Li and Al in ppm of La Gardette Quartz using LA-ICP-MS. GA, GT and GF correspond to three distinct veins which are interpreted as a result of a similar episode of fracturation and crystallization.

III.2. Sample preparation: from starting material to mixture powder

Two types of quartz were used (Fig. 2): (1) an Al-Li rich quartz (100-200 ppm of Li, Fig. 2, Table 1) chosen for its Li content, indeed this Li enrichment causes its homogeneous luminescence and was used to discriminate inherited grains from newly formed grains. This quartz is from La Gardette mine (Oisans, French Alps) formed at 330-400°C (Poty, 1967) studied in several papers (Weiss, 1829; Poty, 1967; Marignac et al., 1967; Boutoux et al., 2010), and (2) a single crystal of a milky quartz (Fig. 3, Table 2) formed in a hydrothermal vein of the Aar Massif (Swiss Alps) at ~250°C, used in previous deformation experimental studies (e.g. Tarantola et al., 2010; Thust, 2014; Stünitz et al., 2017; Richter et al., 2018).

In order to characterize quartz composition of the starting material, we carried out our analyses on double-polished thick sections (~150–200 µm) of vein material, which were, prior to ablation, removed from the glass slide by immersion for more than 24h in acetone and then carefully cleaned with acetone for remains of glue. The margins of the thick sections were subsequently fixed to a new glass plate with cyanoacrylate glue. On these sections, we restricted our analyses to the domains of clear quartz, i.e., free or with a very low density of fluid inclusions which was easy as we used a clean and homogeneous quartz.

Trace element composition of quartz: LA-ICP-MS analyses

The LA-ICP-MS analyses were conducted at ISTO, using a RESolution-SE 193 nm ArF excimer laser with a S155 ablation cell coupled to an Agilent 8900 QQQ mass spectrometer. The ablation chamber of the laser ablation system was flushed with He (350 mL/min) and Ar (15 L/min), to which further Ar (~0.9 L/min) was added on the way to the ICP-MS. The ICP-MS was tuned on NIST612 to low production rates of ThO and doubly charged ^{42}Ca of <0.15 % and <0.30 %, respectively, and to a U/Th ratio of $100 \pm 1\%$. For the quartz analyses, the laser was operated at a frequency of 10 or 5 Hz, set to deliver a fluence of 5-7 J/cm² on the sample surface, using an analytical spot size 50 µm in diameter. These conditions (especially the lower ablation frequency of 5 Hz) were found to provide good ablation results. Back-ground and ablation signals were acquired for 30 and 40s, respectively. For each spot, the concentrations of ^6Li , ^7Li , ^9Be , ^{11}B , ^{23}Na , ^{27}Al , ^{28}Si , ^{39}K , ^{43}Ca , ^{47}Ti , ^{60}Ni , ^{66}Zn , ^{85}Rb , ^{88}Sr ,

III.2. Sample preparation: from starting material to mixture powder

^{89}Y , ^{133}Cs , ^{137}Ba were determined using dwell times of 50ms for each isotope. Before and after each set of ~10–12 sample analyses, three analyses of NIST610 and two analyses of NIST612 have been performed. NIST610 was used as an external standard for calibration of all acquired data. SiO_2 was used as an internal standard following the assumption that each point contains close to 100 wt. %. NIST 612 was analyzed for quality control, which showed that accuracy and precision were $\leq 2\text{--}5\%$. For data reduction, we used the software GLITTER™ (van Achterbergh et al., 2001), where we have rigorously inspected and manually selected background and ablation signals, applying a linear fit to all standard data. Each signal of point analysis was carefully checked to remove any anomalies from fluid inclusions or mineral inclusions choosing the best part of spectra representative of quartz.

Quartz crushing and grain size selection

First, La Gardette quartz crystals were cleaned with dithionite to eliminate iron oxides. Both quartz types were crushed using a ring mill (two metal rings). The milky quartz was also grinded a second time using an agate ball mill to obtain finer grain size (mean $6.351\text{ }\mu\text{m}$, Fig. 5). The finer grain size of the milky quartz was measured using laser diffraction particle size analyzers with a Beckman Coulter LS 13320 in at the University of Tromsø. Then, quartz was grinded using an alumina mortar and grain size was finally sorted by using distilled water columns (Fig. 6), to obtain a grain size of 10–20 μm . This technique is based on the principle of the sedimentation rate, indeed, with the Navier-Stokes equation it is possible to compute the travel time of particles, of a given size, over a specified distance, considering various factors such as the density of the fluid and the starting material. The grain size obtained was verified using laser X-ray particle size analyzers with SEDIGRAPH 5100 in the Bureau de Recherches Géologiques et Minières (BRGM, Orléans; Fig. 7, Table 4).

III.2. Sample preparation: from starting material to mixture powder

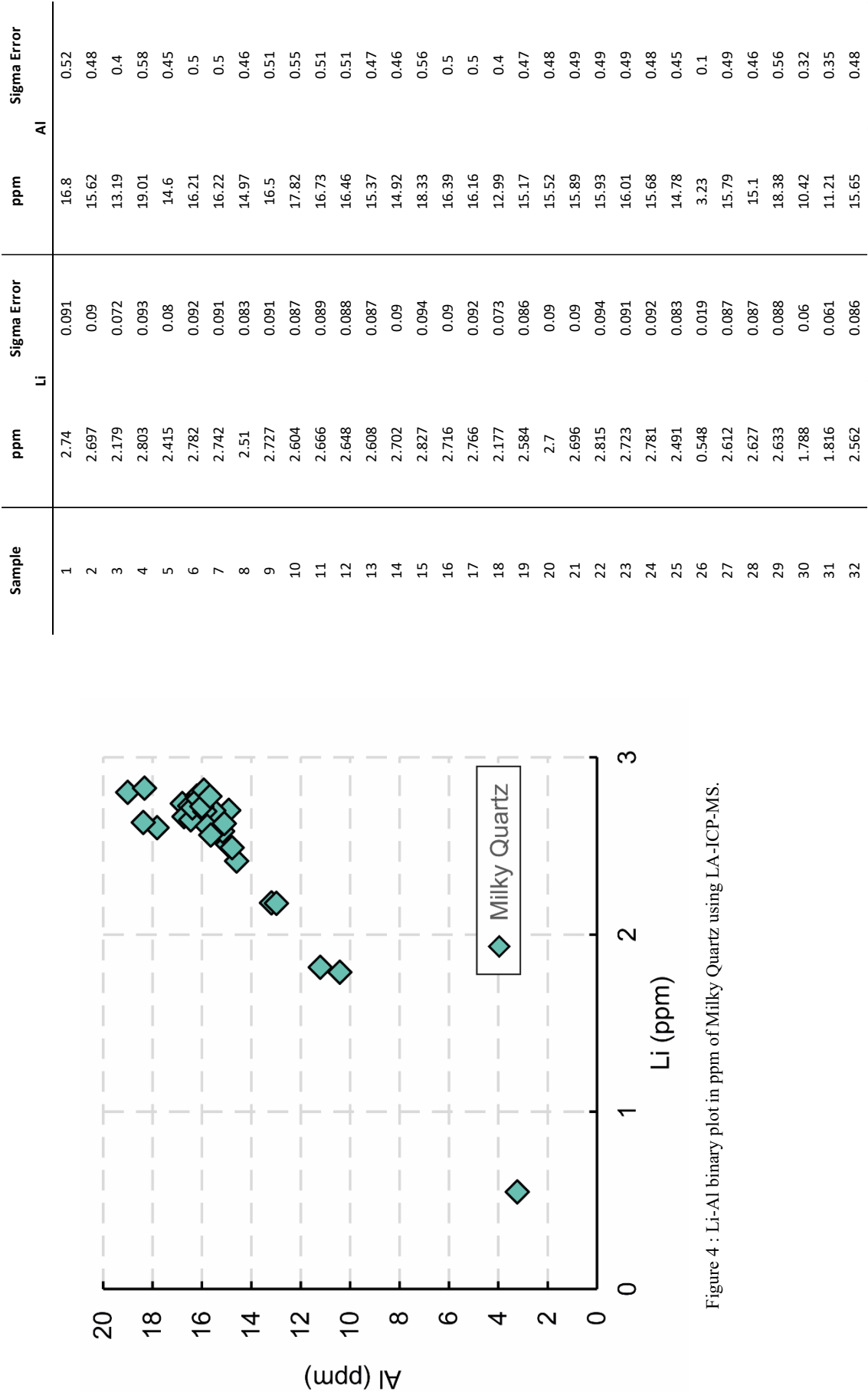


Figure 4 : Li-Al binary plot in ppm of Milky Quartz using LA-ICP-MS.

Table 2 : Trace element compositions in Li and Al in ppm of Milky Quartz using LA-ICP-MS.

Quartz crushing and grain size selection

First, La Gardette quartz crystals were cleaned with dithionite to eliminate iron oxides. Both quartz types were crushed using a ring mill (two metal rings). The milky quartz was also grinded a second time using an agate ball mill to obtain finer grain size (mean 6.351 μm , Fig. 5). The finer grain size of the milky quartz was measured using laser diffraction particle size analyzers with a Beckman Coulter LS 13320 in at the University of Tromsø. Then, quartz was grinded using an alumina mortar and grain size was finally sorted by using distilled water columns (Fig. 6), to obtain a grain size of 10-20 μm . This technique is based on the principle of the sedimentation rate, indeed, with the Navier-Stokes equation it is possible to compute the travel time of particles, of a given size, over a specified distance, considering various factors such as the density of the fluid and the starting material. The grain size obtained was verified using laser X-ray particle size analyzers with SEDIGRAPH 5100 in the Bureau de Recherches Géologiques et Minières (BRGM, Orléans; Fig. 7, Table 4).

Mica composition

Three different mica compositions were used throughout this project (Fig. 2), (1) a F-rich phlogopite monomineralic crystal, (2) a phlogopite monomineralic crystal and (3) a biotite from a standard granite used by [Fauconnier \(2016\)](#). The composition of the micas is presented in Table 3, with respective average content in Si, Ti, Al, Cr, Fe, Mn, Mg, Ca, Na, K as the following: in F-phlogopite, 2.93, 0.03, 1.12, 0, 0.14, 0, 2.75, 0, 0.03, 0.99; in biotite, 2.72, 0.19, 1.60, 0.01, 1.29, 0.02, 0.98, 0, 0.01, 0.94; in phlogopite: 2.83, 0.06, 1.40, 0, 0.28, 0.01, 2.37, 0, 0.05, 0.85. F-phlogopite and phlogopite sheets were cut using scissors, grinded with an agate mortar and crushed using a small blender. Biotite was separated with a magnet and hand-picked to get rid of the impurities (5% of quartz, 1% of feldspar and 1% of other minerals).

III.2. Sample preparation: from starting material to mixture powder

Finally, all micas were sieved to 125-200 μm , 0-63 μm (F-Phlogopite, Biotite) and 63-125 μm (Phlogopite). Then, the starting assemblage was prepared by mixing a powder made of quartz grains to a powder made of mica grains (Fig. 2). Quartz and mica powder from each set were mixed in acetone as a slurry, using an ultrasonic stirrer to ensure uniform grain size and density distribution (De Ronde et al., 2004). Three different assemblages were prepared: (1) F-phlogopite mixed to La Gardette quartz, (2) biotite mixed to La Gardette quartz, and (3) phlogopite mixed to the milky quartz (Fig. 1). The reasons of this choice of mixing is detailed in subsection III.3.7.

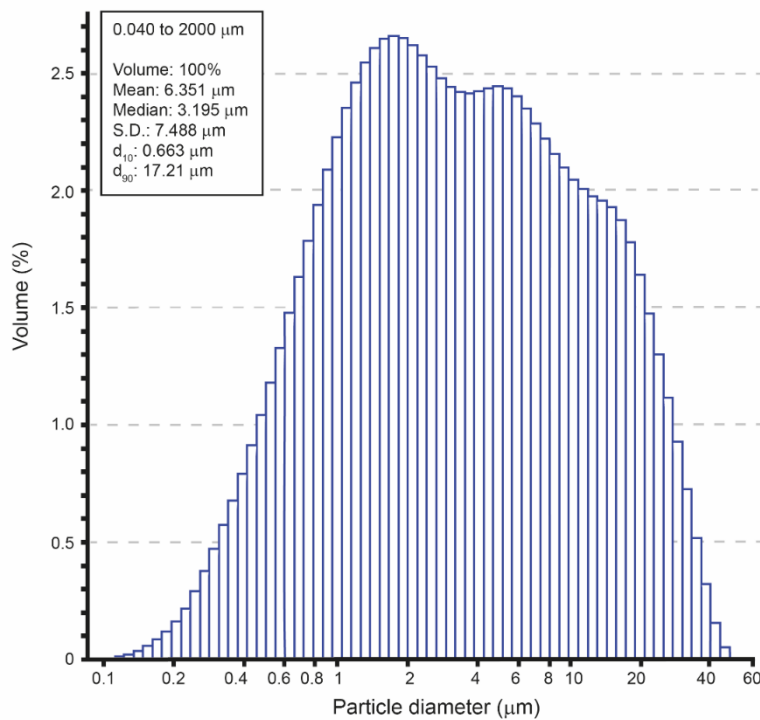


Figure 5 : Grain size distribution of the milky quartz powder after grinding using a ring mill with grain statistics.



Figure 6 : Picture of the distilled water columns used to extract the quartz powders with a grain size range of 10-20 μm .

III.2. Sample preparation: from starting material to mixture powder

Table 3 : Representative chemical compositions of the starting material mica (in wt. % for oxides and in a.p.f.u for number of cations).

Oxide wt. %		N° of cations a.p.f.u.																							
Mica	SiO ₂	TiO ₂	Al ₂ O ₃	Cr ₂ O ₃	FeO	MnO	MgO	CaO	Na ₂ O	K ₂ O	Total	Si	Ti	Al	Cr	Fe	Mn	Mg	Ca	Na	K				
F-Phl	40.74	0.670	13.06	0.000	2.19	0.094	25.73	0.008	0.189	10.43	93.12	2.935	0.036	1.109	0.000	0.132	0.006	2.763	0.001	0.026	0.959				
	40.57	0.478	13.45	0.000	2.32	0.034	25.50	0.001	0.177	10.86	93.39	2.922	0.036	1.142	0.000	0.140	0.002	2.738	0.000	0.025	0.997				
	40.56	0.586	12.87	0.007	2.41	0.000	25.70	0.000	0.230	10.71	93.07	2.932	0.032	1.097	0.000	0.146	0.000	2.771	0.000	0.032	0.988				
	40.21	0.513	13.28	0.000	2.32	0.091	25.10	0.000	0.259	10.83	92.60	2.924	0.038	1.139	0.000	0.141	0.006	2.721	0.000	0.036	1.005				
	40.17	0.626	12.92	0.015	2.32	0.003	25.74	0.000	0.249	10.56	92.60	2.918	0.034	1.106	0.001	0.141	0.000	2.787	0.000	0.035	0.978				
	40.03	0.519	13.21	0.000	2.26	0.000	25.38	0.000	0.205	10.81	92.42	2.916	0.028	1.134	0.000	0.138	0.000	2.756	0.000	0.029	1.005				
	41.16	0.595	13.43	0.009	2.27	0.064	25.55	0.008	0.208	10.86	94.15	2.937	0.032	1.130	0.001	0.135	0.004	2.718	0.001	0.029	0.989				
	40.03	0.616	13.07	0.000	2.13	0.000	25.36	0.000	0.195	10.41	91.81	2.925	0.034	1.126	0.000	0.130	0.000	2.763	0.000	0.028	0.970				
	40.49	0.484	13.19	0.000	2.79	0.067	25.53	0.000	0.204	10.54	93.30	2.922	0.026	1.122	0.000	0.168	0.004	2.747	0.000	0.029	0.970				
	40.80	0.528	13.01	0.000	2.46	0.000	25.32	0.000	0.192	10.77	93.09	2.948	0.029	1.108	0.000	0.149	0.000	2.727	0.000	0.027	0.993				
	40.13	0.626	13.03	0.000	2.46	0.000	25.73	0.000	0.237	10.33	92.55	2.914	0.034	1.115	0.000	0.149	0.000	2.786	0.000	0.033	0.957				
	40.15	0.537	13.03	0.000	2.27	0.033	25.33	0.000	0.206	10.76	92.31	2.927	0.029	1.120	0.000	0.138	0.002	2.753	0.000	0.029	1.000				
	40.66	0.476	13.33	0.000	2.32	0.024	25.31	0.013	0.229	10.77	93.15	2.935	0.026	1.134	0.000	0.140	0.001	2.723	0.001	0.032	0.992				
	40.58	0.609	13.73	0.000	2.52	0.000	25.28	0.000	0.152	10.55	93.42	2.917	0.033	1.163	0.000	0.152	0.000	2.709	0.000	0.021	0.968				
	40.72	0.591	12.91	0.000	2.27	0.000	25.25	0.000	0.211	10.40	92.35	2.956	0.032	1.104	0.000	0.138	0.000	2.732	0.000	0.030	0.963				
Bt	40.60	0.546	13.01	0.026	2.23	0.000	25.46	0.000	0.183	10.70	92.76	2.941	0.030	1.111	0.002	0.135	0.000	2.749	0.000	0.026	0.988				
	39.24	0.703	12.97	0.010	2.34	0.016	25.01	0.000	0.206	10.77	91.26	2.901	0.039	1.130	0.001	0.145	0.001	2.756	0.000	0.029	1.016				
	39.39	0.483	12.90	0.030	2.29	0.071	25.15	0.000	0.200	10.62	91.14	2.912	0.027	1.124	0.002	0.142	0.004	2.772	0.000	0.029	1.002				
	39.57	0.473	13.07	0.015	2.67	0.000	25.19	0.000	0.169	10.65	91.80	2.907	0.026	1.131	0.001	0.164	0.000	2.759	0.000	0.024	0.998				
	39.20	0.471	13.13	0.016	2.17	0.101	25.27	0.000	0.194	10.68	91.23	2.896	0.026	1.143	0.001	0.134	0.006	2.783	0.000	0.028	1.006				
	41.03	0.562	12.98	0.000	2.18	0.119	25.21	0.000	0.204	10.72	93.31	2.953	0.030	1.101	0.000	0.131	0.007	2.737	0.000	0.028	0.984				
	34.78	3.16	18.94	0.1	18	0.34	7.81	0	0.06	9.65	92.84	2.711	0.185	1.740	0.006	0.173	0.022	0.908	0.000	0.009	0.960				
	35	3.19	19.14	0.04	18.35	0.45	7.79	0	0.09	9.38	93.43	2.709	0.186	1.746	0.002	0.188	0.030	0.899	0.000	0.014	0.926				
	35.31	3.07	17.02	0.18	20.55	0.45	8.72	0	0.03	9.46	94.79	2.732	0.179	1.552	0.011	0.330	0.029	1.006	0.000	0.005	0.934				
	34.95	2.72	16.26	0.09	20.08	0.34	9.17	0	0.05	9.63	93.29	2.750	0.161	1.508	0.006	0.321	0.023	1.076	0.000	0.008	0.967				
	35.65	3.23	16.76	0.09	20.36	0.27	8.47	0.07	0.06	9.47	94.43	2.763	0.188	1.531	0.006	0.1319	0.018	0.978	0.006	0.009	0.936				
	35.73	2.83	16.26	0	20.34	0.32	8.98	0.16	0.06	9.29	93.97	2.782	0.166	1.492	0.000	0.324	0.021	1.042	0.013	0.009	0.923				
	35.1	2.56	18.33	0	18.92	0.31	8.79	0.01	0.05	9.33	93.4	2.726	0.150	1.678	0.000	0.1229	0.020	1.018	0.001	0.008	0.924				
	34.3	5.14	18.96	0.17	20.53	0.14	6.79	0.03	0.19	9.15	95.4	2.629	0.296	1.713	0.010	0.316	0.009	0.776	0.002	0.028	0.895				
	33.51	2.75	15.75	0.09	21.18	0.25	9.08	0.04	0.07	9.54	92.26	2.695	0.166	1.493	0.006	0.1425	0.017	1.089	0.003	0.011	0.979				
35.62	3.04	17.28	0.14	20.5	0.26	9.16	0.06	0.1	9.57	95.73	2.724	0.175	1.558	0.008	0.311	0.017	1.044	0.005	0.015	0.934					
Phl	35.41	1.09	14.82	0.056	4.99	0.296	22.72	0.090	0.629	10.04	90.15	2.703	0.063	1.333	0.003	0.318	0.019	2.585	0.007	0.093	0.978				
	35.69	0.91	14.62	0.000	4.62	0.041	22.13	0.135	0.556	9.83	88.54	2.756	0.053	1.331	0.000	0.298	0.003	2.547	0.011	0.083	0.969				
	37.31	1.00	15.43	0.017	4.83	0.070	22.80	0.012	0.350	10.06	91.88	2.768	0.056	1.349	0.001	0.300	0.004	2.522	0.001	0.050	0.952				
	40.41	1.06	17.14	0.103	4.78	0.051	20.09	0.000	0.279	8.49	92.40	2.915	0.057	1.457	0.006	0.288	0.003	2.160	0.000	0.039	0.782				
	40.69	1.11	17.10	0.035	4.87	0.151	19.96	0.027	0.235	9.81	93.99	2.911	0.060	1.441	0.002	0.291	0.009	2.129	0.002	0.033	0.895				
	41.02	1.07	17.20	0.015	4.32	0.141	19.99	0.000	0.306	8.37	92.42	2.945	0.058	1.455	0.001	0.259	0.009	2.139	0.000	0.043	0.767				
	40.54	1.15	17.98	0.047	4.70	0.150	19.93	0.000	0.249	9.65	94.39	2.881	0.061	1.506	0.003	0.279	0.009	2.111	0.000	0.034	0.874				
	41.09	1.11	17.28	0.038	4.42	0.147	19.43	0.000	0.297	10.05	93.87	2.936	0.060	1.455	0.002	0.264	0.009	2.070	0.000	0.041	0.916				
	40.30	1.11	17.69	0.068	4.44	0.110	18.90	0.010	0.268	8.34	91.25	2.932	0.061	1.517	0.004	0.270	0.007	2.050	0.001	0.038	0.774				
	38.06	1.07	15.68	0.036	5.20	0.159	22.73	0.000	0.393	8.49	92.82	2.770	0.059	1.345	0.002	0.317	0.010	2.574	0.000	0.055	0.789				
	38.51	1.12	15.83	0.054	4.57	0.111	22.79	0.000	0.373	9.92	93.27	2.799	0.061	1.357	0.003	0.278	0.007	2.469	0.000	0.053	0.920				
	38.80	1.01	16.23	0.000	4.68	0.186	23.14	0.000	0.370	8.87	93.28	2.801	0.055	1.381	0.000	0.282	0.011	2.490	0.000	0.052	0.817				
	38.41	1.12	15.96	0.019	4.68	0.226	22.75	0.030	0.321	9.36	92.87	2.797	0.061	1.369	0.001	0.285	0.014	2.469	0.002	0.045	0.870				
	37.99	1.13	16.26	0.023	4.49	0.246	22.78	0.000	0.362	8.49	91.77	2.783	0.062	1.404	0.001	0.275	0.015	2.488	0.000	0.051	0.794				
	37.77	1.12	15.45	0.016	4.87	0.187	23.08	0.000	0.376	9.93	92.80	2.772	0.062	1.336	0.001	0.299	0.012	2.524	0.000	0.053	0.929				
39.97	1.06	17.35	0.056	4.38	0.125	21.95	0.000	0.268	8.97	94.12	2.841	0.057	1.454	0.003	0.260	0.008	2.326	0.000	0.037	0.814					
39.82	1.16	16.86	0.026	4.10	0.080	21.91	0.000	0.263	9.97	94.19	2.844	0.062	1.419	0.001	0.245	0.005	2.333	0.000	0.036	0.909					
39.68	1.07	17.20	0.067	4.45	0.090	21.94	0.000	0.290	8.96	93.73	2.836	0.057	1.448	0.004	0.266	0.005	2.337	0.000	0.040	0.817					
39.94	1.12	16.94	0.021	4.53	0.164	21.47	0.000	0.210	10.25	94.64	2.849	0.060	1.424	0.001	0.270	0.010	2.284	0.000	0.029	0.933					
41.67	1.01	17.39	0.006	4.36	0.255	21.66	0.000	0.377	9.57	96.29	2.897	0.053	1.425	0.000	0.253	0.015	2.245								

III.2. Sample preparation: from starting material to mixture powder

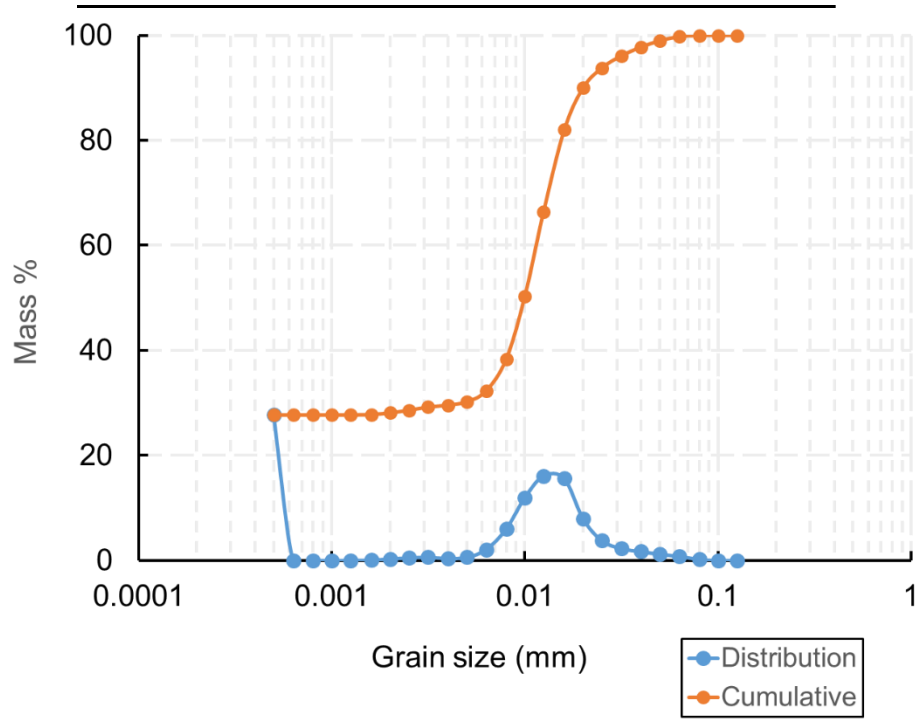


Figure 7 : Grain size distribution curve of La Gardette quartz.

Grain size range (μm)	Grain size Passing mesh (mm)	% distribution	% cumulative
100 to 125	0.125	0	100
80 to 100	0.1	0	100
63 to 80	0.08	0.2	100
50 to 63	0.063	0.8	99.8
40 to 50	0.05	1.2	99
31.5 to 40	0.04	1.7	97.8
25 to 31.5	0.0315	2.3	96.1
20 to 25	0.025	3.8	93.8
16 to 20	0.02	8	90
12.5 to 16	0.016	15.6	82
10 to 12.5	0.0125	16.1	66.4
8 to 10	0.01	12	50.3
6.3 to 8	0.008	6	38.3
5 to 6.3	0.0063	2.1	32.3
4 to 5	0.005	0.6	30.2
3.15 to 4	0.004	0.4	29.6
2.5 to 3.15	0.00315	0.6	29.2
2 to 2.5	0.0025	0.5	28.6
1.6 to 2	0.002	0.3	28.1
1.25 to 1.6	0.0016	0.1	27.8
1 to 1.25	0.00125	0	27.7
0.8 to 1	0.001	0	27.7
0.63 to 0.8	0.0008	0	27.7
0.5 to 0.63	0.00063	0	27.7
< 0.5	0.0005	27.7	27.7

Table 4 : Grain size distribution details of La Gardette quartz.

III.3. Experimental procedure: solid-medium Griggs apparatus

III.3.1. Technical characteristics of the apparatus and procedure

The Griggs-type apparatus is a solid confining medium piston-cylinder apparatus developed in the 60's by David T. Griggs and then improved through the years (Griggs, 1967; Green and Borch, 1989; Tullis and Tullis, 1986). The Griggs apparatus allows reproducing deformation observed in natural strained fields up to high pressure ($< 4\text{GPa}$) and temperature ($< 1200^{\circ}\text{C}$) for various types of samples. The confining pressure is applied through a low-viscosity solid media such as NaCl or KI salts. Simple or pure shear can be applied to samples by creating a differential stress generated by a vertical piston. The tri-axial apparatus consists in a 3-horizontal-platens connected by four vertical columns (Fig. 8). The main hydraulic cylinder, which consists in the confining pressure ram, is held by the medium platen where the deformation ram is supported by the upper platen. These rams are both connected to respective pistons, i.e. σ_3 -piston and σ_1 -piston respectively, the confining pressure piston and the deformation piston made in tungsten carbide (WC) and activated by hydraulic ram. On the lower platen, the pressure vessel, containing the sample assembly and the base plate, is set down (Fig. 8, 9g, h). The sample assembly requires a specific manufactured preparation and will be detailed further. σ_3 -piston and σ_1 -piston are inserted in the end-load piston, set on the pressure vessel and serve to transfer the force between the confining and deformation rams and the WC pistons (Fig. 10a to c). While heating during experiments (through the furnace), the cooling of the apparatus is insured through a water flow in the base plate and the pressure. Three Griggs apparatuses have been used during this work: two “Tullis-modified” press recently arrived at ISTO, Orléans (France) from the University of Tromsø (Norway) and a new generation “Sanchez” apparatus installed at ISTO, Orléans (France). Differences between these two types of apparatus correspond mainly to dimensions of the sample assembly ($\sim 6\text{ mm}$ large for Tullis-modified, $\sim 8\text{ mm}$ for Sanchez) and the recording of the data during the experiments (Tullis-modified rigs have a chart recorder and a computer recorder, Sanchez rig has only computer recording). Most of the experiments have been run using the “Tullis-modified” Griggs apparatus, therefore the following sections will describe specifically the procedure using this apparatus. The two “Tullis-modified” Griggs apparatuses used, Rig 1 (Fig. 8b and 9a) and Rig 2 (Fig. 8a and 9d) have a confining

III.3. Experimental procedure: solid-medium Griggs apparatus

pressure ram controlled by a hydraulic oil pump system, and a load ram controlled by a servo-controlled motor. The load system can either be run in a constant displacement rate or in a constant load setup. Temperatures during pumping and experimental runs are automatically controlled via a Eurotherm proportional-integral-derivative (PID) controller (Fig. 9f). During the experiment, the confining pressure (P_c), vertical displacement of the load piston, and axial load are recorded simultaneously. These parameters are recorded by an analogue chart recorder (sampling frequency 50 Hz), and by a digital data-logging system (LabView and WinDaq) with a sampling frequency of 1 Hz. Two different measurement systems are used for the vertical displacement of the load piston: (1) a direct current displacement transducer (DC-DT) with a resolution of approximately $1\text{ }\mu\text{m}$ and, (2) a noiseless digital linear transformation measurement (DLT) system with a resolution of $0.1\text{ }\mu\text{m}$ (Fig. 8a, b). The confining pressure (P_c) is measured using the oil pressure in the hydraulic pumping system, while the axial load is measured using an external load cell (Fig. 8, 9a, d). More detailed descriptions of the “Tullis-modified” Griggs are available in [Pec et al. 2012](#) and in the PhD theses of [Richter \(2017\)](#), [Marti \(2017\)](#) and [Pec \(2014\)](#).

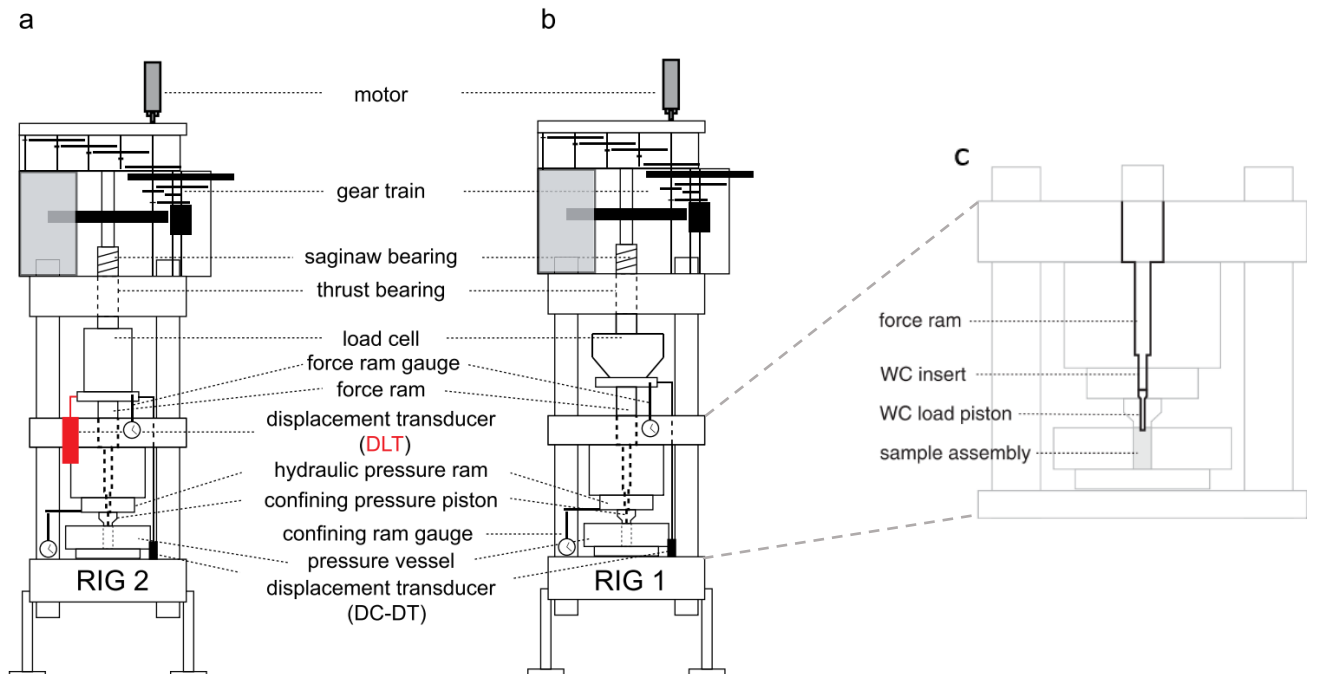


Figure 8 : “Tullis-modified” Griggs-type deformation apparatuses sketches. a) Rig 2 b) Rig 1 and c) Zoom in the assembly sitting part (after [Pec, 2014](#), modified from [Holyoke and Kronenberg, 2010](#)).

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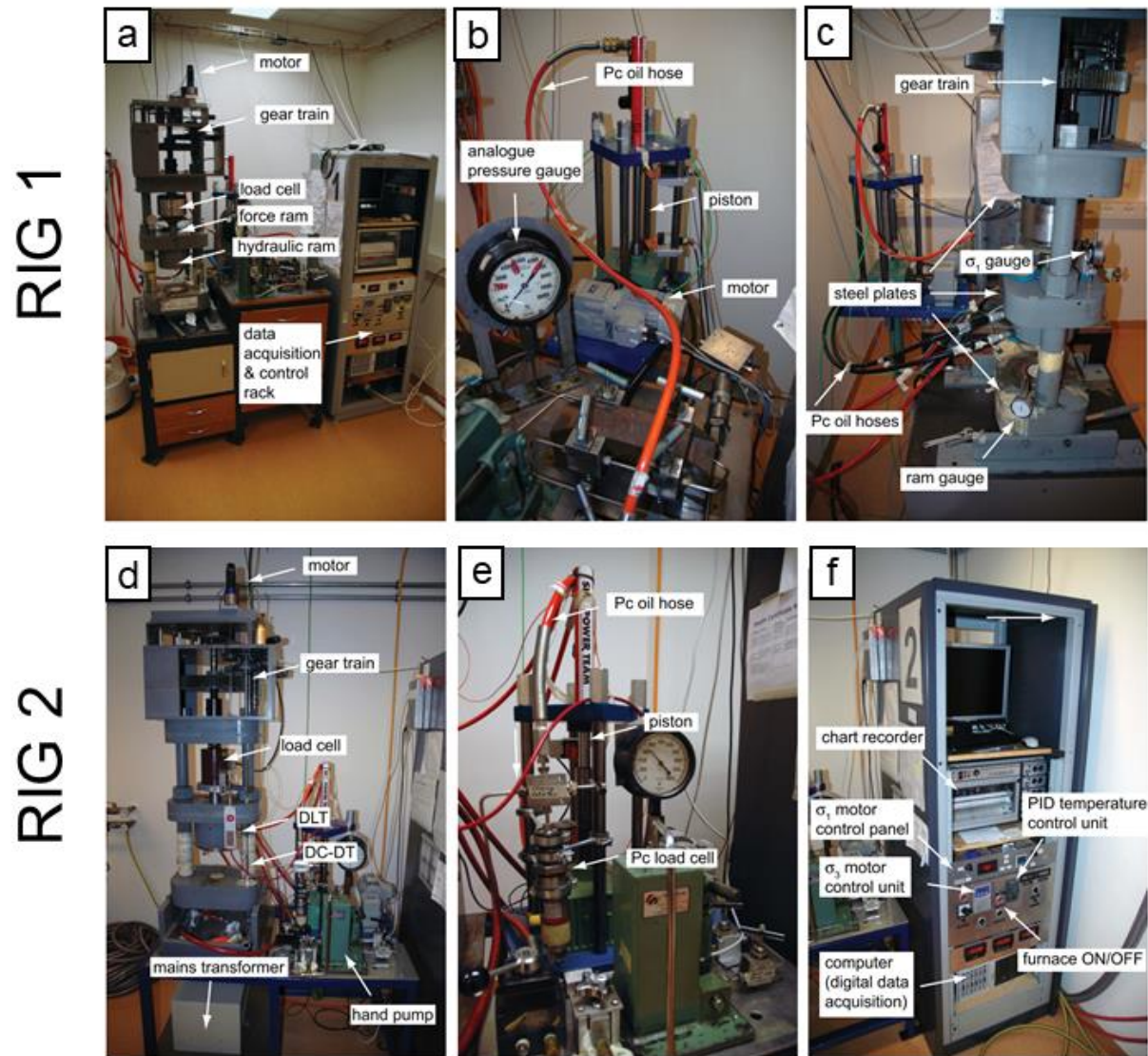
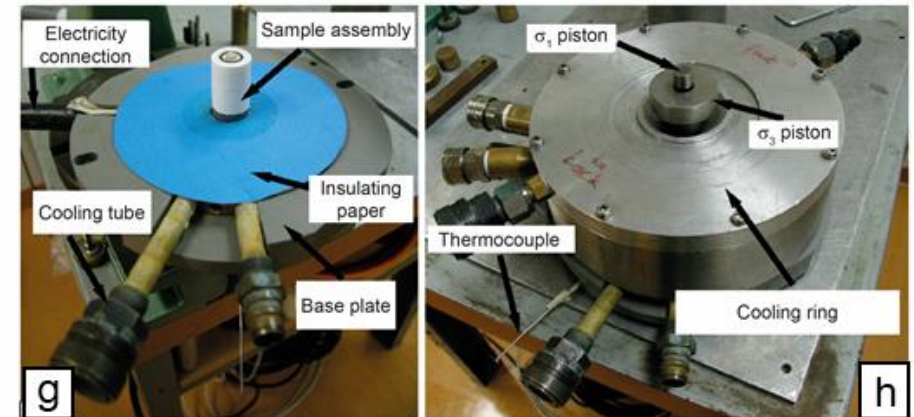


Figure 9 : a) front view of Rig 1 d) of Rig 2, b) automatic pump system of Rig 1, e) of Rig 2, c) back view of Rig 1, f) data acquisition and control rack of Rig 2, (after [Pec, 2014](#)) g) Set up of the sample assembly in the base plate, h) Confining cell fully arranged and ready to be set up below the rig (modified from [Fauconnier, 2016](#)).



III.3.2. Sample assembly

The sample assembly is composed of several pieces that needs a specific manufacturing before each experiment (Fig. 10). The assembly core, placed in a welded Pt-jacket, is composed of two shear pistons including a sample powder and a wrapped Nickel foil (Fig. 9d). Before the seal of the shear sample in the Pt-Jacket, the last is annealed at 900°C during 2 hours in a high temperature atmospheric oven. The assembly core is encircled in a solid-medium composed of two layers of very pure salt (NaCl or KI depending on the experimental temperature) specially created thanks to the pressure-solution process of salt when a pressure is applied on weakly humidified salt. The first layer of salt, called “Inner salt pieces”, is directly placed on the contact of the assembly core and followed by the furnace, composed of graphite and pyrophyllite. The furnace and inner salt piece are drilled in order to install the S-type thermocouple, composed of Pt/Pt-Rh wires in a mullite sheath, on the contact of the Pt-jacket.

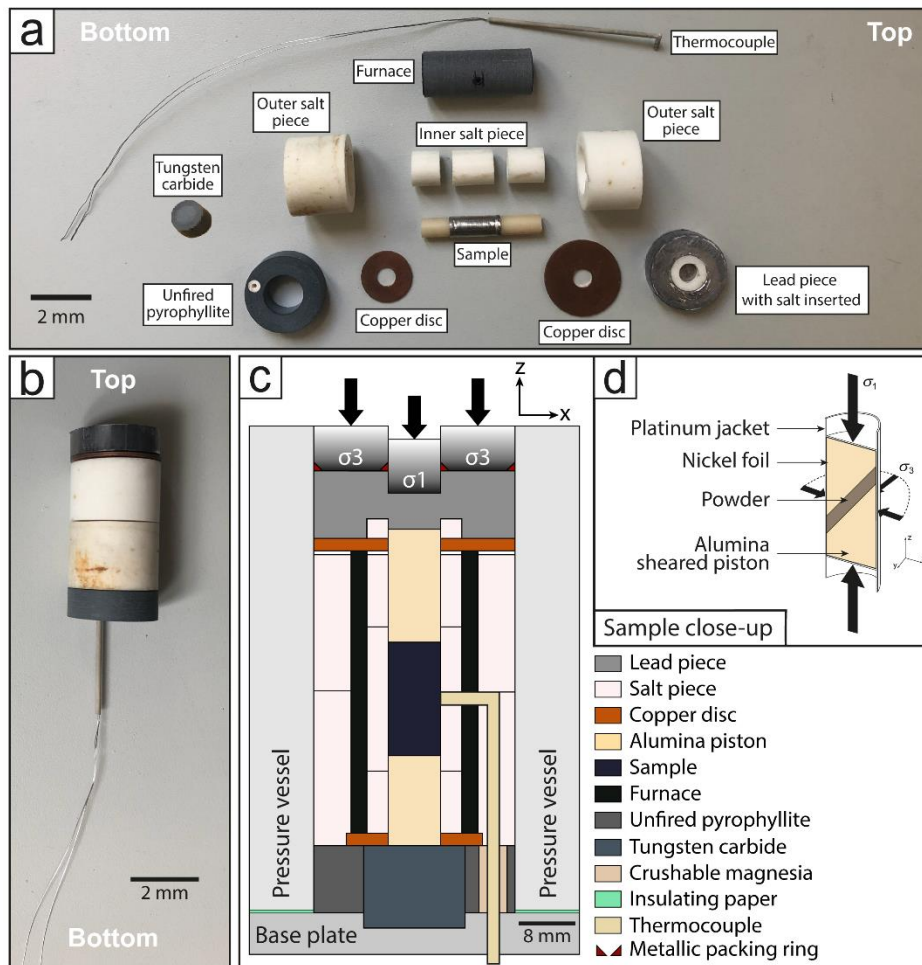


Figure 10 : a) Manufactured pieces of the sample assembly. b) Sample assembly. c) Schematic drawing of the sample assembly in the pressure vessel (modified from Pec et al., 2012; Mansard, 2019). (d) Close-up view on the sample geometry (modified from Pec et al., 2012).

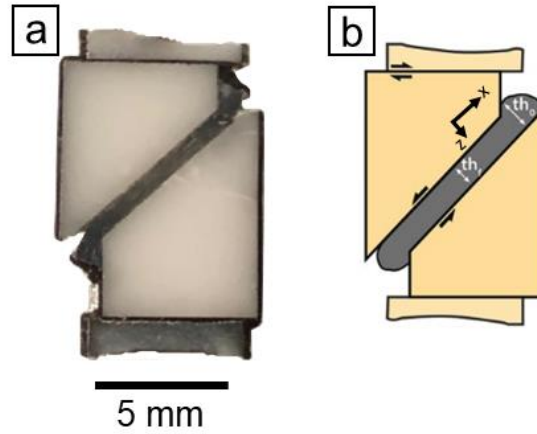


Figure 11 : a) Picture of a deformed sample cut parallel to X-Z plane, b) Sketch of a deformed sample with t_{h0} the initial thickness and t_{hf} the final thickness (after [Mansard, 2019](#)).

A second layer of salt, “Outer salt piece”, constitutes the outermost layer of the sample assembly (Fig. 10a to c). Two copper discs are put in contact on each extremity of the furnace in order to transfer the electric current that allows the heating of the sample assembly. The bottom part of the sample assembly is composed of unfired pyrophyllite drilled and filled by crushable magnesia to hold the thermocouple. A tungsten carbide piece is inserted in the unfired pyrophyllite to ensure the conduction of the current from the base plate to the furnace. On the top of the sample, a lead piece is placed to prevent the deformation of the sample during the pressurization and heating phase (Pumping phase). The lead piece was fabricated with an inner salt ring, which provides better piston guidance especially for high temperature experiments.

III.3.3. Charging the assembly

First step consists in aligning the pressure vessel with the base plate using a hand press. Then the sample assembly fitted together is installed into the base plate while the pressure vessel is suspended to the hand press (Fig. 9g). In order to electrically insulate the pressure vessel from the base plate, an insulating paper is placed around the sample assembly, above the base plate (Fig. 9g). After carefully fitting the pressure vessel to the base plate, the top copper disc following by the lead piece are placed and sealed, on top of the sample assembly, with the σ_3 packing ring (Fig. 10b, c). The cooling ring is set on the pressure vessel with both σ_3 and σ_1 pistons (σ_1 packing ring positioned on the latter) after insulating the thermocouple and bottom part of the base plate. The assembly is completed and positioned in the Griggs

apparatus, pistons must then be aligned with the hydraulic ram. Finally, the thermocouple is plugged in and the cooling system is connected, as well as the electricity. The experiment is then ready to start.

III.3.4. Performing the experiment

An experiment consists of several steps after charging the assembly in the apparatus: (1) the pumping phase, (2) the deformation phase, (3) the quenching phase and (4) the end of the experiment. Each phase is described below.

Pumping phase

During the pumping stage the sample assembly is gradually heated and pressurized (Fig. 13). A constant current is applied to the base plate and conducted through the assembly till reaching the furnace allowing the heating of the sample. Simultaneously, we advance the σ_3 piston alternatively with σ_1 in order to exercise the confining pressure (Fig. 12a), while respecting a maximum distance of 2 mm between these two pistons (Fig. 12a). During the pumping phase, samples were gradually pressurized in steps of 100-200 MPa and then heated by 100°C, at a rate of 20°C per minute. The pumping phase lasted half a day. When the required P-T conditions were reached, a period of hydrostatic hot-pressing overnight (~16 hours) was applied in order to complete the compaction of the sample and reach a stable microstructure.

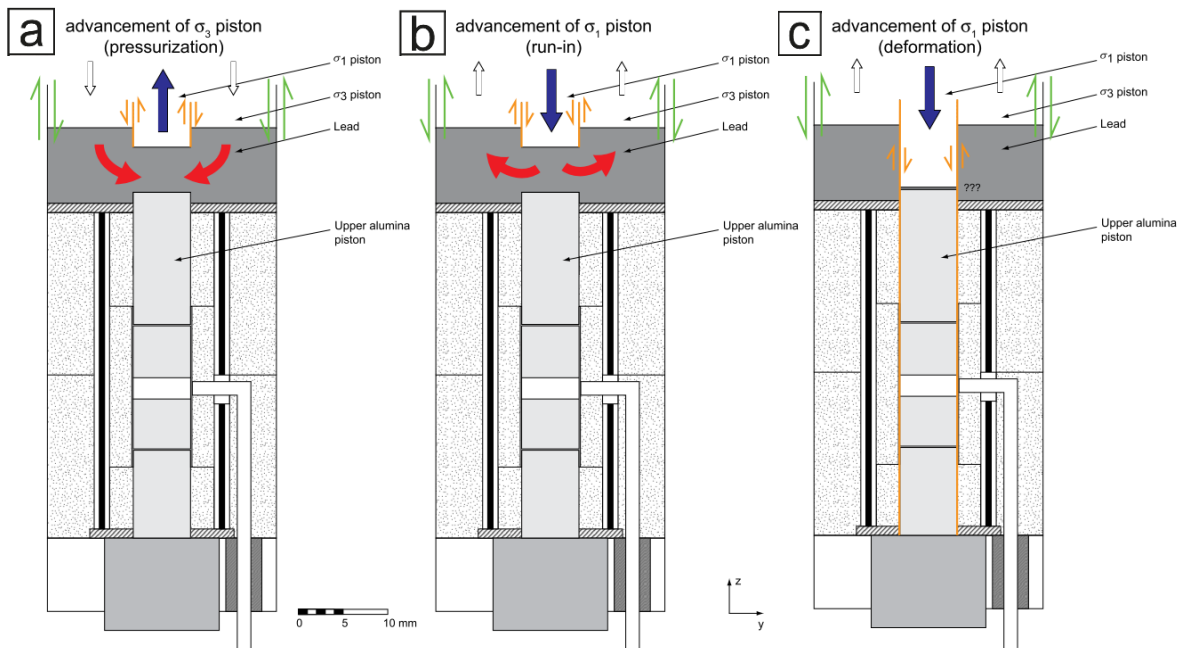


Figure 12 : Forces acting on the pistons and sample during an experiment. a) Pumping phase, b) Pushing through the lead stage, c) after “hit-point”. Green arrows: drag on the pressure vessel, orange arrows: drag on the piston, sample and lead, salt cell interfaces, red arrows: movement of the lead piece (after [Pec, 2014](#) modified after [Tarantola et al., 2010](#)).

Deformation phase

During the deforming phase, the motor of the force ram is activated with a specific speed for constant strain rate and displacement rate experiment. However, another type of experiment possible is with a constant load applied to the sample. Only constant strain rate experiments were performed in this study. The deformation phase consists in two stages (Fig. 12b, c and 13): (1) the friction of the packing rings is observed when the σ_1 piston is passing through the lead piece, (2) the σ_1 piston traverses the entire length of the lead piece and comes into contact with the alumina piston of the sample assembly. Between these two stages is the “hit-point”, exhibiting the start of the sample deformation phase (Fig. 12c). The “hit-point” is defined by curve fitting of the load curve (Fig. 13). Two types of “hit-point” exist (Fig. 13, Marti, 2017): (1) “the classical hit-point” corresponding to the intersection between the gradual and steep slopes; (2) the “lead hit-point” defined by the intersection the lead slope extrapolated and the steep slope. The σ_1 piston is stopped when the amount of strain aimed is reached and the quenching phase is engaged.

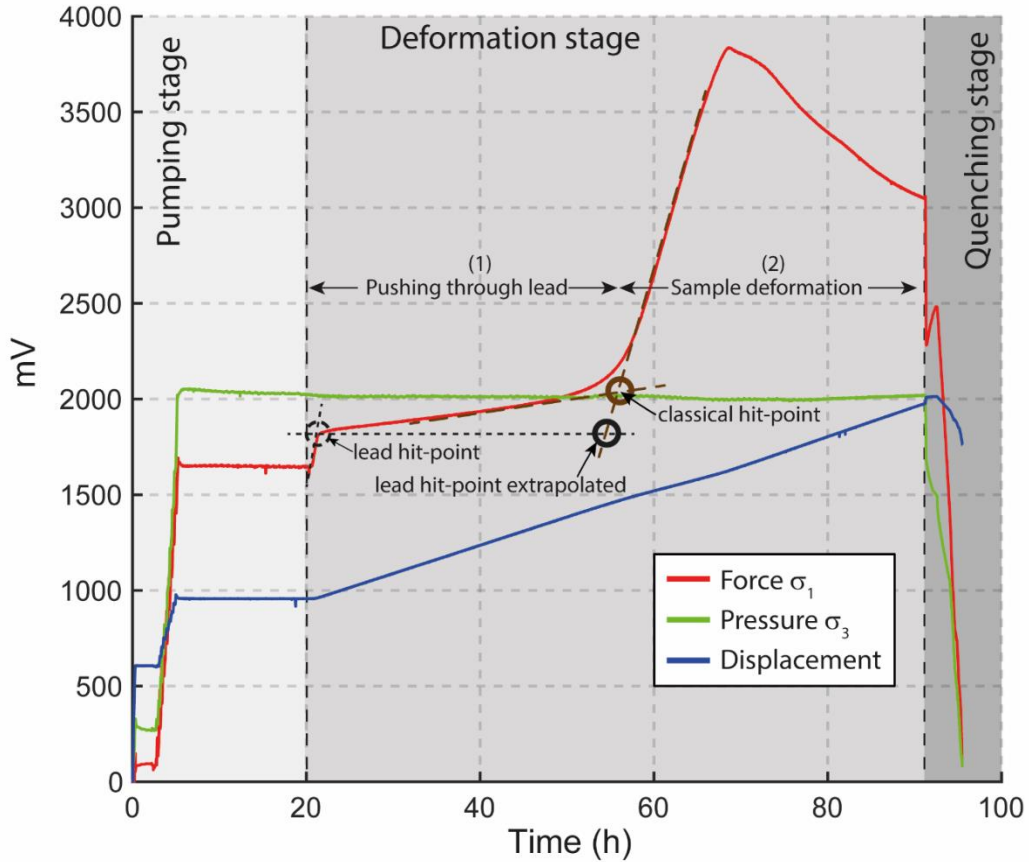


Figure 13 : Chart record of an example experiment, time in hours vs input signal in mV, with in red the force corresponding to advancement of σ_1 the load, deformation ram, in green the confining pressure corresponding to advancement of σ_3 , in blue the vertical displacement of the sample assembly. Classical hit-point corresponds to brown dashed lines and circle. Lead hit-point is indicated by black dashed lines and circles.

Quenching phase and decompression

When σ_1 piston is stopped, a quenching phase (cooling rate of $\sim 150\text{-}300^\circ\text{C}$ per minute) immediately followed the deformation to preserve the microstructures. After dropping the temperature to 200°C , the load on both, σ_1 and σ_3 pistons, is reduced slowly (decompression) while keeping a maximum differential stress between σ_1 and σ_3 of 100 MPa. During the decompression stage the load σ_1 was kept slightly higher than the confining pressure σ_3 to avoid the formation of many unloading cracks. The temperature is decreased to 30°C when $P_c=100$ MPa, and both pistons are pulled out in order to reach 0 MPa simultaneously.

End of experiment

The pressure vessel can be removed from the Griggs apparatus, then the sample assembly is removed from the pressure vessel using a hydraulic press. Salt medium and external layers are carefully extracted, then the orientation of the sample and thermocouple position are marked on the Pt-jacket. After carefully extracting the Pt-jacket, the latter is impregnated in epoxy and cut parallel to in the X-Z plane (the plane perpendicular to foliation and parallel to lineation, Fig. 10a, b) and the shear direction in order to prepare thin-sections and polished sections, for micro-analyses. Fig. 10a, b exhibits the final form of the deformed sample.

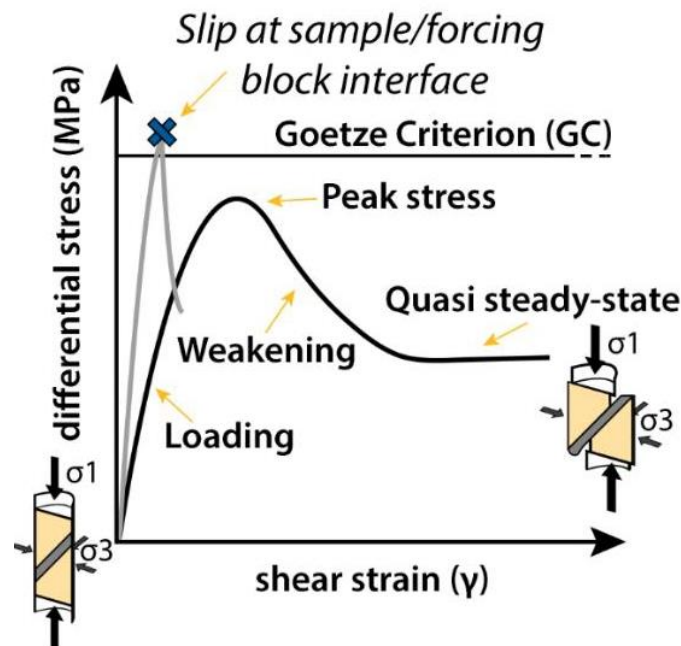


Figure 14 : Terminology used to describe mechanical data as stress-strain curve (Mansard, 2019).

III.3.5. Processing the data

Mechanical data were processed using a MATLAB-based program by Jacques Précigout based on the “rig” program of Matej Pec (<http://mpec.scripts.mit.edu/peclab/software/>) available at <https://www.jacques-precigout.fr/ressources>. The terms used to describe mechanical data as stress-strain curve are illustrated in Fig. 14. A sudden and discontinuous stress and force drop is indicative of slipping at the forcing block interface (cross in Fig. 14), and it can be seen in the sample assembly following the cut. In fact, slip is visible when the sample resides on one side of the sample and has a gap on the other (see number 2 in Fig. 15); in contrast, a sample that is evenly distributed between the forcing blocks denotes no slip (see number 1 in Fig. 15). Observation of slip is also frequently made when the sample deforms above the Goetze criterion. (Mansard, 2019).

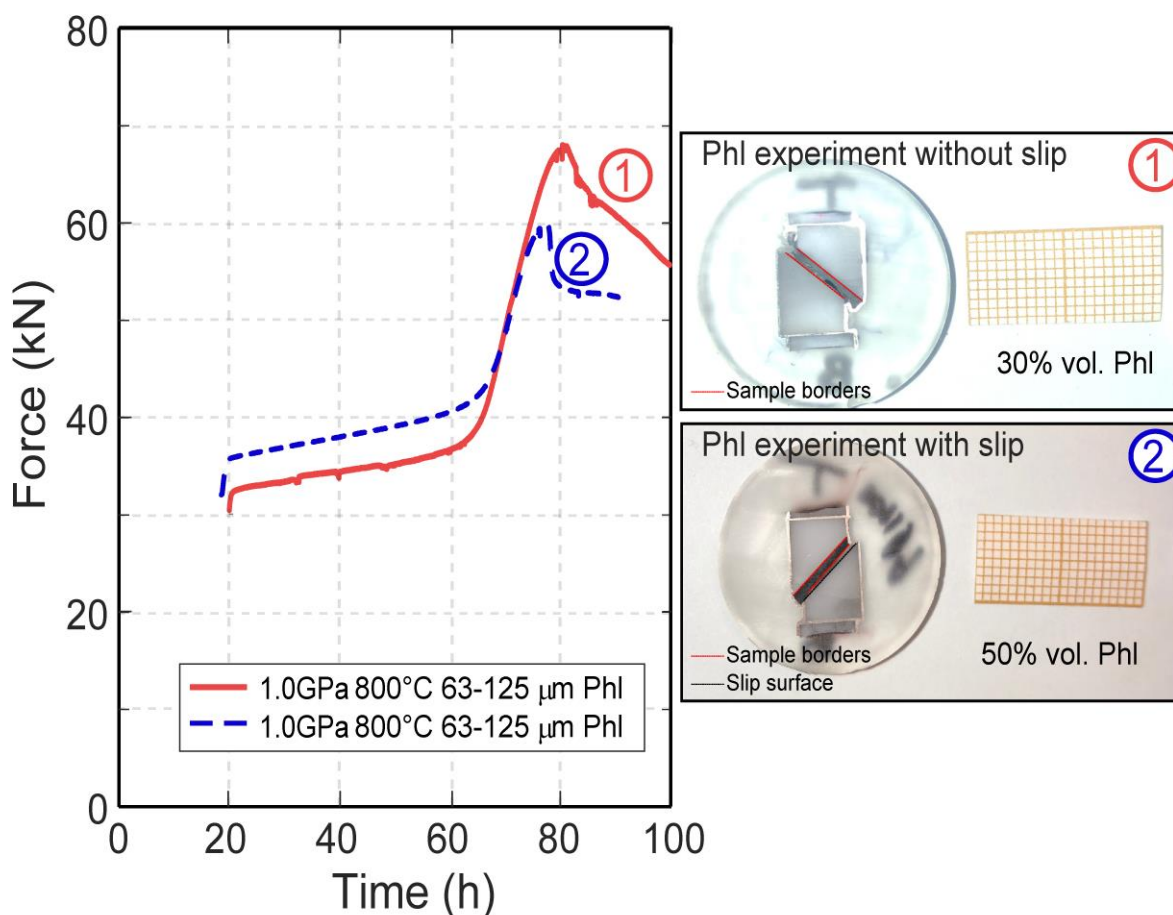


Figure 15 : Force-time evolution for mica-quartz assemblage experiments with corresponding samples cut parallel to X-Z plane, to exhibit slip and no slip.

First step for processing the data is to define the hit-point, we used the “lead hit-point” in all our experiments because fluid inclusions indicate that the load accumulated during “run-in” impacts the sample (Tarantola et al., 2010; Heilbronner et al., 2015). The overall effect of choosing the “lead hit-point” instead of “the classical hit-point” is to decrease the load at a time 0, and thus to increase $\Delta\sigma$ and τ (Fig. 16). As time 0 is advanced, the total shear displacement and consequently the total shear strain are increased. This implies loading added during “run-in” is added to σ_1 thus σ_1 is higher compared to “classical hit-point”.

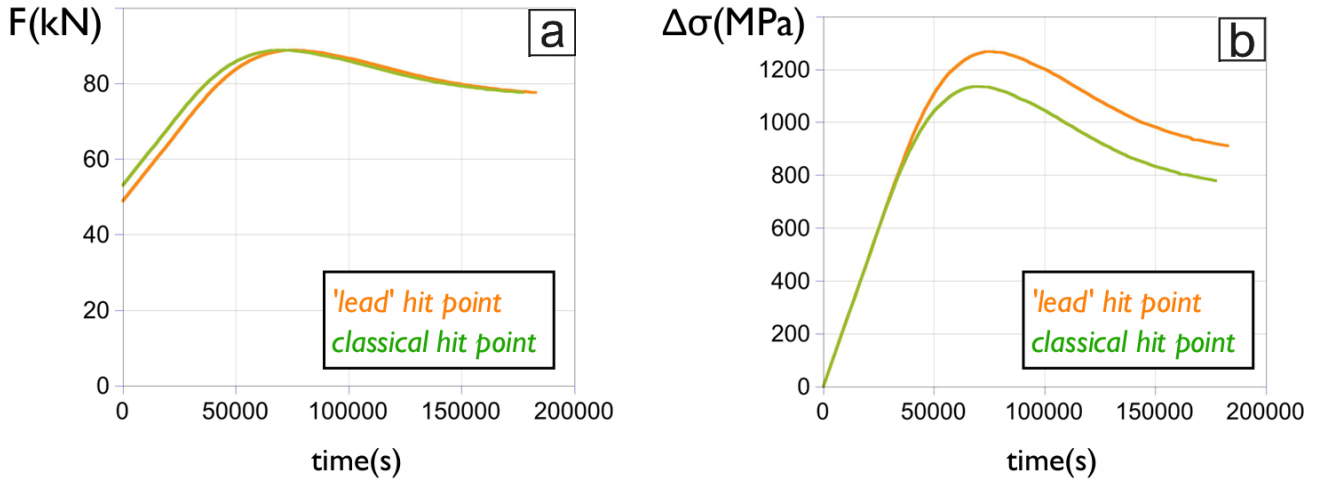


Figure 16 : Example experiments time in seconds vs the force in kN for a) and vs the differential stress in MPa in b), with classical hit point in green curve and lead hit point in orange curve (Heilbronner et al., 2020).

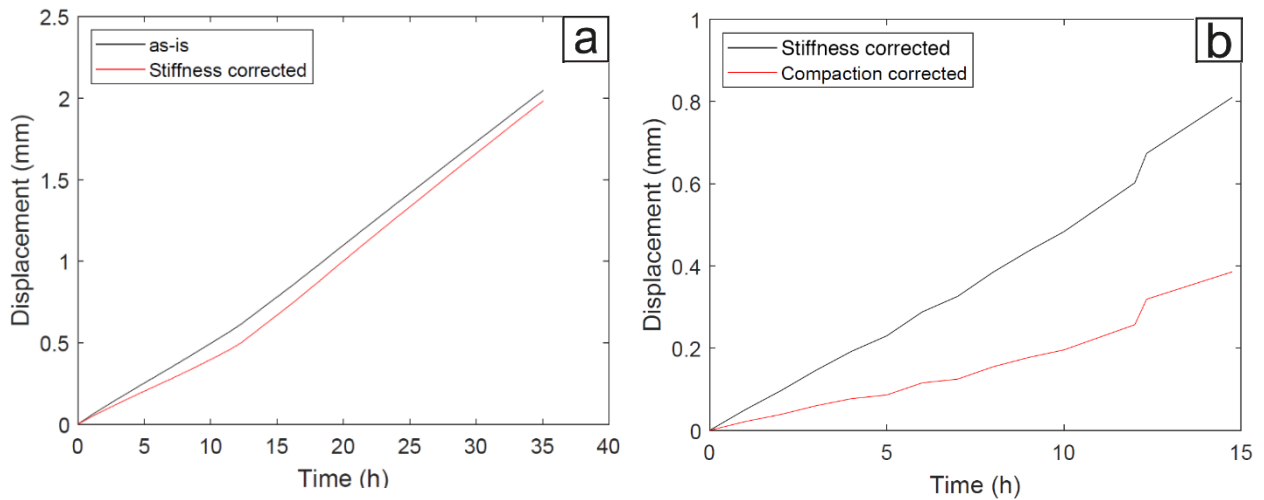


Figure 17 : Example experiment time in hours vs displacement in mm for a) as-is uncorrected data in black curve and stiffness corrected in red, b) stiffness corrected curve in black and compaction corrected showing the overestimation of displacement when applying compaction correction.

After determining the “hit-point”, corrections are applied on the data. The displacement is corrected considering the rig stiffness of each apparatus: Rig 1 stiffness = 3.31 $\mu\text{m/kN}$, Rig 2 stiffness = 5.04 $\mu\text{m/kN}$ (Fig. 17a). For shear experiments, especially the ones using powder material, a compaction correction of the sample is applied to the rig stiffness corrected displacement, however we did not apply this correction as it overestimates the displacement, especially for pure mica experiments. (Fig. 17b). The surface of the sample changes during shear experiments (Fig. 18), and is corrected using the following equation:

$$\text{Shear surface} = 2 \cdot R^2 \cdot \cos^{-1} \left(\frac{d}{2 \cdot R} \right) - d \cdot \sqrt{R^2 - \left(\frac{d}{2} \right)^2}$$

The force associated with the assembly friction is excluded following this equation where the forces are in kN, the displacement in mm and the friction in kN/mm:

$$\text{Force (corrected)} = \text{Force (uncorrected)} - \text{displacement from the hit point} \cdot \text{friction}$$

During the ‘run-in’, the piston moves through lead and the load increases as a function of displacement. One can think of the slope of the run-curve as the base line with respect to which the differential load has to be calculated. The slope was originally attributed to friction between the σ_1 and the σ_3 piston, this correction was called “friction” correction (Heilbronner et al., 2020). Therefore, the force is corrected considering the lead curve extending beyond the “hit-point”, so-called “friction” correction. Based on previous works of Matej Pec and Renée Heilbronner, the coefficient of friction value used is 1.3 kN/mm. The friction correction is used in several studies (Holyoke and Kronenberg, 2010; Pec et al., 2012; Tokle et al., 2023) as it is important to apply to experiments with low differential stresses, if it is not applied it will impact the n-value, and in low differential stress experiments the “friction” correction applies to a higher percentage of the stress compared to samples with higher differential stress. The importance of friction correction lies in accurately determining the differential stress in geological materials using the Griggs apparatus (Holyoke and Kronenberg, 2010). The “friction” correction is still used occasionally to curb apparent strengthening of sample at high strains. However, there is only an empirical basis for this correction – it is not only caused by friction – and in cases where the differential load is small (weak

samples), the resulting load may even become negative (Heilbronner et al., 2020). As there is no current objective work to decide on the use of the “friction” correction, we did not use it.

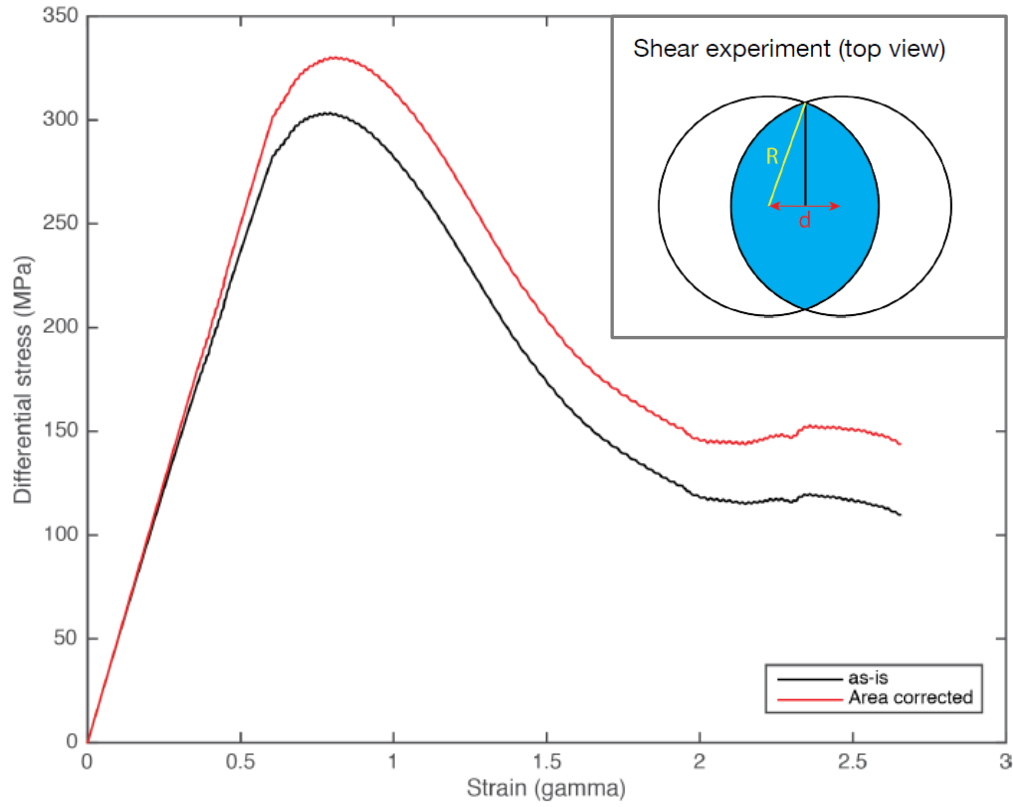


Figure 18 : Strain-stress curve of an example experiment showing the as-is curve in black and area corrected one in red, with on top a sketch of the top view of the shear experiment surface change. R: radius, d: displacement.

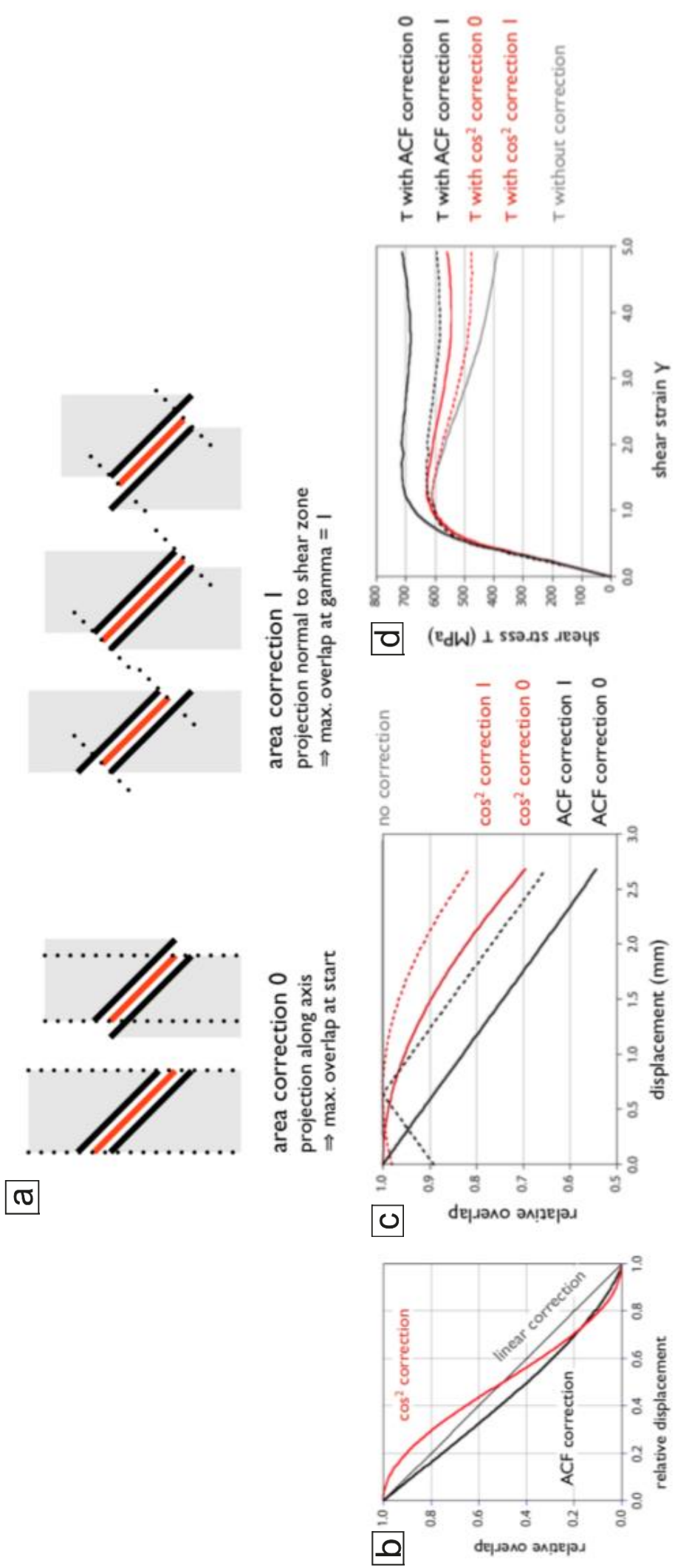


Figure 19: a) Sketch showing the sample geometry with projection along axis and maximum overlap at $\gamma=0$ (area correction 0) and with projection normal to shear zone and maximum overlap at $\gamma=1$ (area correction 1), b) relative displacement vs relative overlap, c) displacement vs shear stress, with the autocorrelated function (ACF) correction in dark curve, $\cos^2$ correction in read curve and no correction (linear correction) in grey curve. Bold curves correspond to area correction 0 and dashed curves correspond to area correction 1 ([Heilbronner et al., 2015](#)).

The area correction applies only to the sample in the 45° pre-cut, as the alumina forcing blocks are assumed to be undeformed. To account for the forcing blocks shifting overlapping areas during the shear experiments, a new area correction procedure was implemented (Fig. 19a, b). The function used to correct the overlap is the autocorrelation function (ACF), however to avoid the discontinuity at $\gamma=1$, the ACF function is approximated by a $\cos^2$ function (Fig. 19a, b, [Marti et al., 2017](#); [Heilbronner et al., 2015, 2020](#)). Calculating the new area correction factor (AC) is as follows:

$$AC = \cos^2\left(\frac{90^\circ \cdot sd}{L}\right)$$

where sd is (the shear displacement parallel to the forcing block) – (the sample interface), L is the maximum possible shear displacement = diameter of forcing blocks/ $\cos(45^\circ)$.

The area corrected differential stress is then calculated as:

$$\Delta\sigma_{AC} = \frac{\Delta\sigma}{AC}$$

The net effect of the area correction is a displacement dependent strengthening (Fig. 19d, [Heilbronner et al., 2015, 2020](#)).

Therefore, the changing overlap of the forcing block interfaces in the sample were corrected for the displacement over the whole period of the sample deformation using the $\cos^2$ area correction (Fig. 19).

All corrections applied in our study are listed in the following recap chart (Table 5).

Correction	Applied?	Why?
Rig stiffness	Yes	Each apparatus has its own stiffness and the displacement must be corrected considering it
Compaction	No	Overestimates the displacement
Surface (area)	Yes	Sample surface changes during shear experiments
Friction	No	Unclear and debated
ACF	Yes	Correct the overlap
$\cos^2$	Yes	Approximating ACF to avoid discontinuity at $\gamma=1$

Table 5: Recap chart of the corrections applied in this study and why.

III.3.6. Limits of the experiments

III.3.6.1. Technical limits

Griggs-type apparatus experiments have limits, especially shear experiments, the main ones encountered are:

- 1) The Pt-jacket can be ruptured leading to losing sample material, cracking can happen mainly when quenching and unloading the experiment (some cracks can also happen during loading). In order to avoid this, it is necessary to really take time on pumping and quenching stages, but depending on the sample strength it is sometimes hard to avoid it even when being careful.
- 2) Touching the sample during the pressurization can happen (as previously mentioned in sub-section III.3.4.) therefore not recording the “hit-point” makes the mechanical data unexploitable, only the microstructures of the sample are exploitable. To avoid this: when advancing σ_1 and σ_3 manually (before 1-1.5 kbar), it is mandatory to advance σ_1 very slowly and stop advancing it the moment when the force value slightly increases.
- 3) Slip at sample or forcing blocks interface making the mechanical data unexploitable (Fig. 14), similarly to extrusion case where the sample is not deformed and the mechanical data recorded another deformation in the assembly (Fig. 15). This is actually difficult to avoid, it can happen even with perfectly parallel and well aligned pistons.

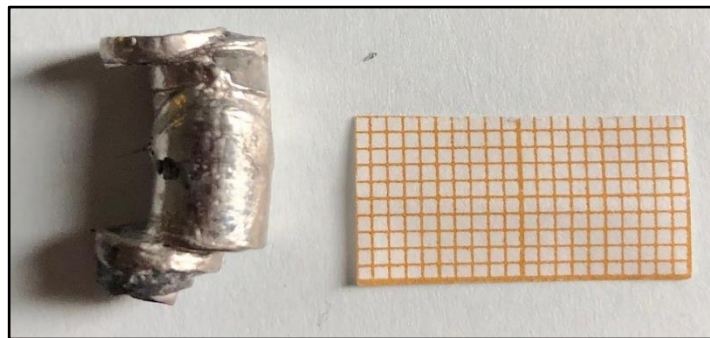


Figure 20 : Example of failed experiment with slip provoking extrusion of the undeformed sample.

- 4) Carbon contamination from furnace by diffusion happens making the samples quite dark after the experiment. Richter (2017) tried to prevent this infiltration by using hematite mixing with the salt. Hematite is reduced to magnetite and will oxidize carbon to carbon dioxide. Richter (2017) pointed out that this buffer was not very efficient.
- 5) NaCl contamination by diffusion is possible in both coaxial and shear experiments (Benjamin Moris-Muttoni and Subhjit Ghosh personal communication). This problem is not systematic but we have no way to prevent it.
- 6) One last contamination therefore possible is Al_2O_3 as well and was observed in olivine experiment (Jacques Précigout personal communication). This can happen but not systematically, and to avoid this possible contamination it is possible to use ZrO_2 pistons.
- 7) Thermocouple break can happen but it is possible to continue the experiment using only the output, however this is only accurate when the output of the current experiment is well-known by previous ones.
- 8) Short-circuit in the assembly can happen and to avoid this it is mandatory to be extremely careful when insulating the thermocouple, and the base plate from the pressure vessel. It is also necessary to often change the insulating paper to keep it efficient.
- 9) A general electrical cut can occur and is inevitable and rare (well it still happened to 3 users in Orléans in the past 3 years).

III.3.6.2. Uncertainties on measurements

Detection limit, accuracy, precision and resolution of the deformation apparatus were addressed by several studies (Borch and Green, 1989; Renner, 1996; Holyoke and Kronenberg, 2010 and Tarantola et al., 2012). Pec (2014) summarized and discussed the probable confidence limits of the apparatus, indicating that the Griggs apparatus is best suited for evaluating strength (or viscosity) contrasts. Furthermore, clear “hit-points” are observed, for strong samples significantly exceeding the “background friction” of the machine, and the interfaces in the load ram ensure accurate force resolution at high loads (Pec, 2014). Conversely, poorly defined “hit-points”, resulting questionable stress-strain curves, are observed for weak samples, the measured stress evolution being concealed by the

deformation of the sample assembly (Pec, 2014). One last uncertainty to know is the precision of stress measurements in experiments with a constant displacement rate which is $\pm 25\text{MPa}$ (Gleason and Tullis, 1995; Chernak and Hirth, 2010; Proctor and Hirth, 2015). The accuracy of the estimation of dynamic friction and drag forces, affecting the σ_1 piston, controls the precision of stress measurements (Soleymani and Kidder, 2021).

Another uncertainty to consider is on the measurement of the temperature. Baïssset, 2023 observed a significant radial temperature gradient between the graphite furnace and the sample. This issue is the cause of both faster wear of the tungsten carbide, which does not withstand high temperatures well, and the melting of the salt when temperatures at samples exceeding 810°C for a pressure of the order of one Gigapascal. In this configuration, the salt begins to convect and becomes conductive, causing current to flow to the platinum capsule, which can melt. The thermal system overheats, and the experiment must be stopped to avoid excessive damage to the deformation cell. Since the temperatures considered in Baïssset, 2023 are close to this overheating point (800°C), it was necessary to design a new assembly to reduce the gradient between the graphite furnace and the sample. Baïssset, 2023 designed a new assembly differing from that described by Moarefvand et al. (2021) by thicker external pieces of salt and pyrophyllite between the graphite furnace and the carbide core. The purpose of these modifications is to increase the temperature gradient between the furnace and the containment chamber to prevent overheating during experiments.

Griggs experiments present uncertainties regarding temperature, caused mainly by the position of the thermocouple which is not directly in contact with the sample. The actual difference in temperature can be measured with the new generation “Sanchez” apparatus which uses the two thermocouples.

III.3.7. Synthesis of the experiments done in this work

Throughout this PhD 28 experiments were carried out including 4 failed experiments linked to some of the limits cited the previous in subsection. A series of 11 exploratory experiments was first performed (Set 1, presented in Chapter VI) in order to constrain conditions (P, T, ϵ , initial powder weight), grain sizes (varied for mica only 125-200 μm , 0-63 μm ; 10-20 μm for quartz) and starting material composition (F-rich phlogopite, intermediate biotite and Al-Li-rich quartz). Experiments from Set 1 were performed at different temperatures (700, 750, 800°C), pressures (10, 15 kb) and strain rates ($3 \cdot 10^{-5}$ to $5 \cdot 10^{-5} \text{ s}^{-1}$). These exploratory experiments (Set 1, presented in Chapter VI) allowed us to constrain the conditions for a series of experiments where phase proportion was varied (Set 2, presented in Chapters IV, V and VI). The latter set consists of 7 experiments performed at a constant bulk shear strain rate (ϵ) of $\sim 10^{-5} \text{ s}^{-1}$, a temperature (T) of 800°C, a confining pressure (P) of 10 kbar and with 0.1% wt. added H_2O , with phlogopite (63-125 μm) and milky quartz (10-20 μm) as starting material. The mixed powder (0.1 g to obtain $\sim 1 \text{ mm}$ thick shear zone at the end of the experiment) was introduced between two alumina pistons of 6.33 mm diameter, pre-cut at 45° (Fig. 10d). The assembly was wrapped around a nickel foil and inserted in a weld-sealed platinum jacket (Fig. 10d). The solid confining medium used was NaCl and the temperature was controlled with an S-type (Pt/Pt-Rh) thermocouple (Fig. 10a to c). Detailed conditions of each experiments are listed in Table 6. The shear strain (γ) is calculated using the total displacement and the final thickness of the sample.

III.3. Experimental procedure: solid-medium Griggs apparatus

Table 6 : Experimental conditions for constant shear strain experiments described in the thesis throughout chapters IV to VI, with only Rig stiffness correction applied on the displacement.

Experiment	Quartz type	Quartz grain size μm	Mica type	Mica grain size μm	Mica proportion		Temperature °C	Confining Pressure		H ₂ O content % wt.	Total displacement stiffness corrected mm	Time		Final thickness mm	Bulk strain rate s ⁻¹	Peak differential stress MPa	Total shear strain γ
					vol.			kbar				hours	seconds				
688KA	Milky	10-20	-	-	0	0%	30	5	0.1	-	-	-	-	-	-	-	-
690KA				-	0	0%				1.60	39.10	140750	1.00	1.75E-05	2172	2.01	
687KA			Phl	63-125	1	100%	800	10		2.06	35.71	128563	0.47	4.82E-05	432	6.20	
672KA					0.7	70%				1.26	23.00	82803	0.98	2.20E-05	637	1.82	
663KA					0.5	50%				1.84	31.95	115020	1.18	1.92E-05	571	2.20	
652KA					0.3	30%				2.00	34.34	123620	0.82	2.79E-05	338	3.45	
684KA					0.3	30%				2.07	36.94	132970	0.62	3.55E-05	1135	4.72	
657KA					0.2	20%				2.00	35.45	127603	0.70	3.17E-05	943	4.05	
659KA					0.1	10%				2.04	36.26	130552	0.83	2.67E-05	1239	3.48	
448BR*					0	0%				2.48	44.65	160729	0.75	2.91E-05	868	4.67	
623KA	La Gardette		F-Phl	125-200	0.3	30%	750	10	0.1	5.35	88.61	319000	0.71	3.35E-05	1341	10.69	
OR85				125-200	0.3	30%	3.22			75.82	272942	0.80	2.09E-05	1090	5.69		
625KA				125-200	0.3	30%	3.20			52.78	190015	0.75	3.17E-05	1137	6.03		
634KA			Bt	0-63	0.3	30%	800			2.51	53.38	192156	0.70	2.64E-05	878	5.07	
627KA				125-200	0.3	30%				2.20	37.28	134193	0.67	3.47E-05	392	4.65	
633KA				0-63	0.3	30%									0.67	3.52E-05	320

* Richter et al., 2018

III.4. Analytical procedure: characterization of samples microstructures and chemistry

III.4.1. Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) was applied to characterize the microstructures at high magnification on polished and carbon-coated (± 20 nm) samples. It can also be used to obtain semi-quantitative information about elements distribution in the minerals in the sample. BSE images have been acquired using a Merlin Compact Zeiss SEM, at 15kV with a working distance of 10 mm at ISTO, Orléans. Another SEM was used, TESCAN MIRA 3 XMU, at BRGM, Orléans at 15 kV with a working distance of 15 mm.

III.4.2. Electron Back-Scattered diffraction (EBSD)

Crystallographic information used to determine deformation mechanisms in quartz was acquired using Electron Back Scattered Diffraction (EBSD). EBSD detector consists of a phosphor screen and a low light condition camera. Following excitation by the SEM column electron gun, certain electrons are diffracted on the planes of the crystal lattice, resulting in the formation of two diffraction cones. These cones are projected as lines known as Kikuchi bands, as a result of the low angle of diffraction and following Bragg's law. By combining multiple diffraction planes, a diffraction diagram exhibiting electron backscatter patterns (EBSP) is formed on the screen. The Hough transform is used to index the EBSP, resulting in Miller indexes. Based on these indexes, the crystal orientation is determined for each measurement point. Initially, the samples were polished using a colloidal silica suspension and then coated with a thin 2 nm carbon layer. Quartz crystallographic orientation was measured using EBSD with an Oxford Instruments Nordlys S detector and on the Zeiss Merlin SEM at the University of Tromsø. Also using an EDAX PEGASUS system (OIM DC 6.4 software) connected to a TESCAN SEM (MIRA3) located at BRGM, Orléans. Crystallographic data were collected using 20 kV accelerating voltage, 70° specimen tilt angle and 15 or 18mm working distance. Detailed maps of ~ 16 mm² were measured with a step size of 0.2 μ m. Data were reduced using the Oxford Instruments Aztec software and the OIM analysis 7.0 software by applying the “cleaning” procedure described by [Précigout et al. \(2017; 2019\)](#), and then processed with the open-source MTEX toolbox for MATLAB (<http://mtex->

toolbox.github.io/). The texture (J; Bunge, 1982) and misorientation (M; Skemer et al., 2005) indices have been calculated to quantify the fabric strength for each map.

III.4.3. Cathodoluminescence (CL)

The CL method relies on the semi-conductivity of specific minerals. When these minerals are excited by an electron gun, they release photons with wavelengths in the visible spectrum. The luminescence is linked to the interaction between an incident electron and the semi-conductive material. When an incident electron transfers a part of its energy to another electron in the valence band of the material, the latter emits a photon in return. The type of material determines the wavelength of the emitted photon, providing insights into the internal structure, mineral growth history, or compositional evolution. CL images have been recorded at BRGM (Orléans, France) with EDAX-Pegasus system equipped with a TESCAN CL-detector (Aurata panchromatic detector with detectable wavelength ranging from 350 to 650 nm, and interchangeable with the Backscatter Electron detector). The conditions of imaging were 5 to 15 kV and 17 to 20 mm working distance. Images were taken with an exposure time between 7 and 15 minutes (depending the desired image quality). Hyperspectral CL (MacRae et al., 2013) images were acquired with xCLent V Pro spectrometer installed in IHP 200F JEOL FEG at MACLE-CVL analytical platform (Orléans), at the following conditions: 15 kV, 100 nA, 150 ms of dwell time and 0.5 or 1 μm step size.

III.4.4. Electron probe micro-analyzer (EPMA)

Electron probe micro-analyzer (EPMA) relies on the same approach employed by the SEM. When the incident electrons, generated by the tungsten head, bombard the sample X-rays are emitted. Each wavelength corresponds to a specific chemical element therefore, the emitted wavelengths are examined using the wavelength dispersive X-ray spectroscopy detector (WDS), which is based on Bragg's law.

EPMA analyses on carbon-coated samples were performed using a CAMECA SX Five and IHP 200F JEOL FEG, at ISTO (Université d'Orléans) and MACLE-CVL analytical platform (Orléans).

- For CAMECA SX-Five microprobe, point measurements for phlogopite were made at 15 kV and 10 nA, with a $\sim 1 \mu\text{m}$ beam size in the WDS mode, using andradite (Ca), albite (Si, Na),

III.4. Analytical procedure: characterization of samples microstructures and chemistry

MnTiO₃ (Mn, Ti), MgO (Mg), hematite (Fe), orthoclase (K), Al₂O₃ (Al) as standards for measuring the respective elements. Element mapping acquisition was done with operating conditions of 15kV, a beam current of 150 nA, 150 ms per pixel for the dwell time and 1 μ m for the step.

- For IHP 200F JEOL FEG, CL images acquisition were coupled with compositional X-ray maps obtained at the same conditions with wavelength and energy dispersive spectroscopy (WDS, EDS). Point measurements for the quartz were done at 15 kV and 5 nA, with a $\sim$ 1 μ m beam size (unfocalized 2 μ m) in the WDS mode, using jadeite (Si, Na), orthoclase (K), MgO (Mg) and Al₂O₃ (Al) as standards for measuring the respective elements.

All compositional X-ray maps, as well as hyperspectral CL maps, were processed with the software XMapsTools 3.4.1 ([Lanari et al., 2014](#)) to obtain element content maps quantified and phase proportion maps.

III.4.5. Nanostructures

III.4.5.1. Focused ion beam (FIB)

Focused ion beam (FIB) allows a micro-machining of the sample surface. Transmission electron microscopy (TEM) requires samples that are thin enough (< 100 nm) to allow electrons passing through. FIB is ideal for locating and micro-machining samples, thanks to its nanometric precision. Using a FIB microscope FEI strata Dual Beam 235 at IEMN (Lille), four ultra-thin foils of 10 μ m $\times$ 5 μ m $\times$ 80 nm in size were cut from three thin sections. First step consists of a milling (rough excavations) from both sides of the sample. This step was done using a 30 kV Gallium liquid metal ion source (LMIS Ga⁺) beam operating at 5 nA. Each pre-cut section was transferred on a TEM copper grid. Then, finer thinning was conducted with a < 100 pA current beam, followed by low-tension cleaning (3 kV) to avoid amorphization of the crystallites.

III.4.5.2. Transmission electron microscopy (TEM)

For nanoscale characterization of microstructures ACCELARM 200 CF TEM/STEM JEOL was used on FIB foils at MACLE-CVL analytical platform (Orléans). Analytical conditions were conducted at a

12 μA current, a tension of 200 kV, leading to a point by point resolution of 1.2 Å and a grid of 0.72 Å for HRTEM (high resolution TEM) mode. This mode allowed the acquisition of bright field and dark field images highlighting defects such as dislocations. Also using HRTEM, selected area electron diffraction (SAED) images, of 1.7 μm to 150 nm of diameter, were used to identify grain orientation after indexation (zone axis). HRSTEM (high resolution scanning TEM) mode was also used with a resolution of 0.8 Å à 200kV, which was constrained to 1.3 Å to 80kV for high-angle annular dark-field imaging (HAADF). This mode was used for elemental chemical mapping and point analysis using an EDX spectrometer. All TEM images (HRTEM and HRSTEM) were processed with the software Gatan Digital Micrograph 3.5.3, 1996–2023 (<https://www.gatan.com>).

Chapter IV – Dissolution-precipitation as
weakening mechanism in experimentally
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IV.1. Introduction

The overall strength of granitic rocks is controlled to a large extent by the rheological properties of feldspar and quartz, which are the main components of the continental crust. Their mechanical properties can vary depending on factors such as syn-tectonic temperature, fluid abundance, and changes in differential stress or strain rate (Stünitz and Tullis, 2001; Stipp et al., 2002; Piazzolo et al., 2002; Menegon et al., 2008; Kilian et al., 2011; Palazzin et al., 2018; Ceccato et al., 2022). In addition, granite deformation is in general accompanied by metamorphic reactions that result in the crystallization of phyllosilicates in shear zones that weaken the rock and localize strain (FitzGerald and Stunitz 1993; Shea and Kronenberg, 1993; Wintsch et al., 1995; Tullis et al., 1996; Wibberley, 1999; Gueydan et al. 2003; Goncalves et al., 2012, 2016; Wehrens et al., 2016; Mansard et al., 2018; Airaghi et al., 2020a; Alaoui et al., 2023). The reasons for such reaction-induced weakening can be schematically decomposed into two distinct types of processes: the intrinsic weakness of phyllosilicates, compared to original quartz and feldspar, and the catalytic effect of phyllosilicates on quartz deformation. (Shea and Kronenberg, 1993; Tullis et al., 1996, Goncalves et al., 2012; Wehrens et al., 2016; Mansard et al., 2018; Airaghi et al., 2020a; Alaoui et al., 2023). The intrinsic weakness of mica is mostly inferred from the observation of natural rocks, where micas are concentrated in zones of strain localization (Bons, 1988; Kronenberg et al., 1990; Wintsch et al., 1995; Gueydan et al. 2003). Nonetheless, mechanical data to support such weakness are rather scarce (Shea and Kronenberg, 1993), and the underlying mechanism of deformation is not well understood. Several studies on naturally deformed mica showed basal slip and kinking along their basal plane, and diffusion creep as deformation processes (Bell et al., 1986; Bell and Cuff 1989; Hippertt 1994; Kronenberg et al., 1990; Mares and Kronenberg, 1993; Meike 1989; Mulch et al., 2006). Additionally, micas also display a recently identified type of defect referred to as "ripplocations" (Aslin et al., 2019, Airaghi et al., 2020b), adding to the complexity of characterizing plastic deformation in micas.

The presence of mica and their interconnection plays also a key role in the deformation and recrystallization of anhydrous phases such as quartz (Tullis and Wenk, 1994; Hunter et al., 2016; Holyoke and Tullis, 2006; Alaoui et al., 2023; Tokle et al., 2023). Several experimental studies

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examined the role of mica on the mechanical behavior of polyphase aggregates (Gottschalk et al., 1990; Shea and Kronenberg, 1992, 1993; Tullis and Wenk, 1994; Rawling et al., 2002; Tokle et al., 2023), highlighting that mica weakens the rock strength by decreasing the CPO of quartz, preventing quartz dynamic recrystallization and by developing weak layers of interconnected micas (Tokle et al., 2023). First, the presence of phyllosilicates, as a second phase, inhibits quartz grain growth through the Zener-pinning effect (Song and Ree, 2007; Hunter et al., 2016). Additionally, micas promote solution-transfer diffusion creep in quartz aggregates (Farver and Yund, 2000; Alcantar et al., 2003; Anzalone et al., 2006; Meyer et al., 2006; Greene et al., 2009), and dissolution-precipitation processes in general (Rutter and Wanten, 2000; Alcantar et al., 2003; Anzalone et al., 2006, Meyer et al., 2006; Greene et al., 2009). In order to quantify the viscosity of two-phase mixtures, some rheological mixing models using end-member flow laws for each phase were developed (Tullis et al., 1991; Dimanov and Dresen, 2005; Huet et al., 2014; Platt, 2015; Rast and Ruh, 2021). These classical models apply to two-phase aggregates described as a mechanical mixture of both pure phases, i.e. they do not consider interphase processes promoted in these aggregates (Pongrac et al., 2022; Alaoui et al., 2023; Tokle et al., 2023).

The general objective of this work is to understand the mechanical properties of the mica-bearing shear zones present in deformed upper crustal rocks. For this purpose, we investigated, by the means of shear experiments at high pressure and high temperature conditions (Griggs-type apparatus), the mechanical strength of mixtures of quartz and phlogopite for variable proportions of the two phases. We analyzed the microstructures in experimentally-deformed samples using SEM, EBSD and cathodoluminescence, in order to unravel the deformation processes and the chemical interactions between the two phases.

IV.2. Results

V.2.1. Mechanical results

The mechanical behavior and bulk strength of the quartz-mica mixture varies according to the volume proportion of phlogopite in the assemblage (Fig. 1). All the experiments were carried out at the same conditions and on the same apparatus to obtain an internally consistent dataset for mechanical strength of investigated aggregates. The sample that sustain the highest differential peak stress of 1239 MPa and 943 MPa respectively are sample made of 10% vol. of phlogopite (10Phl, experiment 659KA) and sample made of 20% vol. of phlogopite (20Phl, 657KA; Fig. 1). Sample 10Phl deforms above Goetze criterion (differential peak stress $\leq$ confining pressure), an empirical criterion delineating stress conditions where rocks deform plastically (Kohlstedt et al., 1995).

Samples that sustain the lowest differential peak stress are those made of 30% vol. of phlogopite (30Phl, 652KA, 338 MPa) and 100% vol. of phlogopite (100Phl, 687KA; 432 MPa, Fig. 1). Samples made of 50% vol. of phlogopite (50Phl, 663KA) and of 70% vol. of phlogopite (70Phl, 672KA) exhibit intermediate differential peak stress of 571MPa and 637MPa, higher than for sample 30Phl. The weakening after peak stress varies strongly, without any strict correlation with the mica proportion, although the largest weakening corresponds to mixtures with a low proportion of mica (Fig. 1a).

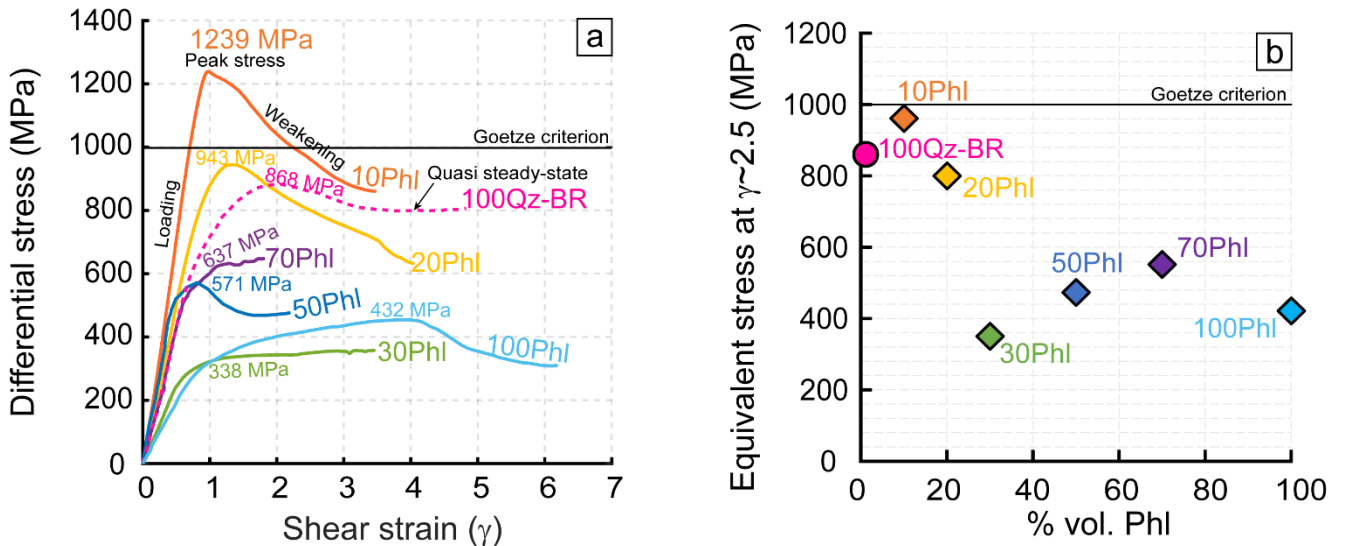


Figure 1 : Mechanical data of the experiment series. a) Stress-strain evolution for mica-quartz assemblage experiments. b) Equivalent stress-Phlogopite proportion evolution for mica-quartz assemblage experiments at $\gamma \sim 2.5$ using 100Qz-BR from Richter et al., 2018. Confining pressure is 1.0GPa, temperature is 800°C and strain rate is $\sim 10^{-5} \text{ s}^{-1}$. Quartz grain size is 10-20 μm in all experiments except in 100Qz-BR (0-100 μm) and phlogopite grain size is 63-125 μm . H₂O content is 0.1% wt in all experiments except in 100Qz-BR (0.2% wt.).

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Experiment	Mica proportion vol.	Total displacement stiffness corrected mm	Time		Displacement rate vertical* mm/s	Shear displacement rate*
			hours	seconds		
687KA	1	100%	35.71	128563	1.49E-05	2.10E-05
672KA	0.7	70%	23.00	82803	1.52E-05	2.16E-05
663KA	0.5	50%	31.95	115020	1.60E-05	2.26E-05
652KA	0.3	30%	34.34	123620	1.57E-05	2.22E-05
657KA	0.2	20%	35.45	127603	1.46E-05	2.07E-05
659KA	0.1	10%	36.26	130552	1.49E-05	2.10E-05
448BR**	0	0%	44.65	160729	1.49E-05	2.10E-05

Experiment	Mica proportion vol.	Final thickness mm	Shear strain rate* s ⁻¹	Equivalent strain rate	Effective viscosity		Equivalent stress		Total shear strain
					Pa s	MPa	Pa	Pa	
687KA	1	100%	4.48E-05	2.58544E-05	1.63E+13	421	421000000	421000000	6.20
672KA	0.7	70%	2.20E-05	1.26979E-05	4.34E+13	552	551620000	551620000	1.82
663KA	0.5	50%	1.91E-05	1.10448E-05	4.28E+13	473	472687000	472687000	2.20
652KA	0.3	30%	2.71E-05	1.5636E-05	2.24E+13	350	350000000	350000000	3.45
657KA	0.2	20%	2.95E-05	1.70584E-05	4.69E+13	800	800000000	800000000	4.05
659KA	0.1	10%	2.53E-05	1.46103E-05	6.58E+13	961	961000000	961000000	3.48
448BR**	0	0%	2.81E-05	1.61969E-05	5.31E+13	860	860000000	860000000	4.67

*calculated on averaging values around a shear strain of 2.5, averaging from 2.48 to 2.54.

**Richter et al., 2018

Table 1 : Experimental conditions for constant shear strain experiments with only Rig stiffness correction applied on the displacement. Area correction $\cos^2$ is applied on the stress. Average displacement, strain rate, stress and viscosity are calculated for a shear strain at 2.5, for experiment at lower shear strain the final one was applied.

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Although the strain rates for all experiments are of the same order of magnitude, significant variations in the absolute values were observed among experiments due to variable final thickness of the different sheared samples. Even if the same amount of powder was used in all experiments, and chosen as to aim at a constant thickness of ~1mm, the final thickness we observed varies largely, as a result of variable material extrusion in addition to simple shear deformation (Table 1). Similarly, the displacement and duration are different for some experiments following technical issues, but most lasted 34-35 hours with a total displacement of ~2 mm (Table 1). In order to overcome differences in displacement and thickness, the effective viscosity (η) of the different deformed quartz-phlogopite aggregates (Table 1), was calculated using the following equation (Paterson and Olgaard, 2000):

$$\eta = \frac{\sigma_{eq}}{\dot{\epsilon}_{eq}} \text{ with } \sigma_{eq} = 2 \times \tau = \Delta\sigma \text{ and } \dot{\epsilon}_{eq} = \frac{\dot{\gamma}}{\sqrt{3}}$$

where σ_{eq} is the equivalent stress, $\dot{\epsilon}_{eq}$ is the equivalent strain rate, τ is the shear stress, $\Delta\sigma$ is the differential stress and $\dot{\gamma}$ is the shear strain rate.

The equivalent stress around $\gamma=2.5$ (Fig. 1b, Table 1) and the effective viscosity (Table 3) show that at first order strength decrease with increasing proportion of mica. A scatter is nevertheless visible in the dataset, as for example the 30Phl is almost as weak as the pure phlogopite experiment. However, experiments in chapter VI show that the stress values measured are highly reproducible when using several types of mica (intermediate biotite and fluorophlogopite). A large decrease in strength is present for a low proportion of mica, i.e. in the range 0-30% vol.

V.2.2. Microstructures

Three samples were selected for detailed microstructural and composition analyses based on significant changes in the mechanical behavior: 10Phl, 30Phl and 50Phl.

In Phl-poor samples (10% and 30% vol.), interconnected layers of quartz (Fig. 2a, b) are visible. These layers are constituted of ~10 μm -thick, elongated grains surrounded by a fine-grained quartz matrix. Conversely, in Phl-rich samples (>30% vol.), phlogopite grains form interconnected layers surrounding angularly shaped quartz clasts (Fig. 2c).

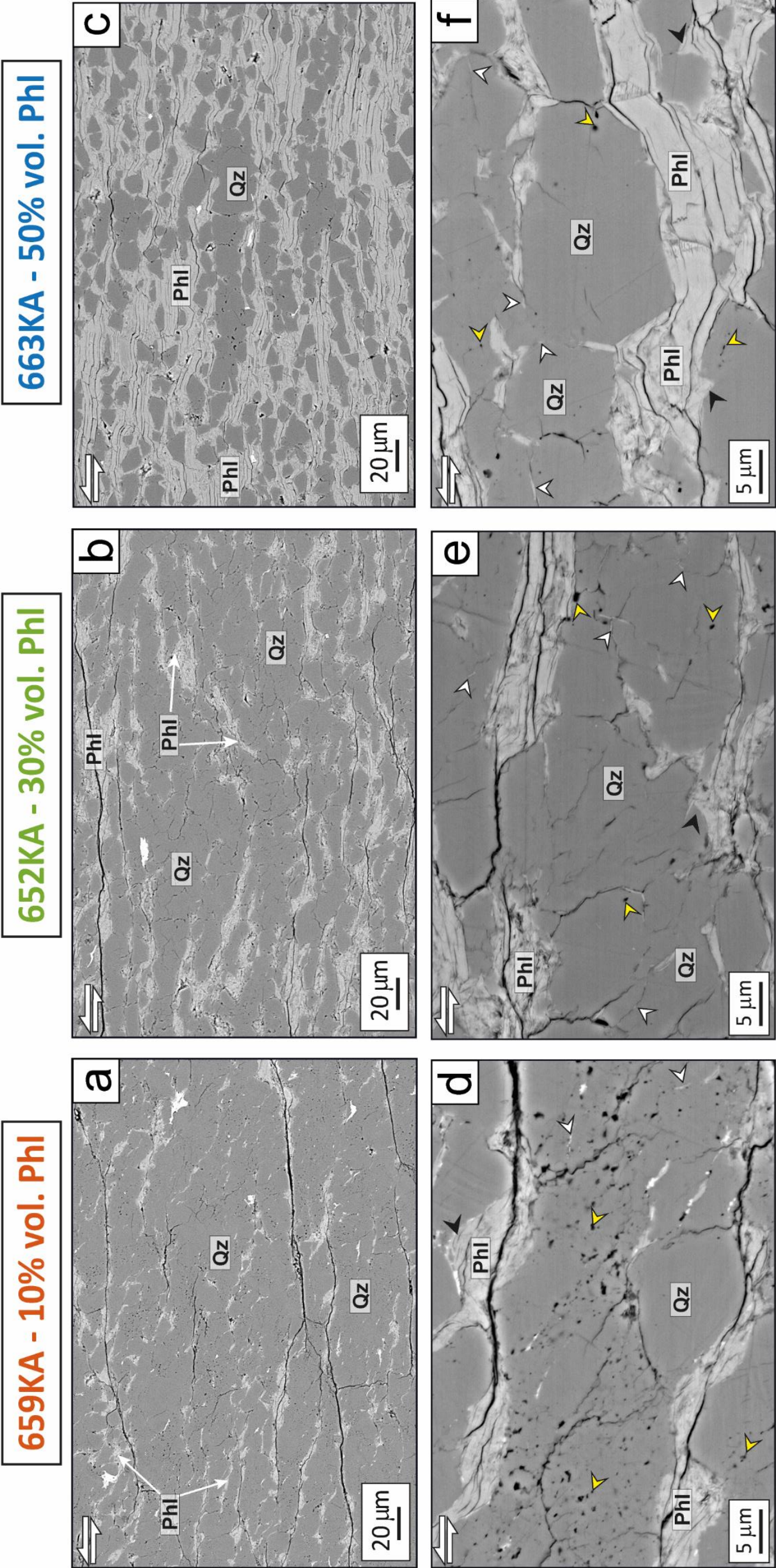


Figure 2 : Microstructural observations from deformed mica-quartz assemblages under a scanning electron microscope (SEM). Backscattered electron (BSE) images at low magnification showing quartz interconnected layers in a) 10Phl and in b) 30Phl, and showing in c) interconnected phlogopite layers in 50Phl. d) BSE image of 10Phl showing high amount of micropores, thin, sheared and elongated relict grains of phlogopite in a quartz matrix. e) BSE image of 30Phl and f) 50Phl showing elongated, kinked and pinched relict grains of phlogopite surrounded by angular relict clasts of quartz, with few micropores. White arrows: tiny phlogopite grains (<5mm). Yellow arrows: micropores. Black arrows: phlogopite pinched and kinked microstructures. Mineral abbreviations are from [Whitney and Evans \(2010\)](#).

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In all deformed experiments, large relict phlogopite grains ($> 5 \mu\text{m}$ with average size $40 \mu\text{m}$, Fig. 2) are elongated parallel to the foliation, grains are folded, kinked and pinched among quartz grains resulting in apophyses (black arrows in Fig. 2d to f). Newly grown small mica grains ($< 5 \mu\text{m}$, Fig. 2) are located within interstices among quartz-quartz grain boundaries (white arrows in Fig. 2d to f). A micro porosity in Phl-poor samples (10% and 30% vol.), with pore diameter in average $< 1 \mu\text{m}$, is located mainly along grain boundaries of quartz (yellow arrows in Fig. 2 d to f).

In summary, regardless of mica proportion, microstructures highlight that mica is the weak phase and the difference in the nature of the interconnected layers could explain the overall decrease in strength with increasing mica proportion. Nonetheless, the expected evolution is monotonic, and is at variance with the local minimum in viscosity observed for 30% vol. Phl. Therefore, in the following section we will examine deformation and crystallographic preferred orientation of quartz grains, to investigate the deformation processes in quartz + mica aggregates.

V.2.3. Deformation and crystallographic preferred orientation of quartz grains

EBSD was used to quantify intragranular misorientations, grain size distribution, shape preferred orientation (SPO) and crystallographic preferred orientation (CPO) of quartz grains in high strain areas of samples 10Phl, 30Phl and 50Phl. CPO was constructed using one-point-per-grain in equal-area lower-hemisphere pole figures (Fig. 3). Grains were defined as areas delimited by high-angle ($\geq 10^\circ$) boundaries (Halfpenny et al., 2012) and a minimum of 5 indexed consecutive pixels on several rows. Low-angle boundaries are identified as segments within the grains showing a misorientation angle $\geq 2^\circ$ between two pixels (Trimby et al., 1998). Quartz grains were discriminated between parent grains $> 8 \mu\text{m}$ and new grains according to the grain size, with have a grain size $< 8 \mu\text{m}$ Table 2 summarizes the characteristics of quartz grains for each experiment.

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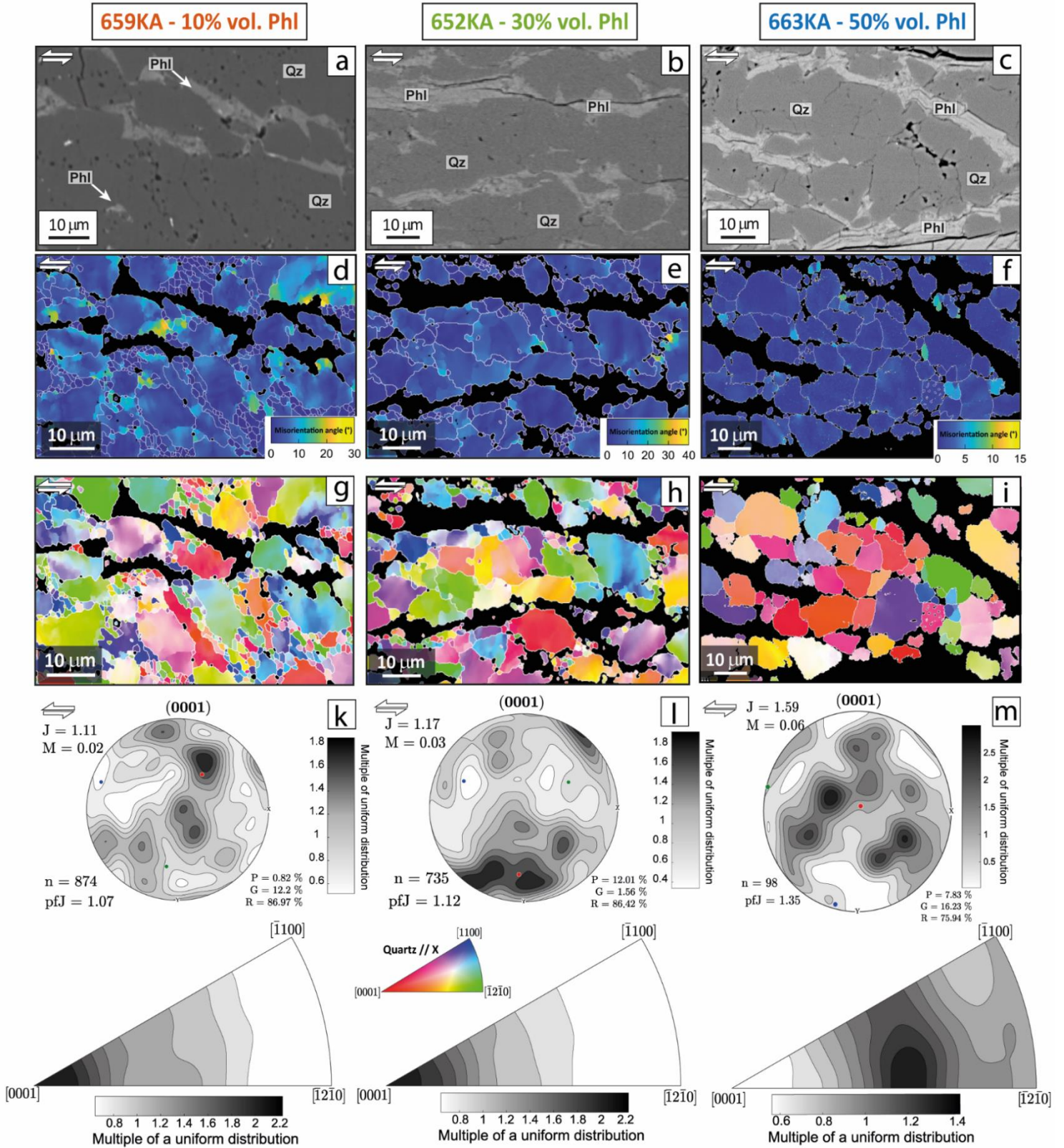


Figure 3 : BSE images of the location for electron backscatter diffraction (EBSD) mapping for a) 10Phl, b) 30Phl and c) 50Phl samples. d), e) and f) corresponding intergranular misorientation maps. g), h) and i) corresponding EBSD maps of quartz orientations parallel to the X direction. j), k) and l) corresponding pole figures showing quartz CPO. All pole figures are lower hemisphere projections plotted as point per grain. J-index, M-index and number of grains (n) are shown for each. Misorientation axes distribution (between 2° and 10°). All contours are multiples of uniform distribution (m.u.d).

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Regardless of Phl proportion, all pole figures show a large scattering of CPO, pointing to a very weak fabric, as attested also by the low values of J and M indexes (1.11 to 1.59 and 0.02 to 0.06, in Table 2 and Fig. 3i, k and m). Intragrain deformation of quartz increases for decreasing mica proportion, and in Phl-poor samples we intra-grain misorientation angles as large as 30° (Fig. 3d, e). In the two samples where grain misorientation is significative, misorientation axes concentrated along [0001] (Fig. 3k, l).

For low proportion of Phl (10 and 30 vol%), quartz grain size is fairly reduced from 10-20 µm to 1-2 µm (Table 2, Fig. 4a), with a narrow distribution (Fig. 4a). In the experiment with 10 vol% of phlogopite, recrystallized quartz grains are located between and surrounding parent quartz grains with a polygonal shape (Fig. 3d, e, g and h), whereas the larger inherited grain show a significant SPO and preferential elongation that defines the foliation formed during simple shear (Fig. 4b, c). To summarize, the decrease in Phl amount affects directly deformation microstructures of quartz (but for the CPO). In the following section, we will examine how the amount of Phl can influence quartz chemically.

Sample	Mica proportion	J	M	Parent quartz grains (>8 µm)			Neoformed quartz grains (<8 µm)		
				Number of grains	Grain size (µm)	Average Aspect ratio	Number of grains	Grain size (µm)	Average Aspect ratio
659KA	10% vol. Phl	1.11	0.02	18	9.75 ± 0.178	2.18 ± 0.945	856	1.41 ± 0.649	1.79 ± 0.558
652KA	30% vol. Phl	1.17	0.03	41	10.66 ± 0.194	1.92 ± 0.730	693	1.763 ± 0.669	1.72 ± 0.578
663KA	50% vol. Phl	1.59	0.06	27	10.34 ± 0.203	1.68 ± 0.440	71	4.399 ± 0.429	1.74 ± 0.649

Table 2 : Crystallographic characteristics of quartz in studied samples for three different mica proportions, with J and M indexes informing on the fabric for all grains in each map. Number of grains, mean grain size and average aspect ratio are given for parent quartz grains and recrystallized quartz grains.

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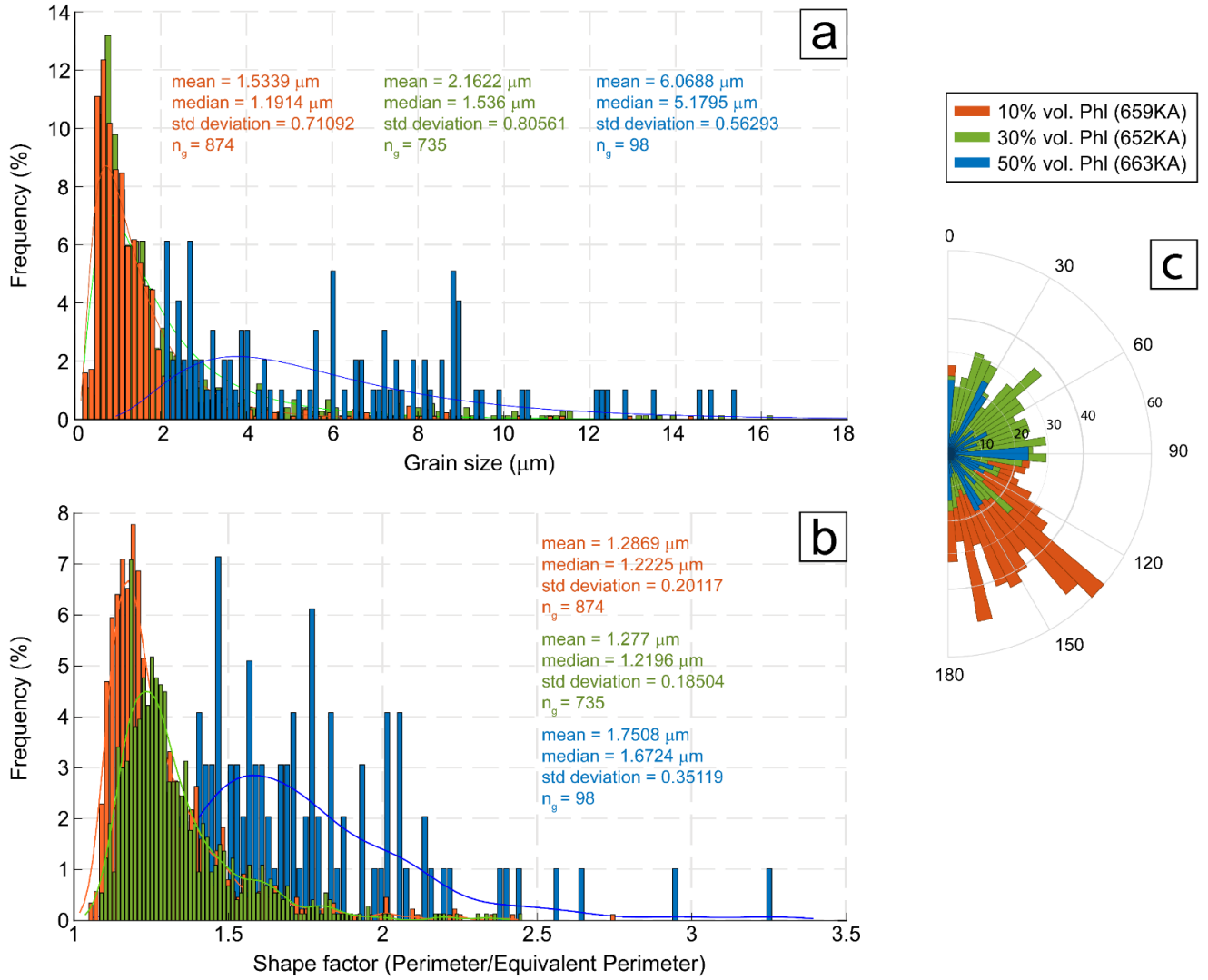


Figure 4 : Grain size and shape properties for all quartz grains. a) Grain size distribution with grain statistics for quartz grains in 10% vol. Phl (659KA), 30% vol. Phl (652KA) 50% vol. Phl (663KA) samples. b) Shape factor with grain statistics for quartz grains in 10% vol. Phl (659KA), 30% vol. Phl (652KA) 50% vol. Phl (663KA) samples. c) Shape preferred orientation for quartz grains in 10% vol. Phl (659KA), 30% vol. Phl (652KA) 50% vol. Phl (663KA) samples. Number of grains is indicated by n_g .

V.2.4. Cathodoluminescence imaging of quartz and EBSD correlation

Hyperspectral cathodoluminescence (CL) images exposes non-luminescent grains corresponding to phlogopite, rare Fe-oxides and fractures (Fig. 5-7-11), and luminescent grains of quartz. Different colors for quartz reveal the existence of two types of quartz in deformed sample (Fig. 5a to d): yellow (Qz1) indicating a dominant emission band at 653 nm (1.9 eV), and blue (Qz2) showing dominant emission bands around 443 nm (2.8 eV) and at 653 nm (1.9 eV). Conversely, in the undeformed starting material the quartz grains are defined only by yellow luminescence (Fig. 5d, 7a). The CL emission from the yellow grains (Qz1) is also characterized by a peak at 2.9 eV for all samples, showing low intensity compared to the starting material (Fig. 5d, 6). For all experiments, blue grains (Qz2) are characterized by peaks at 2.7 eV, 2.9 eV and 3.2 eV (Fig. 5d). Mineral phase maps extracted from CL maps show that the proportion of Qz2 over Qz1 increases with decreasing amount of mica (Fig. 5e). Over all samples, Qz1 can be correlated with parent quartz grains observed in EBSD maps (Fig. 3d to i) forming ~10 μm angular, fractured or sheared grains (Fig. 4a-b). Similarly, Qz2 can be correlated with the small recrystallized quartz grains (Fig. 3d to i) forming smaller grains (less than 5 μm) surrounding Qz1 or filling the cracks cross-cutting Qz1 (Fig. 5a to c).

In conclusion from these observations, with decreasing the amount of Phl we increase the amount of blue luminescence quartz grains (Qz2), knowing that CL colors provide information on the origin of the quartz, and underlying deformation mechanisms, and therefore on the strength.

In the following section, we will investigate mineral composition to decipher why Phl-poor assemblages have a higher amount of Qz2.

IV.2. Results

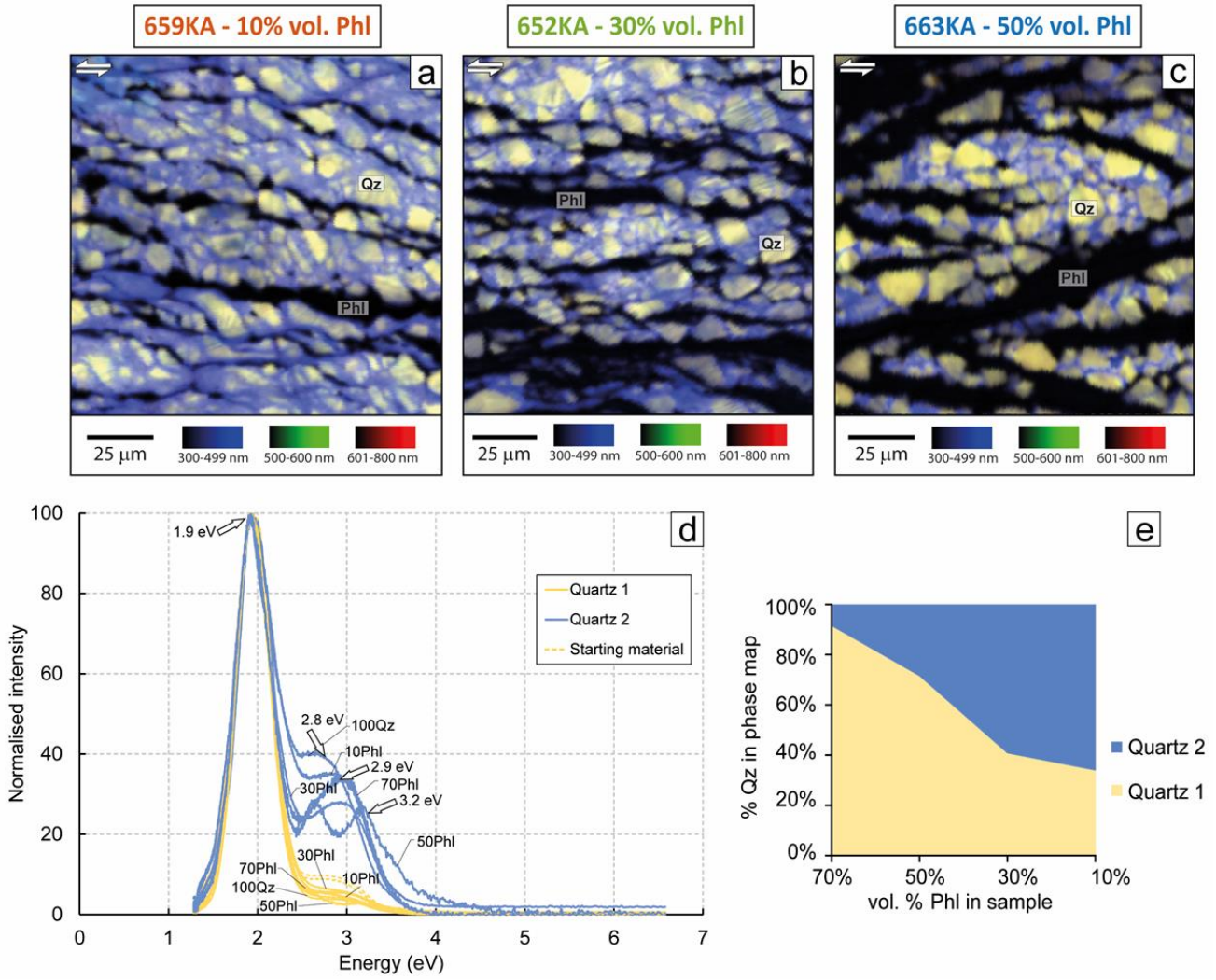


Figure 5 : Hyperspectral cathodoluminescence (CL) imaging from the corresponding samples. BSE images of the location for CL mapping for a) 10Phl, b) 30Phl and c) 50Phl samples. d), e) and f) corresponding CL image. Yellow luminescence (570 nm-2.17 eV) exhibits Qz1, blue luminescence (450 nm-2.75 eV) corresponds to Qz2, and the absence of luminescence in black gathers phlogopite, Fe-oxides and fractures. d) Peak fitted spectrum energy (eV) followed normalised to 100 counts intensity. e) Relative proportions of quartz types, Qz1 and Qz2, in phase map vs. proportion of phlogopite in phase map.

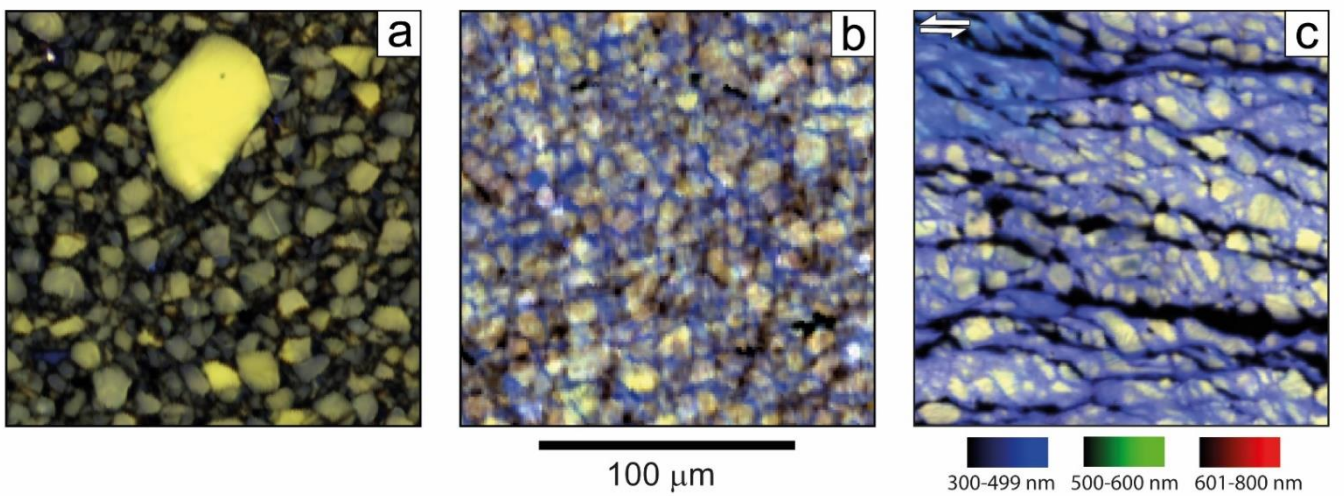


Figure 6 : Hyperspectral cathodoluminescence (CL) imaging from 100% vol. Qz and 10% vol. Phl samples. a) 688KA undeformed cold sample (30°C-5kbar). b) 690KA undeformed sample (800°C-10kbar) to $\gamma=3.5$. c) 659KA deformed 10Phl sample.

V.2.5. Mineral composition

Qz1 has an average aluminum content of 0.059% wt., 0.067% wt., 0.065% wt. Al_2O_3 in the experiments with 10%, 30% and 50% vol. Phl, respectively (Fig. 7d to i). Conversely, Qz2 exhibits a higher aluminum content with an average of 0.086% wt., 0.094% wt, 0.066% wt. Al_2O_3 in the experiments with 10%, 30% and 50% vol. Phl, respectively (Fig. 7d to f).

Reworked quartz material (Qz2) shows a higher mean content in aluminum than quartz relict clasts (Qz1), with a higher aluminum content in Qz2 in 10Phl and 30Phl than in 50Phl (Fig. 8g and h).

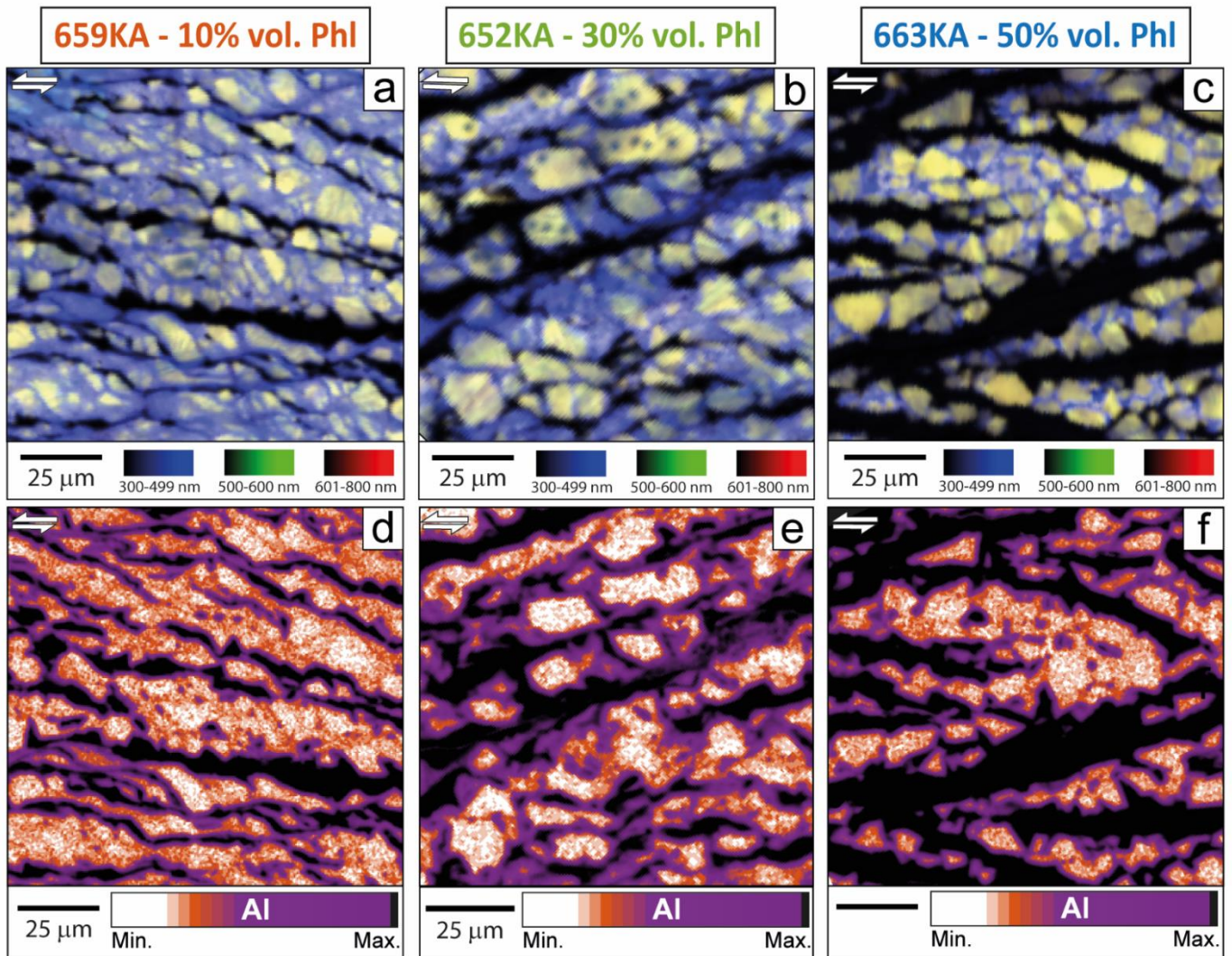


Figure 7 : CL maps and corresponding Al_2O_3 elemental map for a), d) 10Phl, b), e) 30Phl, c), f) 50Phl.

IV.2. Results

The chemical composition of phlogopite weakly varies among samples with homogeneous trend for each sample (Fig. 8c, d). Chemical composition between samples varies slightly for Si content, but is mainly comprised in 2.6-3.2 a.p.f.u. Similarly, alkaline content between samples evolves overall between 0.8-1 a.p.f.u. The largest variations between samples are observed for Al^{VI} and Al^{IV} , respectively 0.05-0.6 a.p.f.u and 0.3-1.3 a.p.f.u. (Fig. 8c, d). However, TEM images show that chemical composition of phlogopite varies among samples for Si/Al in average from 4.92 to 5.93, respectively parent grains to recrystallized grains (Fig. 9e, f), from 2.97 to 3.12 for Mg/Al and from 4.77 to 3.62 for Mg/Fe (Fig. 9d, f).

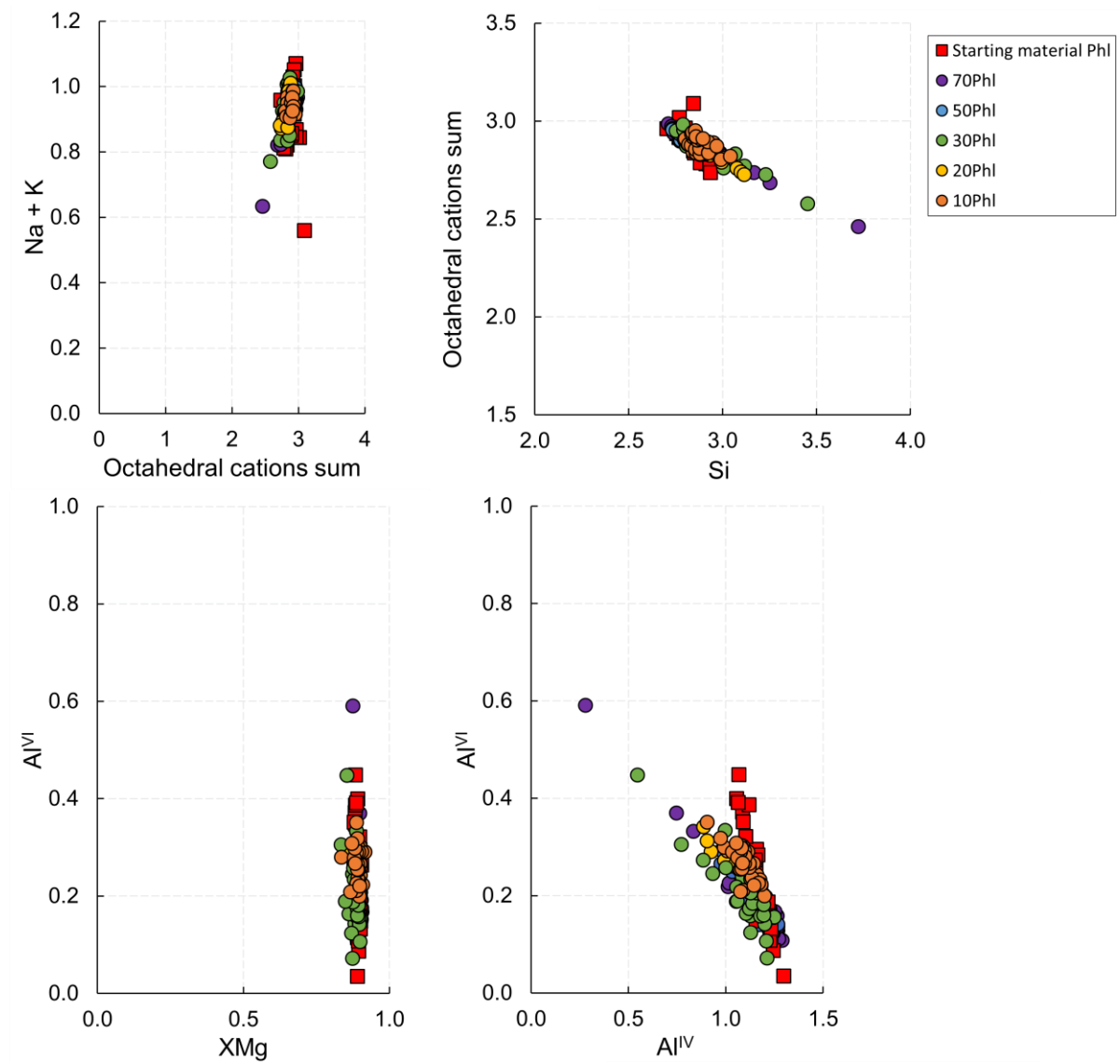


Figure 8 : Plots of EPMA point analyses for phlogopite in a.p.f.u. a) Na+K versus Octahedral cations sum. b) Octahedral cations sum versus Si. c) Al^{VI} versus XMg. d) Al^{VI} versus Al^{IV} .

IV.2. Results

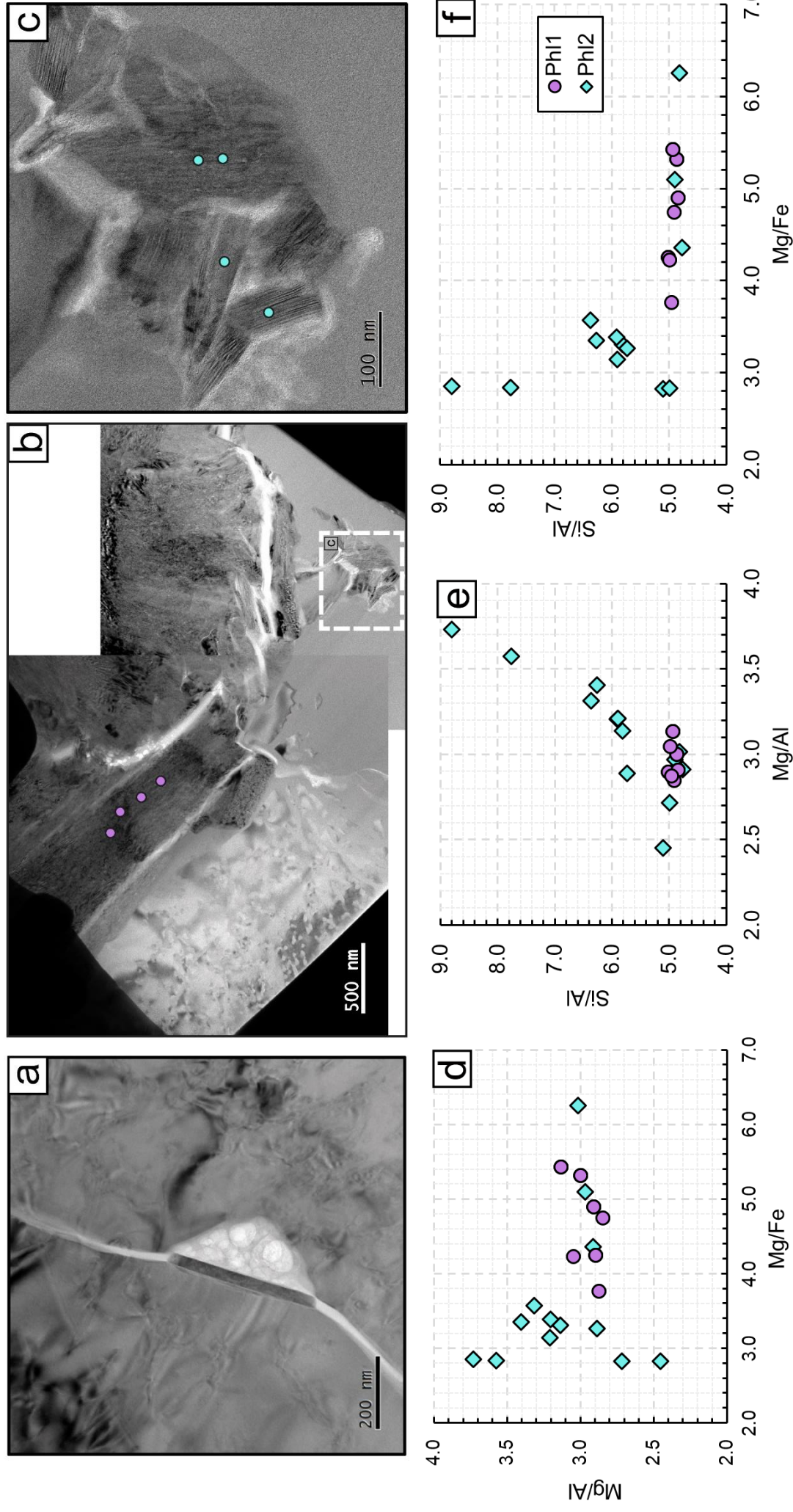


Figure 9 : Transmission Electronic Microscope (TEM) images from: a) Recrystallized phlogopite grain located at quartz grain boundary in 50Phl. b) Parent and recrystallized phlogopite grains in 30Phl with four EDS point analysis location. c) Aggregate of recrystallized phlogopite grains located at quartz grain boundary in 30Phl with four EDS point analysis location. Plots of EDS point analysis for parent phlogopite grains (Phl1) and recrystallized grains (Phl2) of d) Mg/Al vs Mg/Fe, e) Mg/Al vs Si/Al and f) Mg/Fe and Si/Al. The resting point analysis are located in similar areas of the same sample.

IV.3. Discussion

IV.3.1. Quartz boundary processes

We observed that with decreasing the amount of Phl we increase the amount of blue luminescence quartz grains (Qz2), knowing that CL colors provide information on the origin of the quartz. Hence, reworked quartz material (Qz2) shows a higher mean content in aluminum than quartz relict clasts (Qz1), with a higher aluminum content in Qz2 in Phl-poor (<30% vol.) than Phl-rich (>30% vol.) samples.

CL color variations between blue and yellow could be linked to the CL emission band at 1.9 eV linked to structural defects in oxygen in deficient quartz, such as the presence of fewer non-bridging oxygen hole center defects or less OH-groups (Stevens-Kalceff, and Phillips., 1995; Hamers et al., 2023; Götze et al., 2024). Conversely, the emission band at 2.8-2.9 eV correlates with concentration of trace Ti and substitutional Ti^{4+} at Si^{4+} position (Stevens-Kalceff, and Phillips., 1995; Götze et al., 2024; Hamers et al., 2023). Finally, the CL emission band at 3.2 eV has been related in previous studies to Al^{3+} substitution of Si in the quartz lattice (Götze et al., 2001; Stevens-Kalceff, 2013; Hamers et al., 2023), which we were able to quantify with EPMA analyses (Fig. 8). The incorporation of Al^{3+} by diffusion from the alumina pistons is discarded by the fact that no Al signal is detected in the pure quartz experiments.

Farver and Yund, 2000 worked on silicon bulk diffusion in natural quartz aggregates at grain boundary, calculating different diffusion distances for silicon and oxygen for similar temperature to our samples but different confining pressure (Fig. 10). Additionally, they showed that, in hydrothermal conditions, silicon is the species that limits the rate of diffusion in quartz aggregates, with grain boundary diffusion predominating effective diffusion rates.

When comparing EBSD and CL maps (Fig. 11, 12) we observe that reworked blue quartz (Qz2) does not correlate with small neoformed quartz grains (<8 μm) and is localized at grain boundaries suggested that Qz2 is overgrowing Qz1 (arrows in Fig. 11). Therefore, reworked quartz (Qz2) rich in aluminum cannot be formed by aluminum diffusion but its formation implies necessarily precipitation therefore solution-transfer diffusion creep process. The systematic change observed in CL images with different

IV.3. Discussion

reworking mechanism of quartz (Fig. 5, 6, 7), could be linked to the fact that in reworked quartz (Qz2) the composition is not changed by solution-transfer diffusion creep process but something else as a structural defect. Mechanisms of quartz reworking are therefore discussed in the following section.

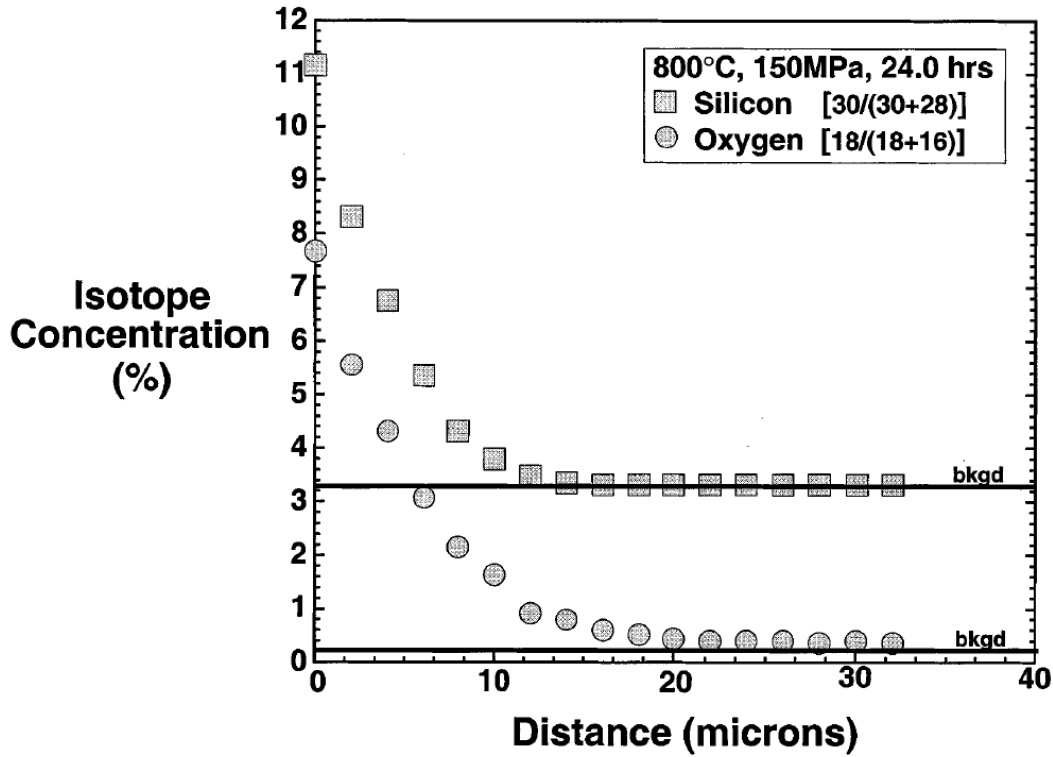


Figure 10: Representative depth profile of silicon and oxygen isotope concentrations vs distance from the surface in quartz aggregate after hydrothermal diffusion anneal at 800°C 150 MPa confining pressure for 24 hours. Bkgd: background values for each element (after [Farver and Yund, 2000](#))

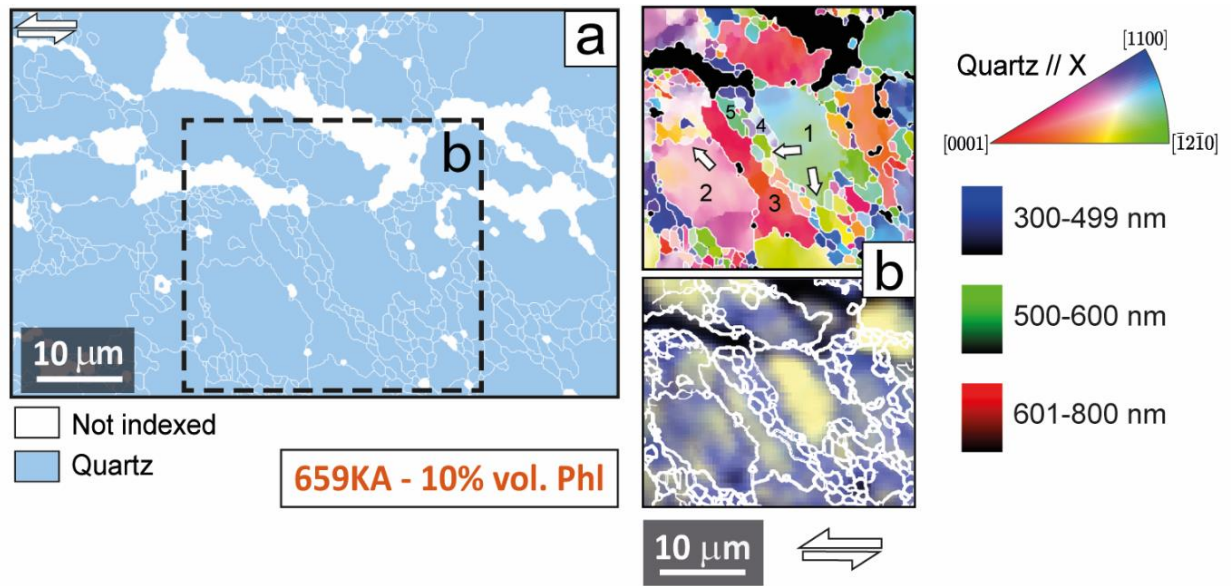


Figure 11: Phase map a) of 10Phl, with b) corresponding EBSD map of quartz orientations parallel to the X direction, correlated to CL map.

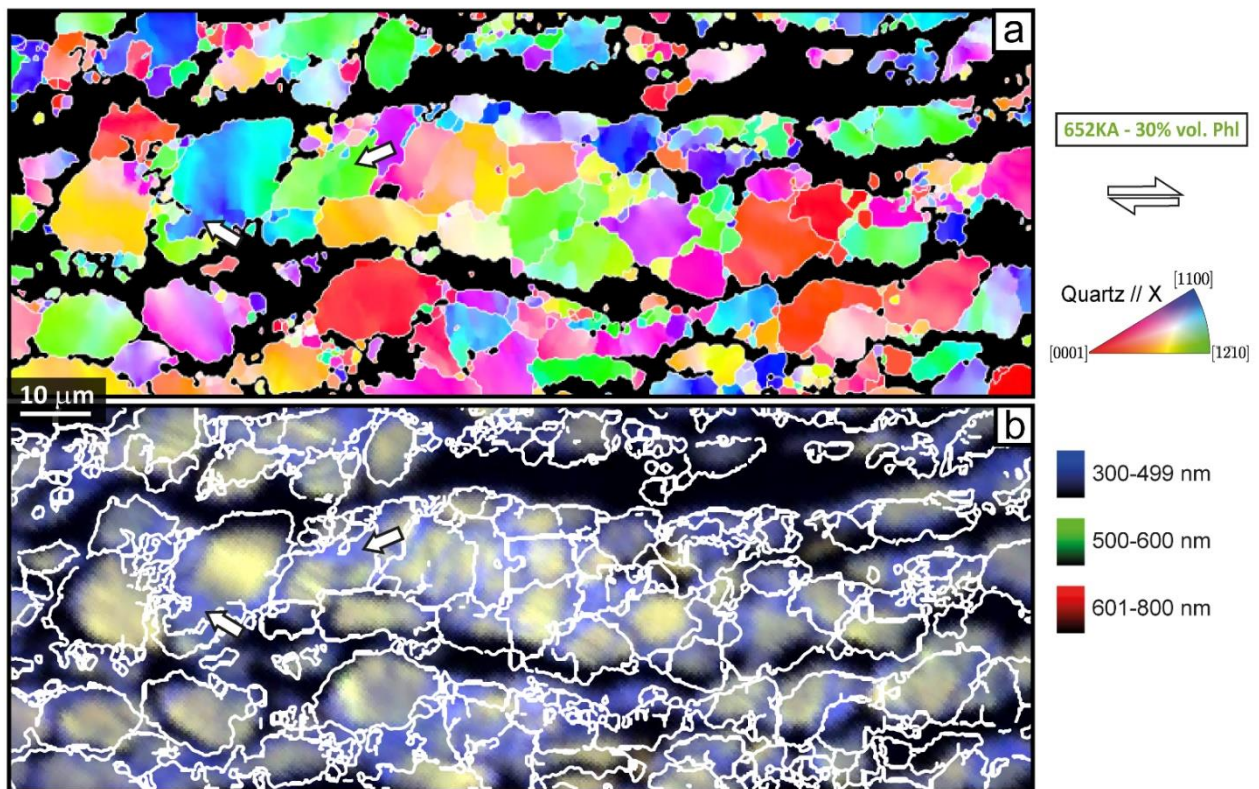


Figure 12 : EBSD map of quartz orientations parallel to the X direction, correlated to CL map in 30Phl sample.

IV.3.2. Mechanisms of quartz reworking

Microstructures and EBSD analyses suggest different mechanisms of quartz reworking in samples with different mica proportions. Overall as J and M indexes of quartz are low, quartz fabric is poor which is compatible with a shear strain of 3-4. Compared to pure quartz experiments of [Richter et al. \(2018\)](#) at similar conditions with pfJ index at 2.0, our samples have pfJ indexes of quartz are of 1.07 and 1.12 for Phl-poor samples which is close. However, our Phl-poor samples do not exhibit any CPO as evidenced by pole figures, conversely to [Richter et al. \(2018\)](#) sample exhibiting peripheral maximum of the c-axes slightly rotated with the sense of shear. On the other hand, strain accommodation in quartz by plastic deformation is observed, especially in Phl-poor (10Phl) samples, and highlighted by elongated Qz1 with higher shape factors and SPO than Qz2, high internal misorientation, core and mantle like structures ([White, 1976](#)). Also, the occurrence of small polygonal grains between and surrounding inherited quartz clasts (Qz1), the homogeneous size of recrystallized grains and the inverse pole figures (IPF) showing that lattice rotation axes across low-angle boundaries cluster around $[0001]_c$ axis suggesting a prism $\langle a \rangle$ slip system indicate that quartz experienced dynamic recrystallization by subgrain rotation (SGR). This mechanism (also known as regime II) is common in experimental samples ([Hirth and Tullis, 1992](#); [Stipp et al., 2002](#); [Stipp and Tullis, 2003](#)). Therefore, to determine whether the use of piezometers is appropriate in multi-phase systems (here mica + quartz) or if other methods are necessary to evaluate stresses in these conditions, we compared the relationship between stress and grain size in our experiments with those from other studies using quartz alone (Fig. 13). Interestingly the only results correlating with the piezometer of [Stipp and Tullis \(2003\)](#) are the Tana quartzite experiments of [Nègre \(2021\)](#). Indeed, [Palazzin \(2016\)](#) results are lower than the theoretical values predicted by the curve of [Stipp and Tullis \(2003\)](#). [Palazzin \(2016\)](#) explains this deviation by the contribution of grain-size sensitive process (GSS) which participate to lower grain size. This could also be the case for [Richter et al. \(2018\)](#) experiments as it does not correlate with the piezometer curves. Finally, in our case of we observed that depending on how we discriminate our recrystallized grains (using the GOS as in [Cross et al., 2017](#) or using the grain size as a criteria) a non-neglectable uncertainty appears. Thus, the use of piezometers of quartz is not in favor of multi-phase systems certainly, but mainly it is not usable for

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samples where diffusion creep is dominant as it was mainly constructed on the base of dislocation creep dominant samples, explaining the discrepancies with other studies (Fig. 13).

EBSD results highlighted that despite a weak bulk fabric the structure is made of dynamic recrystallized quartz making the sample overall strong. However, if we look into EBSD and CL correlation, the recrystallized quartz grains in EBSD maps correlate mainly with the location of blue luminescence Qz2 grains (Fig. 12b). However, blue luminescence Qz2 does not correlate only with recrystallized quartz grains observed on EBSD maps. Indeed, a crystallographic continuity is observed in the misorientations axis distribution of each grain (1, 2 and 3) and does not correlate only to yellow luminescence Qz1 grains, but to both yellow Qz1 and blue Qz2 grains (Fig. 12b). Therefore, CL highlights another mechanism the 10Phl sample is dissolution-precipitation following the amount of Qz2 (Fig. 5a) suggesting that 10% vol. Phl is enough to activate this process. In order to reconcile these conflicting observations, TEM observations are crucial as at the nanoscale, plastic deformation is evident through dense defects and grain misorientation (see following chapter V). However, we do not see clear signs of dynamic recrystallization, such as subgrains or grain boundary migration initiation as observed by [Hirth and Tullis \(1992\)](#).

In sample made of 30% vol. Phl, several observations indicate that the dominant mechanism of quartz reworking is solution-transfer diffusion creep (dissolution-precipitation): (i) high proportion of Qz2 in CL images, (ii) angularly shaped parent quartz grains, (iii) no core and mantle like structures (Fig. 3, 5, 6), (iv) overgrowths of Qz2 resulting from inherited grains Qz1 (Fig. 11). Nevertheless, the occurrence of recrystallized grains with different sizes and different orientation, a prism $\langle a \rangle$ slip system suggested by IPF suggests that recrystallization by SGR is still active.

In Phl-rich samples (>30% vol.), the angular grain shape of parent grains, poor misorientation and the SPO suggest that quartz is practically undeformed plastically, although CL and compositional variation indicate solution-transfer diffusion creep. Indeed, quartz dissolves and precipitates however, the amount of quartz is poor leading to less strain accommodation by quartz and less quartz reworking, even if mica proportion is higher. The main mechanism is mica plastic deformation although a notable secondary mechanism is dissolution-precipitation of quartz.

IV.3. Discussion

Therefore, overall samples dynamic recrystallization by SGR and solution-transfer diffusion creep are the main mechanisms of quartz reworking occurring. The dominance and competition of each mechanism depends on mica proportion as differences in mica proportion results in different distribution and density of symmetrical (quartz-quartz and mica-mica) and asymmetrical (quartz-mica) grain boundaries. Indeed, two mechanisms of recrystallization are present and are defined by similar CL signals, and that we therefore need to find a common explanation that is compatible with both mechanisms which is given by TEM analysis in the following chapter V.

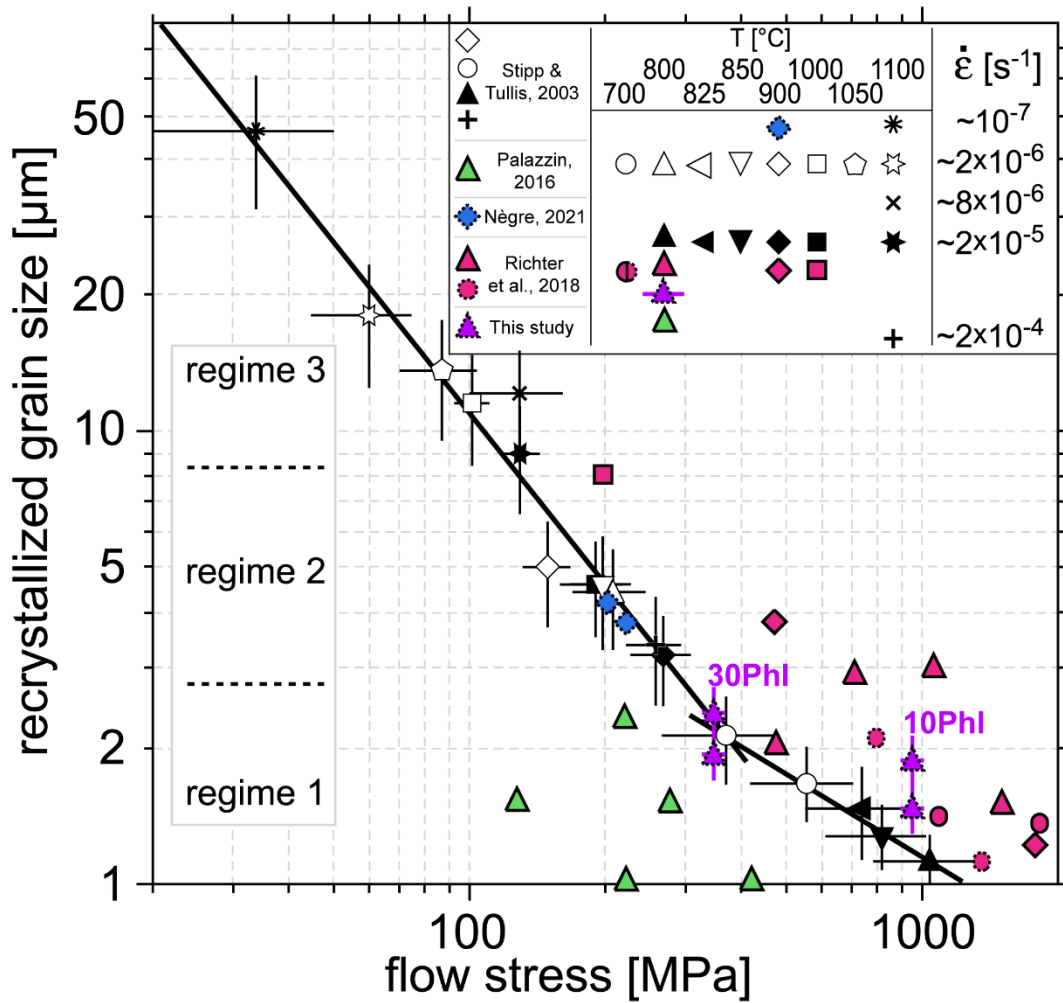


Figure 13: Recrystallized grain sizes for this experimental study and previous ones are plotted with the corresponding flow stress values on the diagram of recrystallized grain size paleopiezometer of quartz from Stipp and Tullis (2003). The two curves represent the relationships estimated by the paleopiezometer for the regimes proposed by Hirth and Tullis (1992). All experiments confining pressure is 1.5 GPa except the ones with dashed contours where it is 1.0 GPa.

IV.3.3. Mica deformation

In mica, deformation occurs through kinking and folding (Fig. 2), typically attributed to basal slip suggesting intragrain plastic deformation for phlogopite (Etheridge et al 1973; Bell et al., 1986; Meike 1989; Kronenberg et al.,1990; Mares and Kronenberg, 1993). However, we find no evidence of basal slip, as there are no basal dislocations observed (evidenced by TEM, see the following chapter V).

The microstructural arrangement of small phlogopite grains and the composition changing (Fig. 2, 9) indicates that phlogopite dissolves and precipitates with varying transport distances when they are found around relict phlogopite grains, in cracks, or in opened quartz grain boundaries. This implies that the primary mechanism underlying the reduction of phlogopite grain size is dissolution-precipitation, rather than dynamic recrystallization or mechanical reduction from kinking (for more evidences on mica nanostructures see the following chapter V).

According to Wilson and Bell (1979), dissolution takes place in a metamorphic fluid and, at the expense of the parent grains that are deformed, causes the initiation and growth of fine-grained, strain-free mica through diffusional processes. This process essentially mimics mineral replacement through dissolution-precipitation and is similar to the chemically-driven recrystallization (Etheridge and Hobbs, 1974; Putnis, 2002, Putnis and Putnis 2007, Ruiz-Agudo et al., 2014). We can suggest that in our samples the process of formation of small new mica is dissolution-precipitation and chemically-driven recrystallization.

IV.3.4. Effect of mica on quartz deformation and dissolution-precipitation as weakening processes in mica-quartz aggregates

Mechanism controlling deformation of each assemblage varies between quartz plastic deformation, mica plastic deformation and dissolution-precipitation processes. Mica influences quartz deformation such as when the structure is composed of interconnected mica (samples with $\geq 50\%$ vol. Phl) quartz deformation is limited (Fig. 2c, f and 3f, i, m). A shift of the dominant deformation mechanism in mica and quartz occurs when increasing the amount of phlogopite. In fact, for high amount of Phl ($>30\%$ vol.), dissolution-precipitation is mainly controlled by deviatoric stress affecting quartz. The effect of phlogopite is significant when quartz, as the strong phase, is the interconnected phase.

Nonetheless mica also has a chemical effect on deformation, as our results show that mica is recrystallized, liberating OH in the system. It was reported in previous studies (Farver and Yund, 2000; Alcantar et al., 2003; Anzalone et al., 2006; Meyer et al., 2006; Greene et al., 2009) that at mica-quartz interface, dissolution is facilitated compared to another type of interface such as quartz-quartz, as water content increases the thickness of the fluid film at grain boundaries (Fig. 14). In fact according to Farver and Yund (2000), our case of solution-transfer creep would be island-channel diffusion along hydrated grain boundaries model (after den Brok, 1998). By promoting dissolution-precipitation of quartz, micas induce quartz to behave as a weak phase, as shown in sample 30Phl. Therefore, adding progressively mica that is considered weak (Wintsch et al., 1995; Gueydan et al., 2003; Wehrens et al., 2016; Airaghi et al., 2020a; Graziani et al., 2020, Tokle et al., 2023), does not decrease continuously assemblage strength. Indeed, there is a trigger point in our case at 30% vol. Phl where all deformation mechanisms are competing and resulting in a rheological shift from theory. This promoting role of phyllosilicates is not integrated in the creep law (Heald, 1955; Meyer et al., 2006; Renard et al., 1997). However, several studies (Becker, 1995; Schwarz and Stockhert, 1996; Farver and Yund, 2000; Rutter and Wanten, 2000; Renard et al., 2001; Alcantar et al., 2003; Anzalone et al., 2006; Meyer et al., 2006; Greene et al., 2009) introduced a new input coupling phyllosilicate with pressure-solution and localizing dissolution along a flat interface (Aharonov and Katsman, 2009). Indeed, our results show the effect of mica-quartz interactions on the strength of the assemblage, weakening it and controlling the assembly strength. These

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interactions take place mainly at grain boundaries where solution-transfer or effective diffusion happen (dissolution-precipitation). Effective diffusion should be a slow process but, when mica is in the assemblage it makes fluid diffusion (dissolution-precipitation) happen faster (Alcantar et al., 2003; Anzalone et al., 2006; Meyer et al., 2006; Greene et al., 2009). Alcantar et al. (2003) and Anzalone et al. (2006) conclude that mica plays a significant role in pressure solution processes due to its unique layered structure and high surface area. The presence of mica enhances the dissolution and reprecipitation reactions occurring at mineral interfaces, thereby facilitating pressure solution. They observed that the rate and mechanism of pressure solution are influenced by factors such as the composition of the confined fluids and the geometry of the mica surfaces. Additionally, they find that the orientation and size of mica particles can affect the extent of pressure solution and the resulting textures and structures formed.

Meyer et al. (2006) shows that the presence of mica enhances the dissolution of quartz by providing active sites for chemical reactions. Surface defects and fluid films on the mica surface act as preferential sites for ion migration and dissolution processes, speeding up the overall dissolution rate of quartz. Quartz dissolution by mica surfaces displays selective behavior along specific crystallographic planes. This selective dissolution results in characteristic etch patterns and surface textures on the quartz surface, indicating that mica influences the spatial distribution of dissolution in geological materials. Preferential dissolution along specific crystallographic planes can impact the redistribution of material and the development of structural features in geological materials undergoing pressure solution.

Greene et al. (2009) highlights the significant role of mica in mediating electrochemical processes associated with pressure solution phenomena. Mica's unique properties, such as its layered structure and large surface area, provide numerous active sites for electron transfer processes, thereby enhancing electrochemical activity. These active sites promote redox reactions, which play a vital role in both dissolving and precipitating minerals during pressure solution. Moreover, the interlayer spaces within mica serve as conduits for ion transport, facilitating the movement of ions between mineral surfaces and the surrounding fluid. This facilitated ion transport is crucial for mediating electrochemical reactions, leading to the enhanced efficiency of pressure solution processes. The orientation and size of mica

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particles further influence the distribution and kinetics of electrochemical reactions, resulting in spatial variations in pressure solution rates and patterns.

Another thing that could be triggering solution-transfer diffusion creep at grain boundaries is the difference between chemical potential as we have a two-phase aggregate. Indeed, dissolution is activated by a stress difference in chemical potential of the solid between the dissolution and precipitation zones, resulting in mass transfer through a fluid (Paterson, 1973; Weyl, 1959). The chemical potential gradient between mica-quartz might trigger faster dissolution. In hydrostatic (800°C-10kbar) quartz assemblage without mica, dissolution-precipitation still happens as water is present in the system compared to cold quartz assemblage (30°C-5kbar) showing no dissolution-precipitation (Fig. 6a, b). However, dissolution-precipitation in hydrostatic quartz experiment is less pervasive than in a deformed assemblage (Fig. 6), following the stress difference in chemical potential. Therefore solution-transfer diffusion creep appears to be enhanced by the presence of phyllosilicates, which was reported previously in the case of natural sample and could be applied to all hydrated minerals. Graziani et al., 2020 pointed out that only 10% vol. of hydrated minerals is necessary to activate dissolution-precipitation, which is what we observed as well in our experiments.

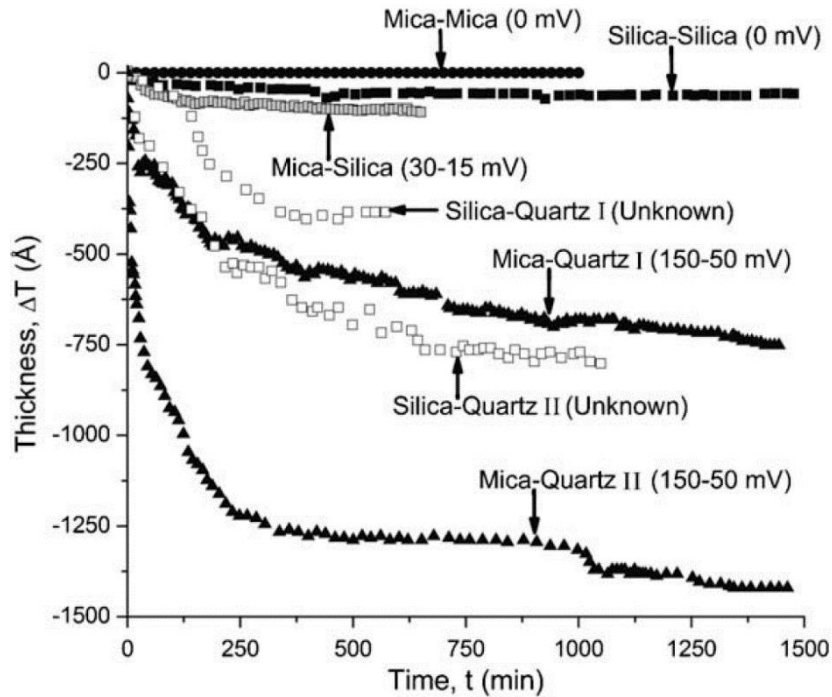


Figure 14 : Representative dissolution rates measured in seven different experiments with five different combinations: mica-mica, (amorphous) silica-silica, silica-mica, silica-quartz, and quartz-mica in 30 mM CaCl₂ solution at pH 2.8–3.2 (after Greene et al., 2009).

IV.3.5. Strength of mica-quartz aggregates relative to pure end-members

Viscosity plots (see below Fig. 15) allow a direct comparison with the iso-stress and iso-strain rate formulation obtained by averaging the rheological behavior of pure phases between two end-members (Voigt, 1928; Reuss, 1929) to describe the rheology of two-phase assemblages. (Tullis et al., 1991; Tullis and Wenk 1994; Dimanov and Dresen, 2005; Huet et al., 2014; Tökle et al., 2023). Overall experiments adding mica decreases the peak stress thus the strength compared to a 100% vol. Qz without changing drastically the viscosity (same order of magnitude). Beyond this first-order trend, viscosity does not decrease systematically with increasing mica proportion (weak phase) as expected from rheological mixing models (Tullis et al., 1991; Tullis and Wenk 1994; Dimanov and Dresen, 2005; Huet et al., 2014; Tökle et al., 2023) and as assumed in other conceptual models (Rast and Ruh, 2021). These rheological mixing models replicate experimental and field data, but they do so in an average manner that has several drawbacks (Huet et al., 2014; Platt, 2015; Ceccato et al., 2020). Mixing models inadequately account for chemical interactions that weaken rocks, leading to underestimated minimum strength and neglect of intergranular processes, resulting in an oversimplified mechanical mixture of pure phases (Dimanov and Dresen, 2005; Ceccato et al., 2020; Pongrac et al., 2022; Alaoui et al., 2023). This study finds that mica-quartz aggregates exhibit complex mechanical behavior not explained by classical models, with significant changes in viscosity and microstr

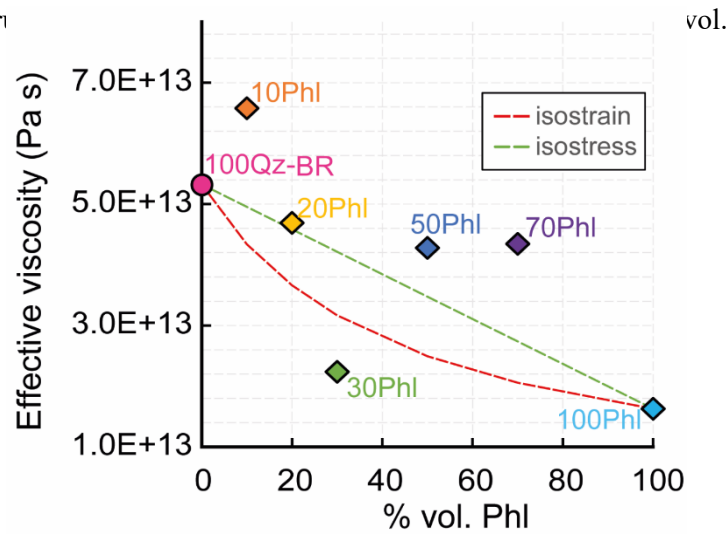


Figure 15 : Viscosity-Phlogopite proportion evolution, dashed lines correspond to iso-strain/iso-stress models using 100Qz-BR from Richter et al., 2018. Confining pressure is 1.0GPa, temperature is 800°C and strain rate is $\sim 10^{-5} \text{ s}^{-1}$. Quartz grain size is 10-20 μm in all experiments except in 100Qz-BR (0-100 μm) and phlogopite grain size is 63-125 μm . H₂O content is 0.1% wt in all experiment except in 100Qz-BR (0.2% wt.).

IV.4. Conclusion

The study concludes that the presence of mica significantly influences the mechanisms of quartz reworking and the overall deformation behavior of mica-quartz aggregates. The key findings are:

- **Quartz Reworking Mechanisms:** The reworking of quartz occurs through two main mechanisms: dynamic recrystallization by subgrain rotation (SGR) and solution-transfer diffusion creep (dissolution-precipitation). The dominance of each mechanism depends on the proportion of mica present in the sample. In Phl-poor samples (<30% vol. Phl), SGR is more prominent, while in Phl-rich samples (>30% vol. Phl), dissolution-precipitation becomes the dominant mechanism.
- **Role of Mica in Quartz Deformation:** Mica facilitates quartz dissolution and precipitation by providing active sites for chemical reactions, enhancing ion transport, and promoting electrochemical activity. This leads to selective dissolution of quartz along specific crystallographic planes and influences the spatial distribution of dissolution, resulting in characteristic etch patterns and surface textures.
- **Influence on Grain Size and Stress Correlation:** The addition of mica affects the relationship between stress and grain size, with results deviating from classical piezometric predictions. This suggests that piezometers based on dislocation creep dominant samples are not suitable for multi-phase systems where diffusion creep is significant.
- **Chemical and Mechanical Interactions:** Mica's chemical interactions, including the release of OH and the influence on fluid films at grain boundaries, enhance dissolution-precipitation processes, weakening the quartz and altering the overall strength of the aggregates. The study highlights that a specific mica proportion (around 30% vol. Phl) triggers a rheological shift, resulting in a complex mechanical behavior not predicted by classical rheological mixing models.
- **Impact on Aggregate Strength:** While adding mica generally decreases the peak stress and strength of the aggregates, the viscosity does not decrease systematically with increasing mica proportion. This non-linear behavior suggests that classical models oversimplify the mechanical mixture of pure phases and fail to account for the weakening effects of chemical interactions and intergranular processes.

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Overall, the presence of mica in quartz aggregates plays a crucial role in mediating deformation mechanisms, enhancing dissolution-precipitation processes, and influencing the mechanical properties and strength of the aggregates in a manner not fully captured by traditional models.

Chapter V – Micro and nanostructures of mica-
quartz assemblages and their implication on
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V.1. Introduction

As seen in the previous chapter, (chapter IV), micas are considered as weak phases in natural shear zones. Experimental results show that increasing proportion of mica in the assemblage decreases bulk strength and promotes shifting in the deformation mechanism of quartz. Dissolution-precipitation and dynamic recrystallization of quartz have been demonstrated in the previous chapter, however question remains on the mechanisms of deformation of mica and intracrystalline deformation of quartz.

Several studies on naturally deformed mica often suggest basal slip as the main mechanism in mica, and it is sometimes related to kinking and diffusion creep but direct evidence of basal slip is lacking (Etheridge et al 1973; Bell et al., 1986; Bell and Cuff 1989; Hippertt 1994; Kronenberg et al.,1990; Mares and Kronenberg, 1993; Meike 1989; Mulch et al., 2006). At the nanoscale, mica appear as an alternating dark and light layer in high magnification TEM images, corresponding to TOT and interlayers respectively (Fig. 1).

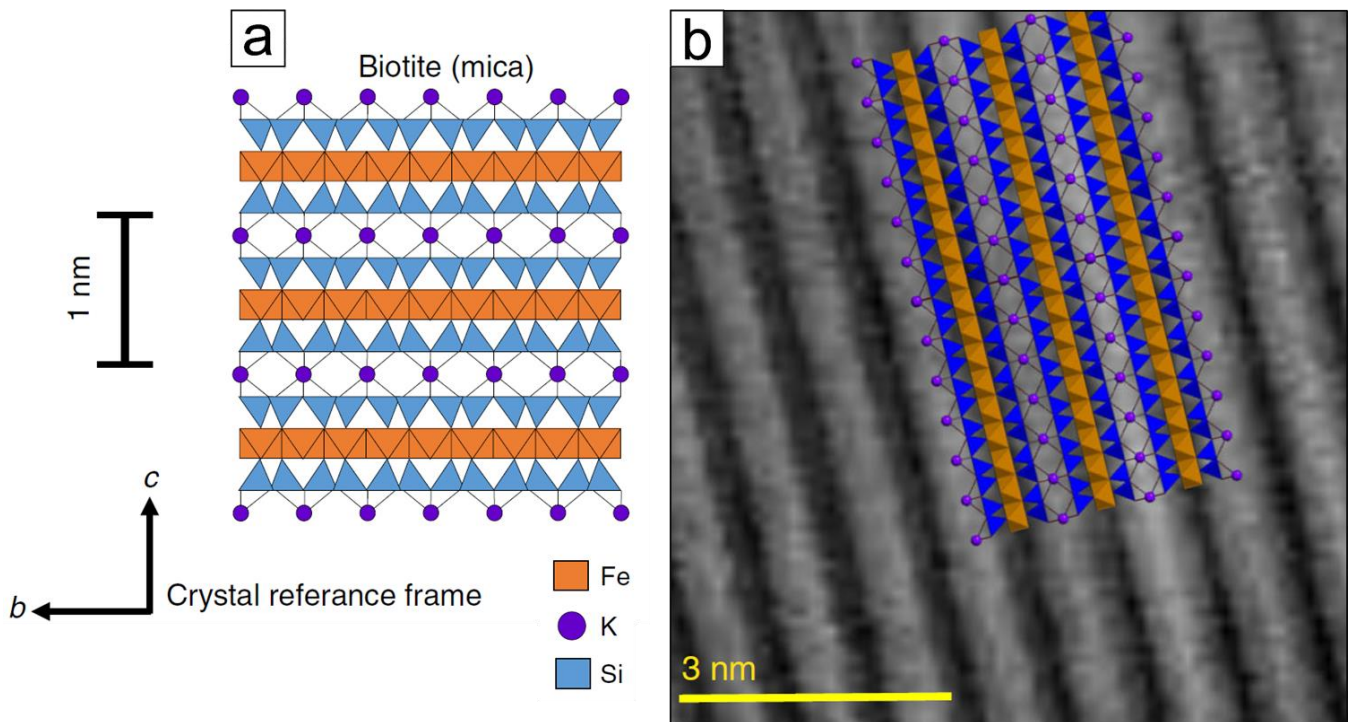


Figure 1 : Phyllosilicate structures and TEM interpretation. a) Atomic structure model of biotite with valences identified by polygons which indices are occupied by oxygen atoms or OH groups. Here an interlayer cation (K^+) is required to balance the overall negative charge of its tetrahedral (T) and octahedral (O) layers. b) High-magnification TEM lattice fringe image with biotite atomic structure model overlain with dark areas center on O layers, intermediate areas show T layers and light areas indicate interlayers (modified from Aslin et al., 2019).

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At this scale, dislocations, kinking, delamination structures and low angle grain boundaries (LAGB) are often observed (Fig. 2, [Dewey, 1965](#); [Hörz, 1970](#); [Bell and Wilson, 1981](#); [Bell et al., 1986](#); [Kogure and Murakami 1998](#); [Misra and Burg, 2012](#); [Aslin et al., 2019](#); [Airaghi et al., 2020b](#)). Recently, a new type of defect has been documented in biotite referred to as "ripplocations" (Fig. 2, [Aslin et al., 2019](#), [Airaghi et al., 2020b](#)). In this kind of defects, the expansion (3-4 nm wide) of the interlayer along the c-axis of the biotite grain propagates as the geometric arrangement of conjugate lattice expansion arrays (Fig. 2, [Aslin et al., 2019](#)). Ripplations open by unloading during uplift or thin sample preparation for TEM observation but revealed within the biotite grains where strain is stored during deformation (Fig. 2, [Aslin et al., 2019](#)).

Dislocation creep is the main deformation process for large grain sizes and high stresses, while dissolution-precipitation takes over for smaller grains and lower stresses.

This study investigates nanostructures of experimentally deformed phlogopite-quartz assemblages, presented in chapter IV, and their implication on strain accommodation at micrometric scale. For this purpose, we analyzed the nanostructures in experimentally deformed samples using FIB, TEM, in the areas where EBSD and CL were previously acquired (see chapter IV). In order to better understand the different deformation processes controlling rheology, microstructures produced at grain boundaries, images acquired with TEM at the subgrain scale allow to identify defects type and their distribution in quartz and mica. This approach can unravel processes occurring at phase and grain boundaries, such as growth and lattice deformation. Through a systematic application of high-resolution analysis to experimental findings, it becomes feasible to compare nanoscale deformation and growth mechanisms in assemblages composed of different proportions of quartz and mica.

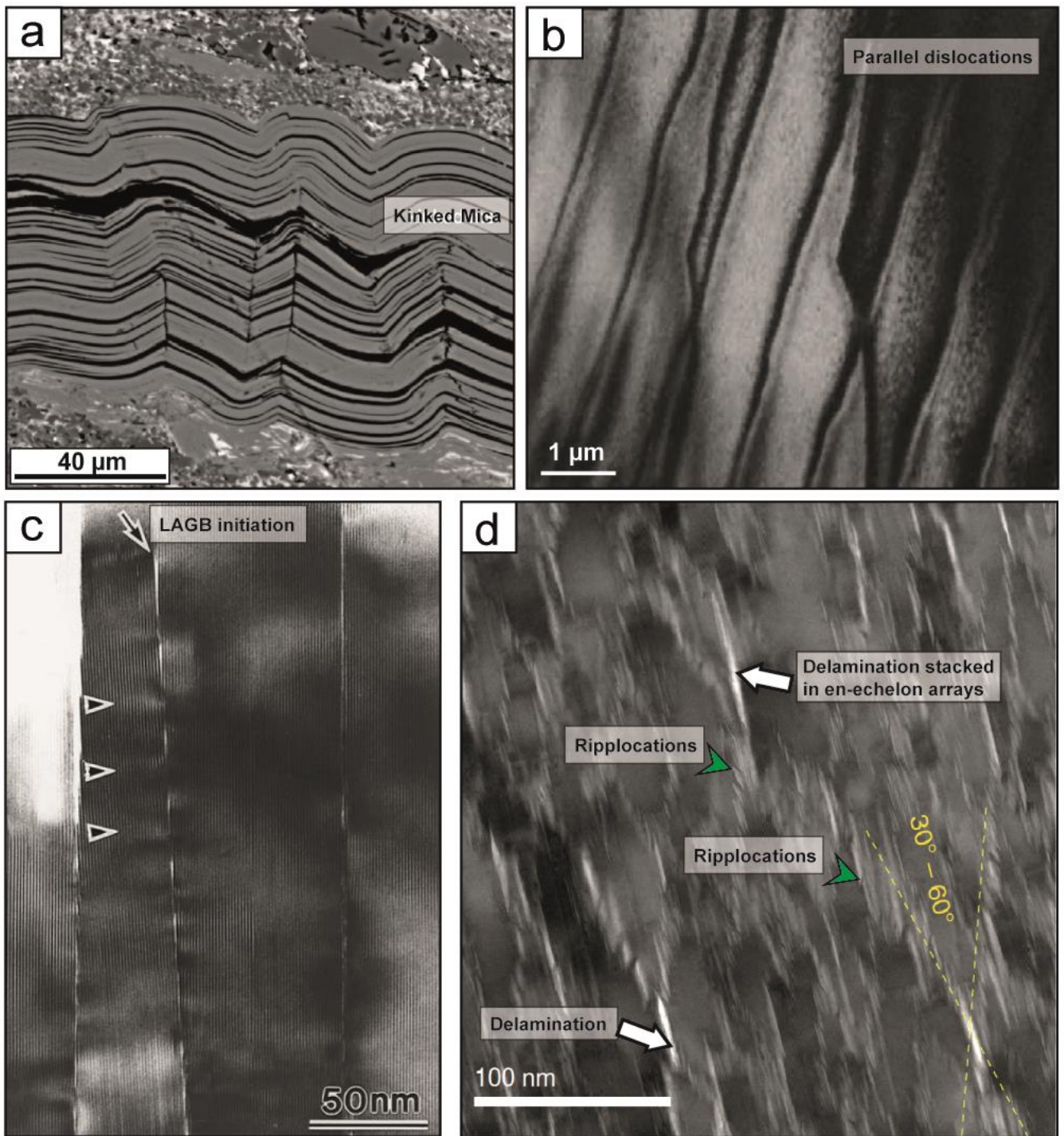


Figure 2 : a) Backscattered electron (BSE) image of kinked mica from [Anderson et al., 2021](#). b) Brightfield TEM image of natural biotite of a (001) section showing parallel dislocations from [Bell et al., 1986](#). c) HRTEM image of chlorite where beam direction is $\langle 010 \rangle$ and the arrow indicates the point where a LAGB is initiated from [Kogure and Murakami, 1998](#). d) Brightfield TEM image of biotite from mylonite where streaks are revealed to be up to 60 Å wide, lozenge-shaped lattice expansion (or delamination shown by white arrows) structures of the biotite basal planes, with en-echelon stacking and ripplocations (green arrows) from [Aslin et al., 2019](#).

V.2. Results

V.2.6. Deformation microstructures in phlogopite grains

SEM results presented in detail in chapter IV indicate that the deformation microstructures in large relict phlogopite grains ($> 5 \mu\text{m}$ with average size $40 \mu\text{m}$, Fig. 3) are mainly folds, kinks and pinches among quartz grains resulting in apophyses (Fig. 3). Newly grown small mica grains ($< 5 \mu\text{m}$, Fig. 3) are located within interstices among quartz-quartz grain boundaries (Fig. 3). So far it was not possible to index mica with EBSD in our samples, and as mica do not luminesce the grains appear as black on CL images.

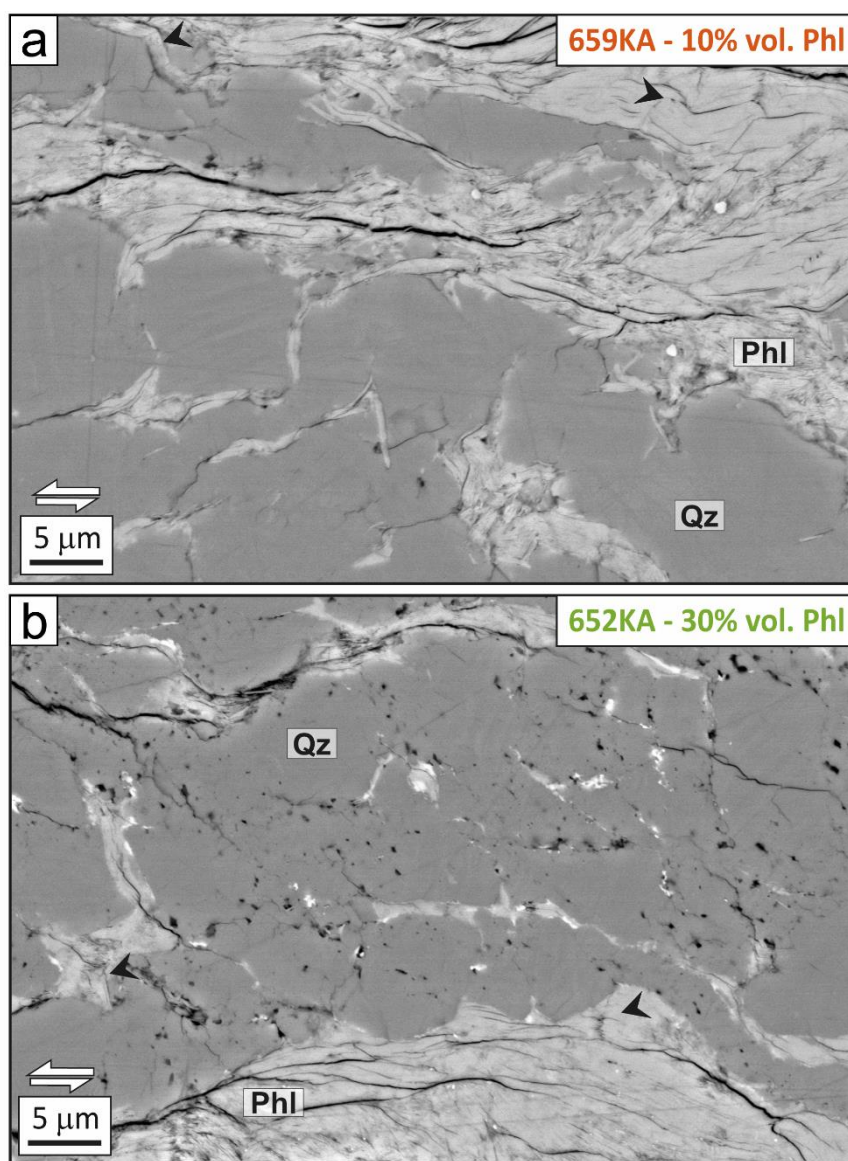


Figure 3: Microstructural observations from deformed mica-quartz assemblages under a scanning electron microscope (SEM). Backscattered electron (BSE) images at low magnification in a) 10Phl and in b) 30Phl. Black arrows: phlogopite pinched and kinked microstructures. Mineral abbreviations are from [Whitney and Evans \(2010\)](#).

V.2.7. Deformation microstructures in quartz grains

EBSD results obtained and presented in chapter IV restricted in high strain areas of sample 10Phl in order to interpret the microstructural framework (intragranular misorientations, grain size distribution, shape preferred orientation and crystallographic preferred orientation) in which nanostructural analysis were made (Fig.4). Parent quartz grains and recrystallized quartz grains were discriminated depending on the grain size: parent grains are larger than 8 μm and recrystallized grains are smaller than 8 μm (Fig. 4). Most parent quartz grains (in mean $9.75 \mu\text{m} \pm 0.18$, yellow CL grains in Fig. 4a) in sample 10Phl exhibit intragrain misorientation angles of 10-30° (Fig. 4d). Recrystallized quartz grains (in mean $1.41 \mu\text{m} \pm 0.65$, blue CL in Fig. 4a) are located between and surrounding parent quartz grains (Fig. 4c and d). These recrystallized grains have a polygonal shape and show barely intra-grain misorientation (misorientation angles are close to 0°, Fig. 4d). Consequently, parent grains and the recrystallized grains are characterized by different orientations (Fig. 4c). Pole figure and the low J and M indices (1.11 and 0.02, Fig. 4b) indicate weak fabric, with misorientation axes concentrated along [0001]_c (Fig. 4b). A significant SPO is noticeable 20-40°W with respect to the X axis (Fig. 4a, c, d and 5b, c). This sample is defined by a unimodal shape factor distribution for recrystallized grains, with a mean of 1.28 ± 0.18 and elongated grains parallel to the foliation (Fig. 5a, b).

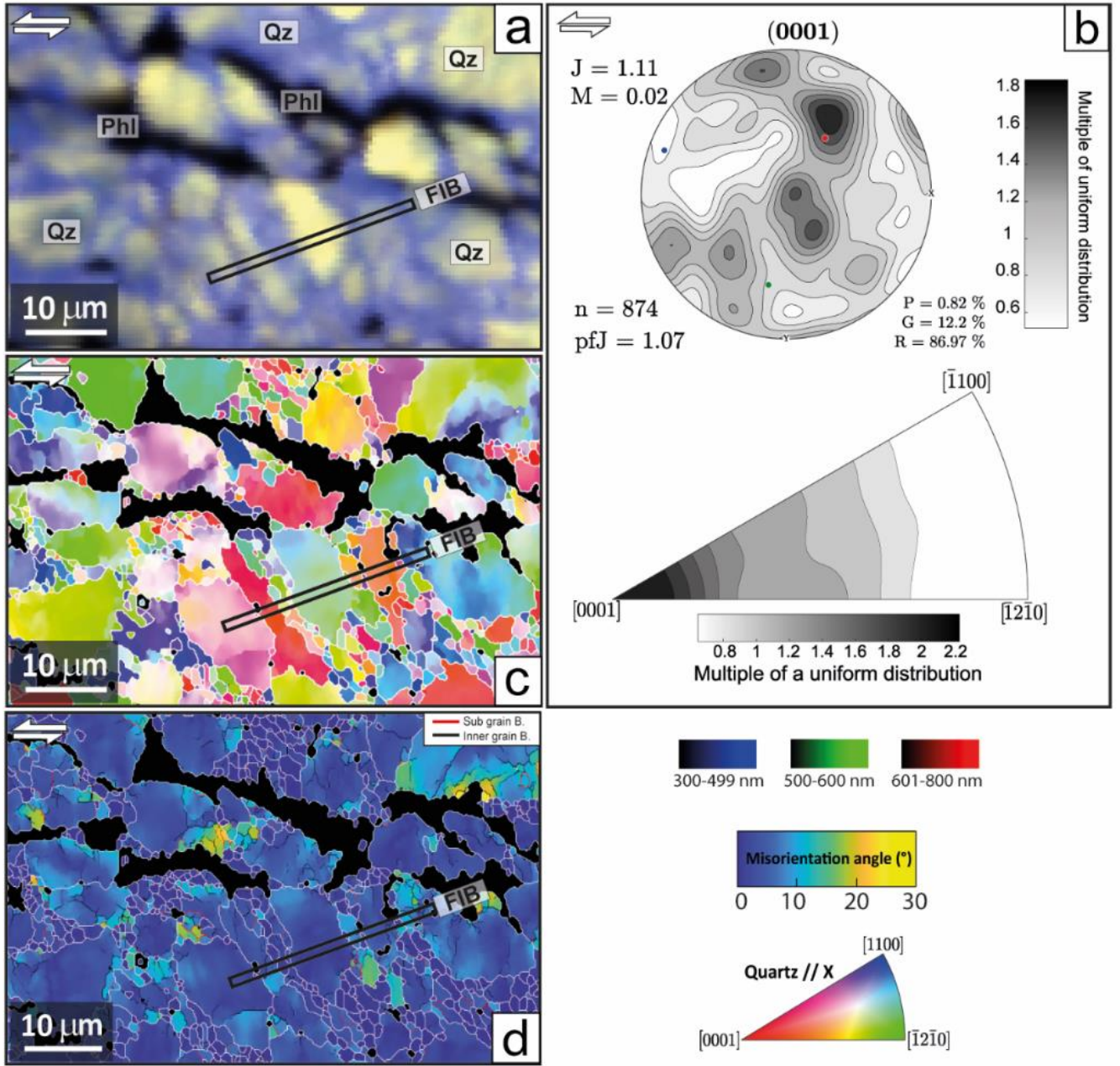


Figure 4 : a) Hyperspectral cathodoluminescence (CL) imaging from 10Phl sample. Yellow luminescence (570 nm-2.17 eV) exhibits Qz1, blue luminescence (450 nm-2.75 eV) corresponds to Qz2, and the absence of luminescence in black gathers phlogopite, Fe-oxides and fractures. b) Pole figures showing quartz CPO and inverse pole figures showing the distribution of correlated misorientation axes and c) corresponding EBSD map of quartz orientations parallel to the X direction and d) intergranular misorientation map with sub-grain boundaries in red and inner grain boundaries in black. All pole figures are lower hemisphere projection plotted as point per grain. J-index, M-index and number of grains (n) are shown for each. Misorientation axes distribution are between 2° and 10°. All contours are multiples of uniform distribution (m.u.d).

V.2. Results

Hyperspectral CL images expose non-luminescent grains corresponding to phlogopite, rare Fe-oxides and fractures, and luminescent grains exhibiting quartz. Different colors for quartz reveal the existence of two types of quartz (Fig. 4a): yellow (parent grains in EBSD forming $\sim 10\ \mu\text{m}$ angular, fractured or sheared grains) indicating a dominant emission band at 653 nm (1.9 eV), and blue (recrystallized grains in EBSD forming smaller grains (less than $5\ \mu\text{m}$) surrounding or filling the cracks cross-cutting parent grains) showing dominant emission bands at 443 nm (2.8 eV) and at 653 nm (1.9 eV). Yellow grains (parent) show high (25 to 40°) intra-grain misorientation angles in EBSD maps while recrystallized blue (in CL) grains show low or no intra-grain misorientation (Fig. 4d). Focused Ion Beam (FIB) foils were cut across parent and recrystallized grains. The exact location of the FIB is shown in Fig. 4. It is of note that microprobe analysis (conditions detailed in chapter III) resulted in the amorphization of a layer of $1.5\ \mu\text{m}$ thickness at the emplacement of the FIB section, hence nanostructural observation were carried out deeper in each FIB window (Fig. 5a).

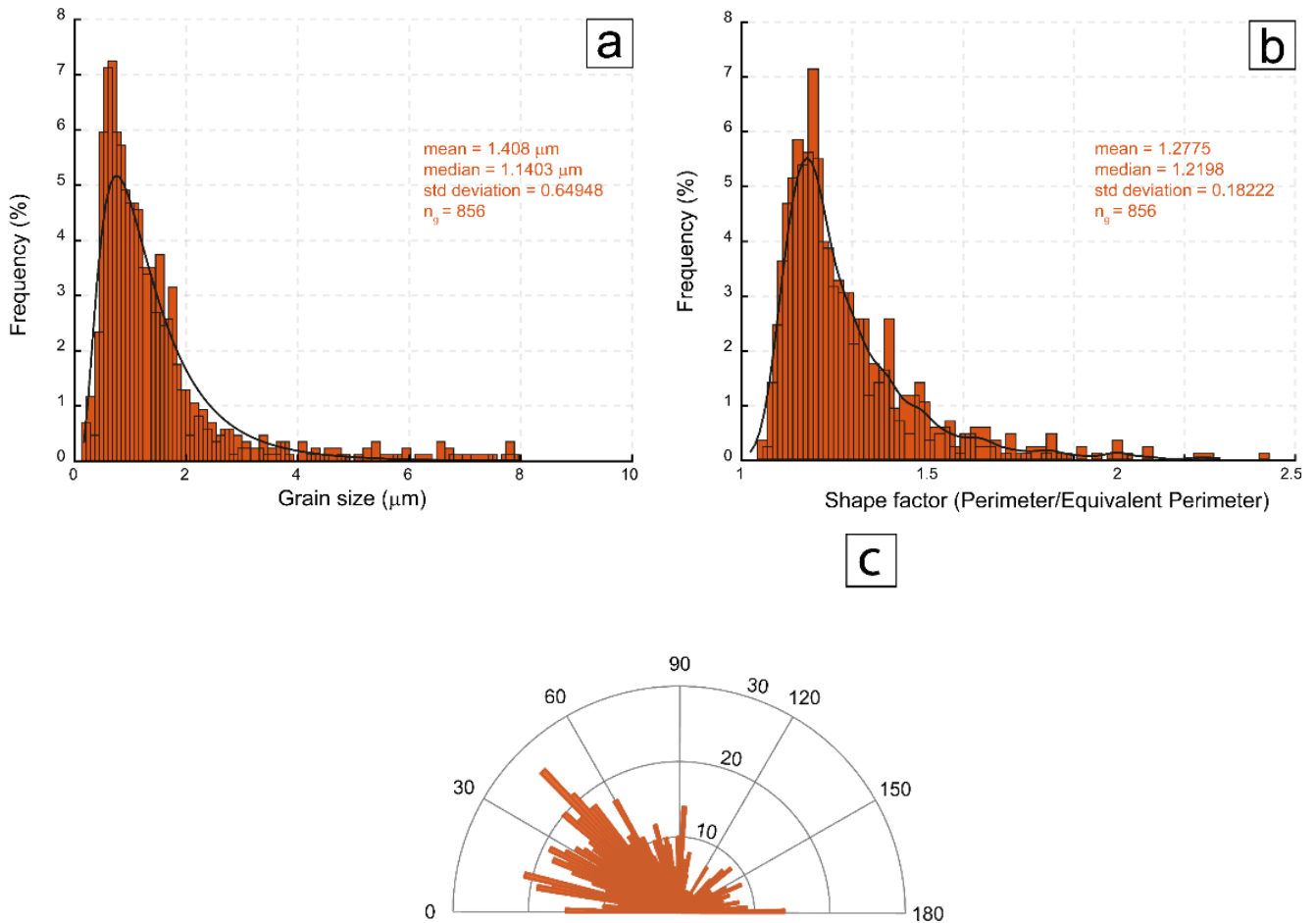


Figure 5 : Grain size and shape properties for recrystallized quartz grains in 10Phl acquired with EBSD (different from Chapter IV Figure 4 which considers every single grain). a) Grain size distribution with grain statistics for recrystallized quartz grains. b) Shape factor with grain statistics for recrystallized quartz grains. c) Shape preferred orientation for recrystallized quartz grains. Number of grains is indicated by n_g .

V.2.8. Nanostructures

Nanostructures in quartz

Parent quartz grains have a grain size larger than $5\ \mu\text{m}$ (Fig. 5a) and show weak intragrain misorientation (Fig. 4a, 6c-d). They are defined by very high defect density in various oriented planes (Fig. 6, 7a). Indeed, by tilting the sample in two directions of various degrees a high density of defects is always observed in parent grains (Fig. 7a, b). Because of the thickness of the FIB foil, single dislocations cannot be imaged, but rather the strain field around them (Fig. 7b, c). We observed no evidence of subgrain boundary forming, as no regular patterns were observed in dislocation densities such as progressive arrangement of dislocations towards grain boundaries to define polygonal shapes (Fig. 7a, b, 8b).

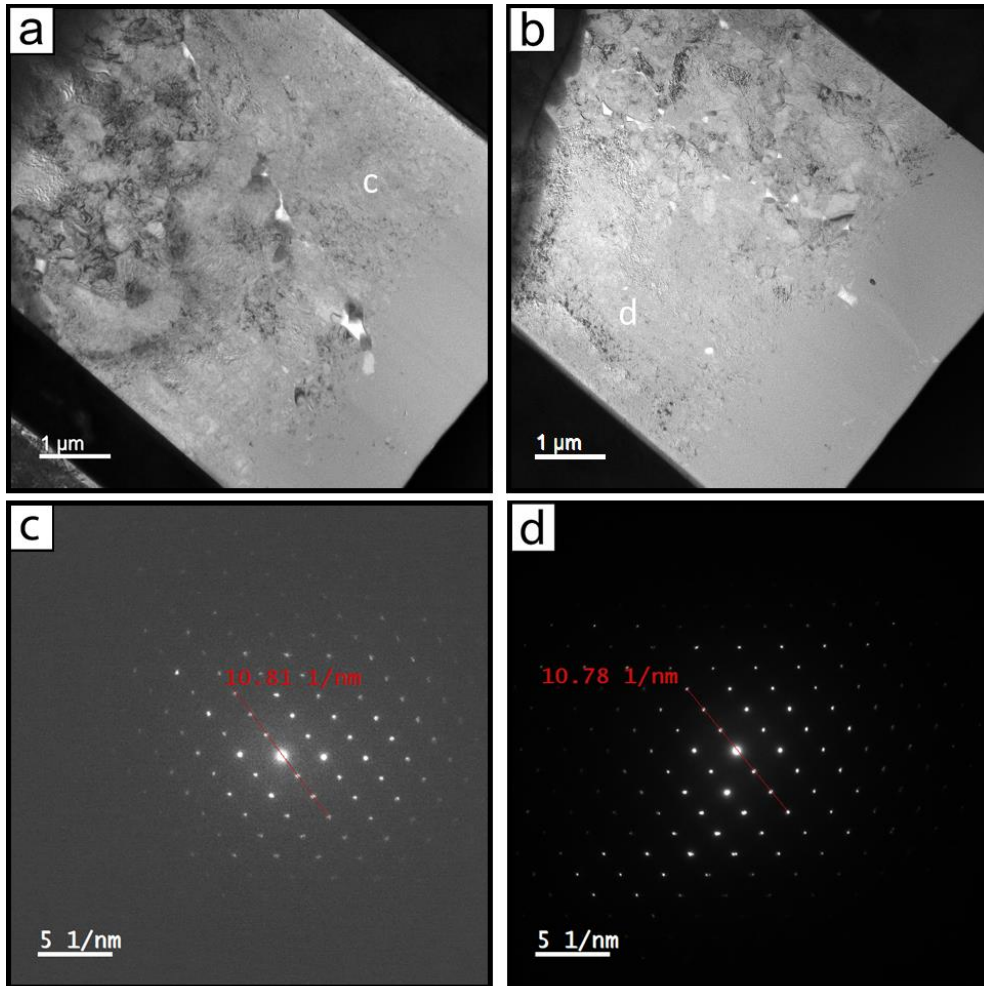


Figure 6 : a-b) Brightfield transmission electron microscope micrograph of focused ion beam of 10Phl thin-section, with location of selected area electron diffraction (SAED) images of parent quartz grain in 10Phl sample (659KA) with different intragrain misorientation in two different grains c) shows spreaded dots compared to d) as well as different lightning of the diffraction pattern.

V.2. Results

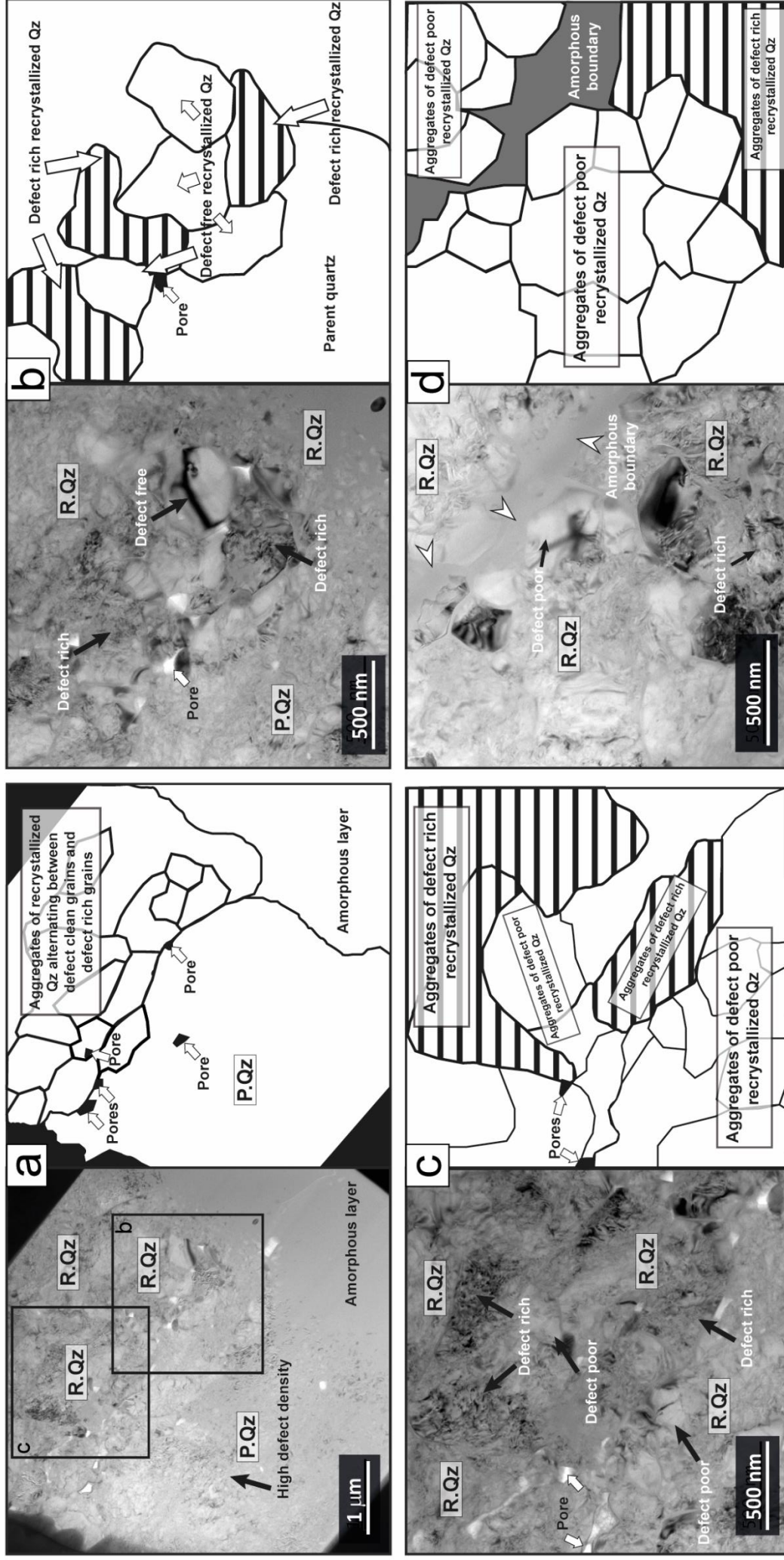


Figure 7: Brightfield transmission electron microscope micrographs of focused ion beam of 10PhI thin-section showing nanostructures in quartz and corresponding interpretative sketch.

a) Focused ion beam window of interest with a parent quartz (P.Qz) grains and aggregates of recrystallized quartz (R.Qz) grains. b-d) Aggregates of recrystallized quartz (R.Qz) grains alternating between defect free grains and defect rich grains, sometimes in contact with the parent grain b). In d), an amorphous grain boundary is visible and indicated with white arrows.

V.2. Results

Recrystallized quartz grains have a grain size from 1 μm to 100 nm and polygonal shape (Fig. 7b, d, c). Recrystallized grains, are often arranged in aggregates (500 nm to 1 μm in size) around parent grains. Among these aggregates, some grains are defects-rich whereas others are devoid of defects (Fig. 7b, c, d). From diffraction patterns, it appears that, in certain aggregates (including defect-rich and defect-poor grains, see Fig. 7b, c and 8a), the constituting small grains are misoriented of only 1-1.5°. These values are calculated with reference to the same zone axis from the tilt angles (α and β) imposed to the TEM across the grain using the equation proposed by [Qing, 1989](#). The orientation of recrystallized quartz grains in aggregates is often different from the orientation of parent grains (Fig. 9).

Pores, with a size of ~ 100 nm, large are observed at the interface between parent quartz grains and the recrystallized quartz (Fig. 7b, c, 8b). This is consistent with the high density of pores observed at the microscopic scale (Fig. 3b, and chapter IV) along grain and subgrain boundaries (Fig. 7a, b, c and 8b).

Grain boundaries are underlined by 5-80 nm thick contours composed of Si only (100%) lacking of diffraction pattern (white arrows in Fig. 7d, 8). This is interpreted as silica amorphous layer.

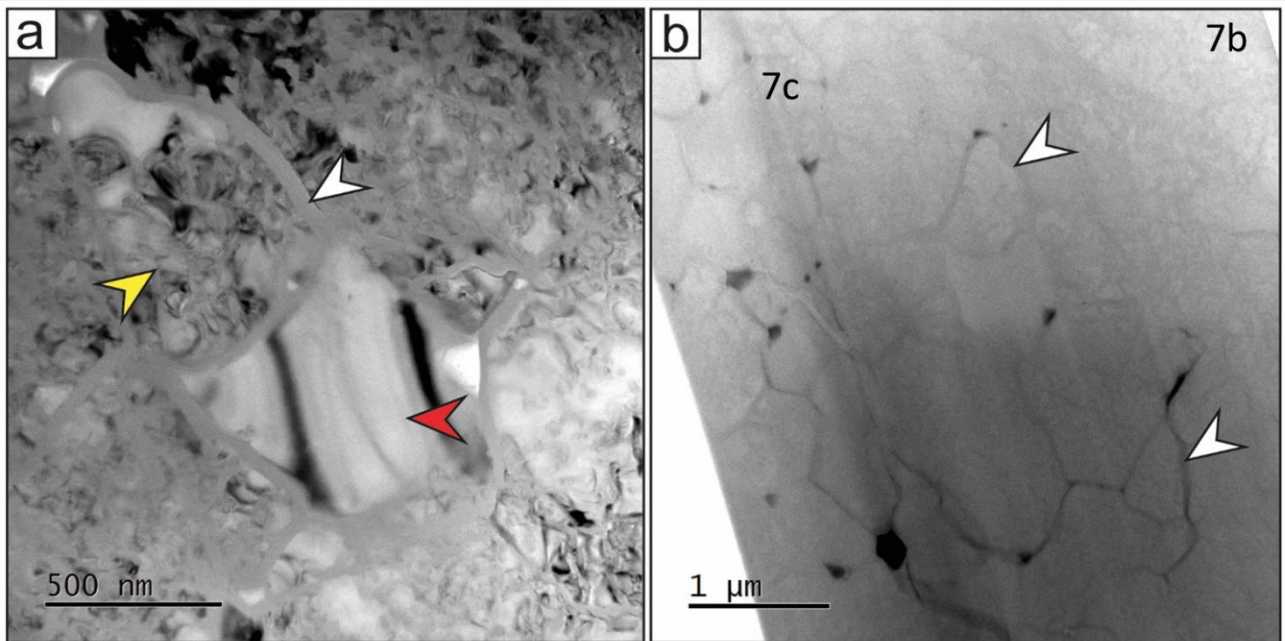


Figure 8: a) Bright field image of quartz recrystallized grains with white arrow indicating the amorphous grain boundary, red arrow shows the defects-poor grain and yellow arrow the defects-rich one. b) Dark field image of 10Phl sample with white arrows showing amorphous material with location of figure 7b and c.

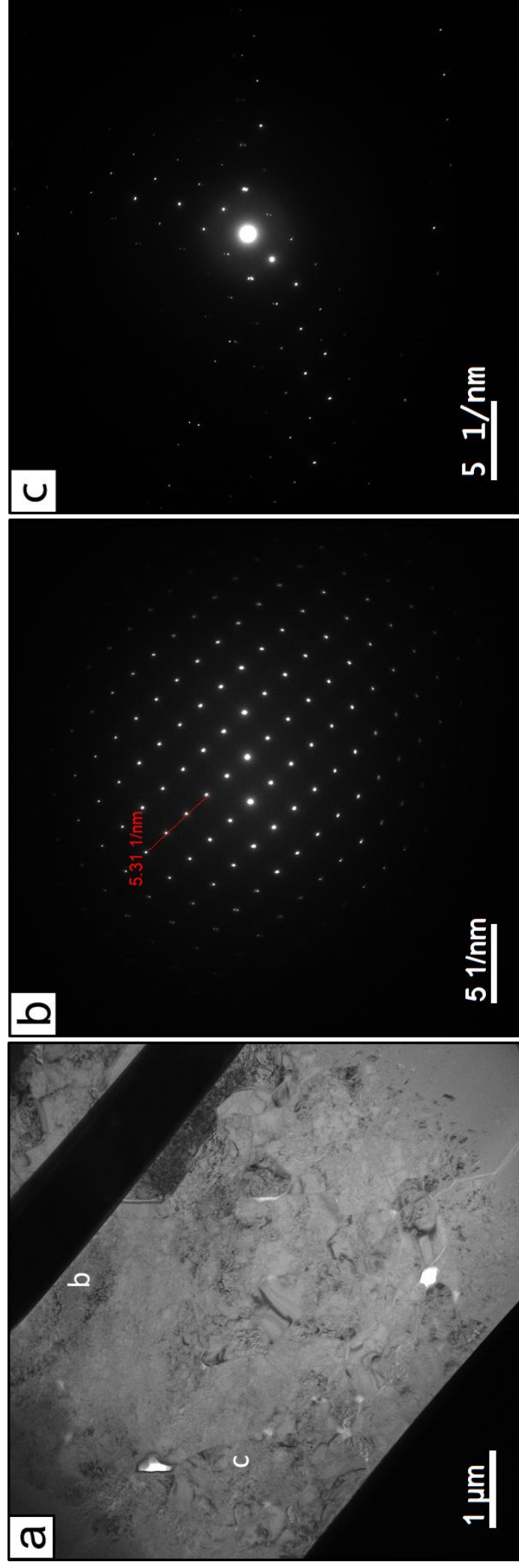


Figure 9: a) Brightfield transmission electron micrograph of focused ion beam of 10PhI thin-section, with location of selected area electron diffraction (SAED) images b) of a parent quartz grain and c) of a recrystallized quartz grain indicating different orientation between parent and recrystallized grains.

Nanostructures in mica

In mica-bearing areas, the grain size of mica grains varies from 125 μm (parent grains) to less than 5 μm (small grains seen at microscale in Fig. 3 and in chapter IV). At the nanoscale the largest mica grains observed (parent grains) have an average size of 7 μm while the smallest grain size is 300 nm (Fig. 10a, c and 13). Larger mica grains are kinked as shown by a periodic and symmetric change of color contrast in TEM images (Fig. 10a and b). Folding wavelength is of the order of 500 nm for mica crystallites 350-600 nm thick. Small micas (up to ~ 30 nm-thick) are visible in two nanostructural positions: (i) in opened grain boundaries among quartz (Fig. 10c) and (ii) in aggregates around larger micas (Fig. 10a, b, 13).

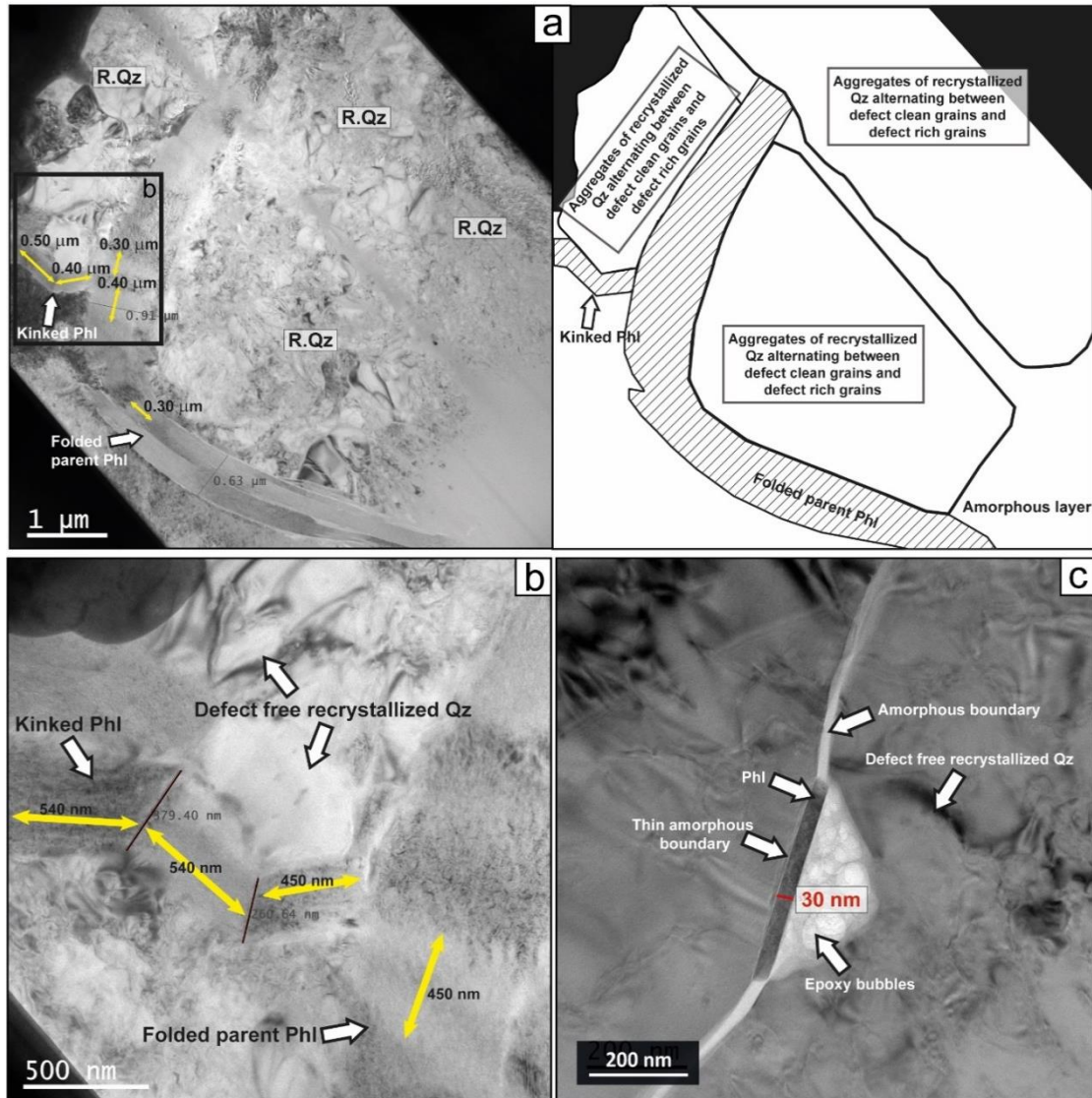


Figure 10: Brightfield transmission electron microscope micrographs of focused ion beam of 10Phl thin-section showing nanostructures in phlogopite. a) Folded parent phlogopite grain and its interpretative sketch, b) Zoom in the folded phlogopite grain and kinked phlogopite grain with yellow lines with arrows indicating the length of kink axes and distance between kink axes, c) Recrystallized mica grain located in amorphous layer between quartz grains, red line indicates the thickness of the mica. R. Qz: recrystallized quartz, Qz: quartz.

V.2. Results

Several intragrain defects are observed at TEM in mica crystallites:

- (1) lattice expansion parallel to the basal plane (A-site opening) (Fig. 11a and “delamination” in Fig. 11c). Here the regular distance between atomic layers of 1 nm is widened to 1.6 nm. Widening of the interlayer space is characterized by increasing electron density signal (thicker white layer in Fig. 12c, d).
- (2) dislocation in larger mica grains (Fig. 11d);
- (3) in the most deformed crystallites, delamination defects are arranged in ripples aligned along two axes at an angle of $\sim 30\text{-}40^\circ$. They disappear simultaneously when tilting the sample (Fig. 11a-c);

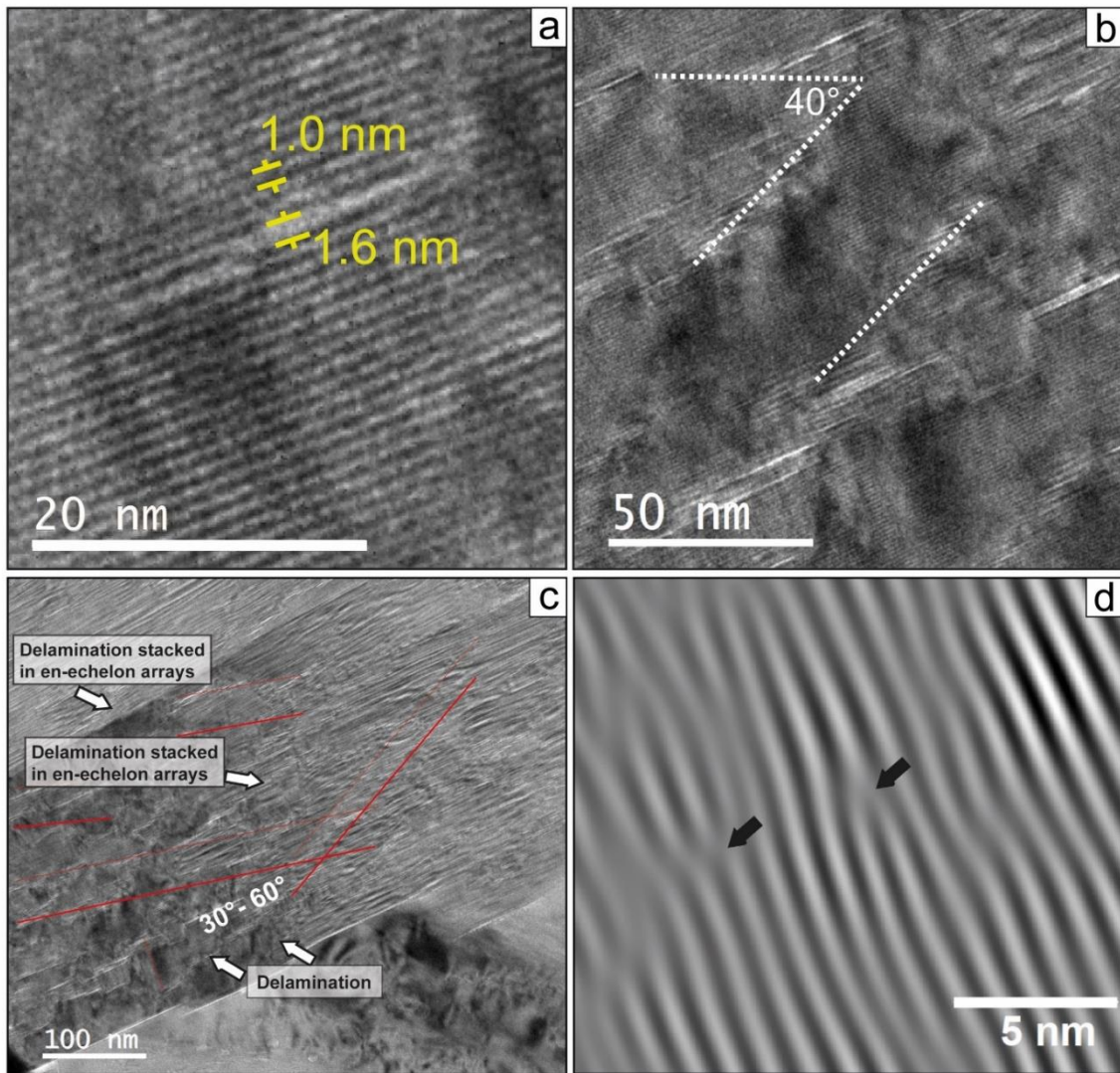


Figure 11 : High resolution brightfield transmission electron microscope micrographs of focused ion beam of 10Phl thin-section showing intragrain defects in phlogopite. a) Dilatancy site with lattice expanding from 1.0 nm to 1.6 nm (=delamination defects). b) Spatial arrangement of delamination defects (i.e. ripplocation-like structures). c) Diamond-shaped expansion structures arrays, with acute angles between 30° to 60° . Bright streaks correspond to delamination structures. Red lines show main directions of aligned delamination and in the zoom the length of expansion structure, d) Fast Fourier transform (FFT) filtered image of a high resolution brightfield image with dislocations indicated by black arrows showing distortion of basal planes.

V.2. Results

- (4) triangular defects with higher refraction contrast than phlogopite, along the crystallite boundaries (Fig. 12a). They appear in bright field images and show no atomic layers inside. In each side of the triangular defect, the phlogopite atomic layers are misoriented of $\sim 5^\circ$ (Fig. 12a). These defects are therefore associated to low angle grain boundaries (LAGB in Fig. 12a);
- (5) amorphous layers parallel to sub-parallel to the basal plane evidenced by a higher electron density due to a lack of crystallographic planes (Fig. 12b-d). At each side of the amorphous layer, the atomic layers of mica are misoriented of $\sim 4^\circ$ (Fig. 12b);

Small mica grains are deformed as well exhibiting intragrain defects similarly to larger mica grains, however their size and the resolution prevent us from observing in details each type of intragrain defects.

V.2.9. Phlogopite composition

Energy dispersive spectroscopy (EDS) point analysis reveal composition differences between the large (~ 500 nm in average) and small (~ 50 nm in average) phlogopite grains. Small grains exhibit larger variations in the chemical composition than larger grains and are systematically Fe-richer, Mg and Al-poorer (Fig. 13c, d, e). In small grains, Si/Al ratio varies from 5 to 9, while large grains values are clustered around 5 (Fig. 13d). Mg/Al ratio varies more in small grains as well with values comprised between 2.45 to 3.75, conversely in large grains Mg/Al ratio is in average 3. In small grains Mg/Fe ratio varies between 2.9 to 6.1, while in large grains values are comprised between 3.9 to 5.2.

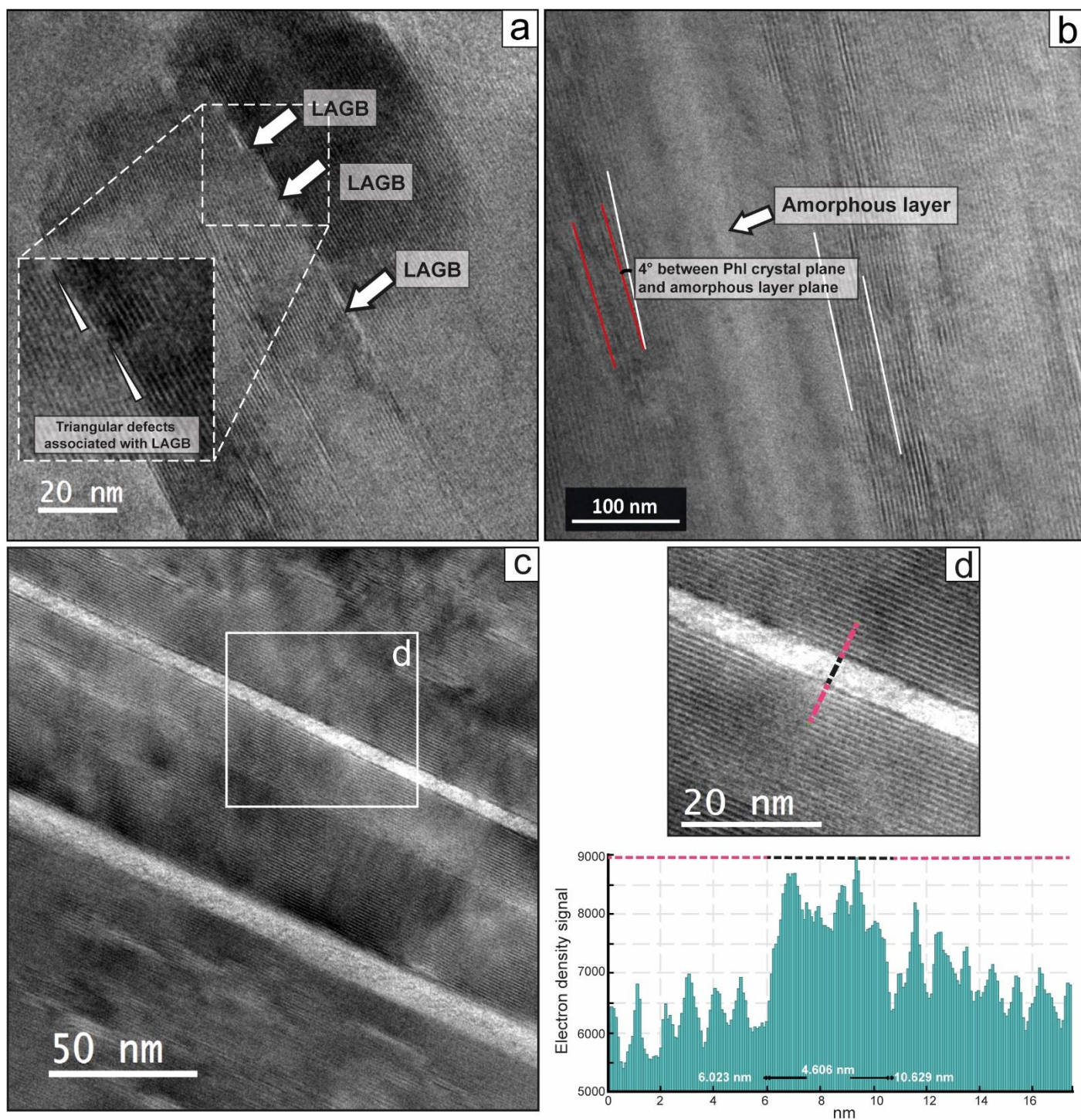


Figure 12 : High resolution brightfield transmission electron microscope micrographs of 10Phl sample showing intragrain defects in phlogopite. a) Low-angle grain boundaries (LAGB) with zoom on triangular defects associated with it, b) zoom in phlogopite grain with an amorphous layer showing different plane than phlogopite crystal plan with a 4° angle difference, red lines indicate phlogopite crystal plane and white lines indicate amorphous layer planes. c) Crystal planes and amorphous layers and d) closer look in the shift from crystal plane to amorphous layer with a profile cutting both layers and showing electron density signal variations in corresponding plot.

V.3. Discussion

V.3.1. Quartz deformation processes

Micro and nanostructures in the Phl-poor (10Phl) sample indicate that strain is accommodated in quartz at least partially by plastic deformation. Plastic deformation is attested by the existence of core and mantle like structures at microscopic scale (as the ones describe by [White, 1976](#)), by parent quartz grains with elongated sigmoidal shapes and higher shape factors and SPO than starting grains. The distribution of quartz misorientation axes across low-angle boundaries shows dominant prism $\langle a \rangle$ slip system (Fig. 4b), and the homogeneous grain size of recrystallized grains indicate that quartz experienced partial dynamic recrystallization by subgrain rotation (SGR). This mechanism (also known as regime II) is observed in experimental samples at P-T conditions of 15kbar and 600-1100°C ([Hirth and Tullis, 1992](#); [Stipp and Tullis, 2003](#)), while in natural samples at P-T conditions from 2.5-3kbar 450-500°C ([Stipp et al., 2002](#)) down to 350-380°C depending on mica and fluid contents in the bulk rock ([Alaoui et al., 2023](#)). At the nanoscale, plastic deformation is attested by the high density of defects in parent grains, and misorientation in the parent grains (Fig. 7). However, we do not observe any clear evidence of nanostructures related to dynamic recrystallization as the one observed by [Hirth and Tullis \(1992\)](#): no clear subgrains, no initiation of grain boundary migration. We do not observe dislocations aggregates gathering at grain boundaries defining polygonal domains as to be expected for dominant dynamic recrystallization ([Hirth and Tullis, 1992](#)). Furthermore, the recrystallized grains in our samples have very contrasted densities of dislocations (from high density, such as parent grains to very low densities) conversely to [Hirth and Tullis \(1992\)](#). These observations suggest that plastic deformation resulting in dynamic recrystallization is present but it is not the dominant deformation mechanism in quartz deformation.

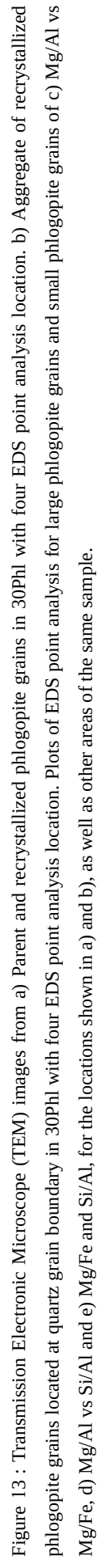
Finally, compared to EBSD, the recrystallized grains seen at TEM are substantially smaller (at the EBSD the average grain size is $\sim 1.41 \mu\text{m}$ for the smallest grains (Fig. 4a), compared to 100 nm observed at TEM) but organized in patches with misorientations $< 2^\circ$. Therefore, the grains will be classified as subgrains, as in EBSD grains were defined as areas delimited by high-angle ($\geq 10^\circ$) boundaries ([Halfpenny et al., 2012](#)) and a minimum of 5 indexed consecutive pixels on several rows. Low-angle

V.3. Discussion

boundaries are identified as segments within the grains showing a misorientation angle $\geq 2^\circ$ between two pixels (Trimby et al., 1998). This implies that the "grains" detected by EBSD are actually collections of much smaller nanometric grains with common crystallographic orientation. Indeed, EBSD can detect small misorientation angles ($<1^\circ$), but it analyzes a volume of $1\ \mu\text{m}^3$, therefore a collection of grains/subgrains, if the latter have a size of 100 nm. Consequently, the difference between TEM and EBSD is not an issue with precision in the measurement of crystallographic orientation, but an issue of spatial resolution, which explains why we do not see these recrystallized grain patches in EBSD but we do in TEM.

The recrystallized domains also show composition variations relative to parent grains: they have a strong blue luminescence, as well as a high Al content in comparison with the starting material. Considering that intra-crystalline Al diffusion in quartz is too low at the timescale and temperature of the experiments, the sole mechanisms that may lead to the Al enrichment during recrystallization is dissolution-precipitation. Please note here that grain boundary migration is a process based on dissolution precipitation, as the material forming one grain is dissolved and reprecipitated immediately and virtually at the same location, but in the adjacent grain. Therefore, dissolution-precipitation may account for the defect-free grains that were observed in recrystallized aggregates (Fig. 7) as well as their enrichment in Al. By comparing CL, EBSD and nanostructures we propose that areas of recrystallized grains (Fig. 4c) are actually made of a mixture of grains that are dynamically recrystallized by subgrain rotation (Fig. 4b and d) and grown by pressure-solution (Fig. 4a, Fig. 7).

Therefore, recrystallization of quartz encompasses two main processes acting together and imbricated at the smallest scale: dynamic recrystallization and dissolution-precipitation, the latter giving rise to the blue cathodoluminescence signature and Al enrichment. Solution-transfer fluid film diffusion at grain boundaries (dissolution-precipitation) is known to be enhanced by phyllosilicates in a quartz + mica assemblage compared to a pure quartz assemblage (Farver and Yund, 1999; Anzalone et al., 2006; Meyer et al., 2006; Green et al., 2009), which can account for the very large proportion of Al-enriched/CL-blue recrystallized quartz in the mica + quartz deformation experiments analyzed here.



V.3.2. Phlogopite deformation processes

In mica, deformation is accommodated by kinking and folding (Fig. 3), which are frequently interpreted as due to basal slip (Etheridge et al 1973; Bell et al., 1986; Meike 1989; Kronenberg et al., 1990; Mares and Kronenberg, 1993). We do not observe any evidence of basal slip, as we do not observe any basal dislocation (dislocation in (001) glide plane), compared to Meike (1989) and Kronenberg et al. (1990).

Triangular defects, associated with LAGB, with higher refraction contrast are found along crystallite boundaries in phlogopite, (Fig. 12a), similarly to the ones described in chlorite by Kogure and Murakami (1998) and Airaghi et al. (2020b). The development of LAGB has been recognized to be due to growth defects during layer-by-layer growth on the free crystal surface in phyllosilicates (Kogure and Murakami 1998). Triangular defects associated with LAGB observed in phyllosilicate crystallites precipitating from a solution can result from growth defect of mica grains or from the deformation (Kogure and Murakami 1998; Airaghi et al., 2020b).

We suggest that these triangular defects may be the initial stage of high-density defects which might evolve in amorphous layers with a low angle to the basal plane (Fig. 12a-c). LAGB are aligned parallel to the atomic planes of crystallites as the continuous amorphous layers (Fig. 12a). Misorientation angles at each side of the LAGB ($\sim 5^\circ$) is similar to the one observed at each side of the continuous amorphous layers. We suggest that continuous amorphous layers may form by merging high densities of LAGB. This evolution in defects distribution also support the idea that LAGB observed in our samples are mainly related to deformation.

The viscosity of amorphous material is indeed orders of magnitudes lower than the one of crystalline mica (for quartz see Di Toro et al., 2004). The existence of amorphous layers subparallel may favor kinking or favoring slip (as in Fig. 12b-d). The variable orientation angles between amorphous layer and basal planes suggest that kink accommodates deformation.

Expansion of the interlayers also reveal the existence of some dilatancy in micas. This could be linked to local differential stresses at the Phl-Qz boundary (Aslin et al., 2019). As can be deduced from the fact that defects disappear simultaneously when the sample is tilted, defects are arranged in ripples which

are aligned along two axes at an angle of about 30° and included in the same crystallographic plane (Fig. 11c). These defects are similar to the “ripplocations” mentioned in the studies by [Aslin et al. \(2019\)](#) and [Airaghi et al. \(2020\)](#). Rippllocations represent a mechanism in phyllosilicate that is able to explain the accommodation of strain normal to atomic layers without brittle damager (Fig. 11a-c). They can potentially accommodate large amount of elastic strain energy release due to the accumulation of layer-normal strain in ([Aslin et al., 2019](#)). In fact, rippllocations may evolve into kinking as the basal plane parallel compression energy allows rippllocations to migrate and merge, forming a kink band. This strain can cause kinked microstructures in deformed mica suggesting that kinking can be either a result of basal glide or rippllocations (Fig. 14, [Aslin et al., 2019](#)).

As we do not observe any evidence of basal slip, following the model of [Aslin et al., 2019](#) (Fig. 14) we suggest that in our samples rippllocations lead both to kink bands and conjugate expansion arrays.

Dissolution-precipitation involve a compositional change between the parent and product grains ([Etheridge and Hobbs, 1974](#); [Putnis, 2002](#); [Putnis and Putnis 2007](#); [Ruiz-Agudo et al., 2014](#)), as observed in the chemical analyses we carried out, which show two populations of micas (Fig. 13). The newly-grown micas have a much larger variability in composition than the starting ones, probably reflecting the influence of the local composition of the minerals and the fluid phase.

Beyond intragrain defects in phlogopite, variations in the chemical composition between large and small mica grains along with the microstructural position of small mica grains (in opened quartz grain boundaries, cracks or around large mica grains) point to dissolution-precipitation of mica, with variable transport distances from nanometric scale (*insitu* to the large mica grains) to micrometric scale (far from large mica grain in opened quartz cracks). Hence, kinking and dilatancy in mica grains is accompanied by dissolution-precipitation, the main mechanism of mica grain size reduction. As other defects do not suggest grain size reduction by dynamic recrystallization nor by mechanical grain size reduction resulting from kinking, we infer that grain size reduction is mainly due to dissolution-precipitation. Our results agree with what [Aslin \(2019\)](#) observes, that is dissolution of biotite in natural mylonites was concentrated in regions of high internal strain energy (kink band boundaries) and along grain boundaries.

Conversely to what observed [Wilson and Bell \(1979\)](#), in our samples, micas are not strain-free. Phlogopite are thus characterized by dissolution-precipitation creep and by no intracrystalline plastic deformation. TEM observations pointed that dissolution-precipitation was identified in our experiments as the main mechanism of mica grain size reduction, conversely to quartz where grain size reduction is linked to both dynamic recrystallization and dissolution-precipitation.

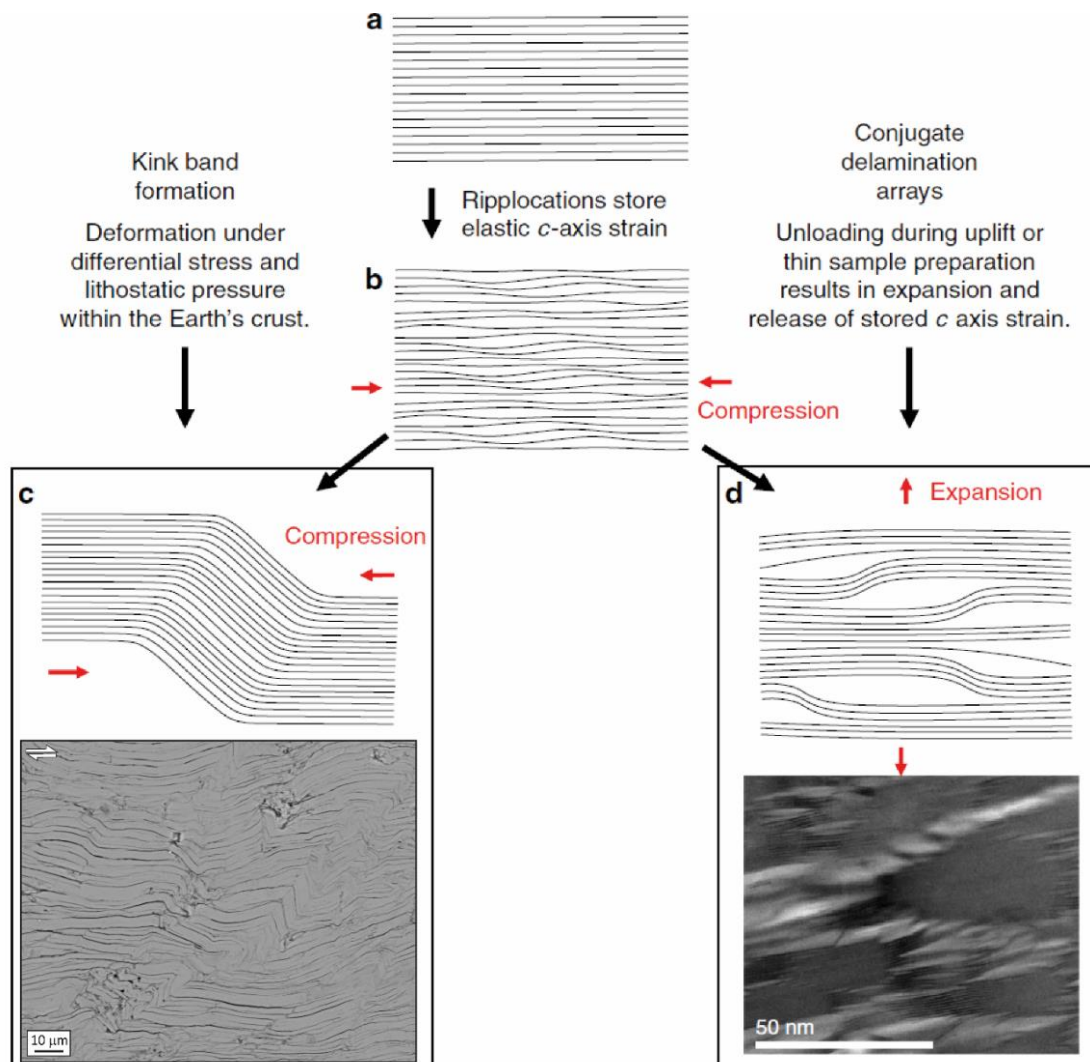


Figure 14 : Conceptual model modified from [Aslin et al., 2019](#) showing how ripplocations lead to kink bands or conjugate expansion structure arrays with a) an undeformed lattice experiences differential stress due to compression parallel to the basal planes, resulting in numerous small bulk ripplocations that store the resulting *c*-axis parallel strain energy. b) The basal plane parallel compression generates sufficient energy for ripplocations on different layers to migrate and merge, forming a kink band between two opposite sign boundaries. c) The elastic *c*-axis strain energy transforms into permanent strain on the formation of mature kink bands, potentially resulting in the kinked microstructures found in micas from deformed rocks. d) Biotite lattice parts without kinking contain ripplocations containing elastic *c*-axis strain energy. TEM analysis reveals conjugate arrays of expansion structures and delaminations, facilitating grain-scale parallel expansion.

V.4. Conclusion

In summary, we have investigated micro- to nanostructures of quartz and mica to decipher deformation processes and strain accommodation at grain to atomic scale.

From this study the following conclusions can be drawn:

- (1) recrystallization in quartz proceeds through a combination of dynamic recrystallization by subgrain rotation (SGR) and grain-boundary migration by dissolution-precipitation. Aggregate of submicron-sized grains, sharing similar orientations, are a mixture of defect-rich and defect-free grains (100's of nm to 1 μm in size), resulting presumably from subgrain rotation and grain-boundary migration, respectively. Grains identified by EBSD are, in reality, groups of considerably smaller grains that share a common orientation in crystallography. Similarly, the CL blue and bright luminescence, as well as the enrichment in Al, observed in aggregates composed of the submicron-sized grains is presumably the consequence of those of grains that grew by grain boundary migration.
- (2) Major defects observed in mica are dislocations, triangular defects (associated with LAGB), amorphous layers, lattice expansion (delamination), and ripplocations, highlighting no intracrystalline plastic deformation (except locally with kinking), and chemical variation between parent grains and product grains, dissolution-precipitation creep. However, in the case of mica, dissolution-precipitation is the only mechanisms responsible of the grain size reduction.

Preface

This chapter presents a comparative study on experimentally deformed mica-quartz assemblages with different starting material than the samples presented in the chapters IV and V.

It was intended to explore different compositions of the starting micaceous material in order to investigate the role of composition and stability of mica on the development of shear zones, mechanical behavior and strain accommodation.

The systematic study of the mechanical and reactional behavior of different type of biotite-rich systems may therefore provide evidence upon the impact that protolith composition in upper and mid crustal shear zones may have on the deformation of crustal rocks.

Chapter VI – Effect of mica composition on the
strength of mica-quartz assemblage: a
comparison between fluorine-rich phlogopite,
phlogopite and Fe-Mg biotite

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VI.1. Introduction

Micas are ubiquitous in crustal shear zones and strongly influence mechanical response of protoliths. Several experimental studies examined the role of mica on the mechanical behavior of polyphase aggregates (Gottschalk et al., 1990; Shea and Kronenberg, 1992, 1993; Tullis and Wenk, 1994; Rawling et al., 2002; Tokle et al., 2023), highlighting that mica weakens the rock strength by decreasing the CPO of quartz, preventing quartz dynamic recrystallization and by developing weak layers of interconnected micas (Tokle et al., 2023). The weak mechanical behavior of phyllosilicates is primarily attributed to the highly layered and, consequently, anisotropic structure of their crystal lattice that may promote deformation parallel to the basal plane (Kronenberg et al., 1990). In mica structure, interlayers are filled by cations as K^+ (muscovite and biotite), Na^+ and Ca^{2+} that balance charges (Aslin et al., 2019). Phyllosilicates also promote solution-transfer diffusion creep in quartz aggregates (Renard et al., 1997; Farver and Yund, 2000; Anzalone et al., 2006; Meyer et al., 2006; Greene et al., 2009). However, different types of mica exist. The main difference consists in the cations filling the interlayer (K^+ , Na^+ , Ca^{2+}) and the octahedral sites (Al^{3+} , Mg^{2+} , Fe^{2+} , Fe^{3+} , Ti^{4+}) as well as halogens replacing the OH groups (as fluorine). These differences seem to affect the intrinsic strength of mica. In deformed natural samples, microtextures suggest that biotite is weaker than white mica, (Wilson and Bell, 1979; Bell et al., 1986) and that biotite is more susceptible to fluid-mediated dissolution/recrystallization and solid-state recrystallization than white mica (Wilson and Bell, 1979; Bell et al., 1986). In fault zones containing mylonites, muscovitic mica fish tend to be preserved, conversely accompanying biotite often exhibits significant deformation or recrystallization (Dahl and Dorais, 1996). Experiments involving shortening of single mica crystals across a broad range of temperature and confining pressure also indicate that muscovite is mechanically weaker than biotite under similar experimental conditions (Kronenberg et al., 1990; Mares and Kronenberg, 1993).

All these observations suggest that the chemical composition of mica may play a crucial role on his aptitude to deform and hence on rock strength. Dahl and Dorais, 1996, for example, proposed that (001) dislocation glide in biotite is rate-limited by its fluorine content. Hence, a significant substitution,

F(OH)₋₁, coupled with the simultaneous removal of hydroxyl H⁺ towards the interlayer cavity, can enhance the mechanical strength of biotite through two mechanisms: (1) elimination of K⁺-H⁺ repulsion, thereby reinforcing the interlayer bonds, and (2) enabling K⁺ to penetrate further into the interlayer cavity, resulting in a geometry less favorable to basal slip (Dahl and Dorais, 1996). However, variations in the chemical composition of mica may not only modify the intrinsic mechanical resistance of mica itself but also their stability field, with major consequences on the stability of the entire deforming assemblage. Fluorine, for example, is the most soluble halogen in silicate melts by replacing oxygen in tetraedra and by coordinating with aluminum, it triggers depolymerization of the glassy network, which decreases the viscosity of silicate melt (Grousset et al., 2015). Fluorine solubility decreases with increasing proportion of silica (Mysen and Richet, 2005). Dissolution of a F-rich mica could then result in a more acid fluid (then in the case of a F-depleted mica) at grain boundaries. This may affect the solubility of quartz and ions diffusion rates, as observed for Ca²⁺ enriched solutions (Alcantar et al., 2003; Anzalone et al., 2006; Meyer et al., 2006; Greene et al., 2009).

In F-bearing solutions, the solubility of silicon (Si) increases as well as the one of Si-F compounds, suggesting that fluorine (F) may facilitate the breakdown of the silicon-oxygen (Si-O) bond in silicates, thereby releasing silicon into fluids (Xing et al., 2019). Complexing in F-rich fluids can also help promote element exchange through the dynamic interfacial reaction zone. This implies that F might enhance reaction kinetics and make selective element transportation that further controls the reaction texture and product phase composition (Duan et al., 2023).

Despite all these results, the effect of composition variations of mica on the strength of mica-rich rocks is rarely investigated and not quantified. In particular two questions remain unanswered: (1) is the type of mica the dominant factor controlling the rock strength? (2) do different types of mica affect the behavior of quartz and hence microstructures?

This study compares, by the means of shear experiments at high pressure and high temperature conditions (Griggs-type apparatus), the mechanical strength of mixtures made of quartz and mica assemblages deformed at the same conditions and equal mica proportions (30% vol. mica), but using

different types of biotite: a fluorine-rich phlogopite (F-Phl, F up to 5 wt. %), a Fe-Mg biotite (Bt) and the F-depleted phlogopite (Phl, F < 0.57 wt. % for undeformed Phl single crystal) presented in chapters IV and V. We analyzed the microstructures and mineral composition in experimentally-deformed samples using SEM, EPMA and CL, in order to explain difference in strength and to decipher the deformation processes and the chemical interactions between the two phases.

VI.2. Results

Shear experiments (Griggs apparatus) were performed using mica-quartz assemblages from powders made of 30% vol. mica and 70% vol. quartz, at 750°C and 800°C, 1.0 GPa and bulk strain rate $\sim 10^{-5} \text{ s}^{-1}$, with variable mica grain sizes (conditions details are listed in Table 1). Experimental procedure is the same as the one described in chapters III and IV. Most experiments are deformed to a shear strain higher than 4, while some experiments are undeformed since that they were stopped before reaching hit-point (i.e. undeformed F-Phl at 700°C, 10 kbar, Table 1) or stopped during the pumping stage (undeformed Bt at 500°C, 6 kbar, Table 1). It is important to note that in the F-Phl and Bt-bearing mixtures, Li-rich quartz from La Gardette, Oisans, Alps is used while in the Phl-bearing mixtures it was used the milky quartz from Aar massif (central Alps, see chapter III).

VI.2.1. Mechanical results

The mechanical behavior and bulk strength of the quartz-mica mixture varies according to the type of mica in the assemblage (Fig. 1a, b).

F-Phl-bearing samples systematically show higher values of peak differential stresses (higher bulk strengths) than all other samples. Furthermore, most F-Phl samples deform above Goetze criterion (differential peak stress $\geq$ confining pressure) with peak stress between 1090-1341 MPa except the fine grain sample (grain size of 0-63 μm and a peak stress of 878 MPa, Fig. 1a, b). Samples with Bt are as weak as the Phl-bearing ones, but significantly weaker than the ones with F-Phl (peak stresses of 320-392 MPa, Fig. 1a). It is worth noticing that the replica of Phl-bearing experiment shows a strong behavior (as strong as F-phlogopite), incoherent with other Phl or Bt-bearing samples (Fig. 1a, b).

Figure 1 : Mechanical data of the experiment series. a) Stress-strain evolution for mica-quartz assemblage experiments, dashed curve corresponds to an experiment performed on the Sanchez Griggs apparatus. All experiments are 30% vol. mica and 70% vol. quartz. b) Effective viscosity-mica proportion evolution, dashed lines correspond to iso-strain rate/iso-stress models using 100Qz-BR from Richter et al., 2018 for $n = 3-5$ and calculated for differential stresses at $\gamma=2.5$. Confining pressure is 1.0GPa, temperatures are 750°C and 800°C and strain rate is $\sim 10^{-5} \text{ s}^{-1}$. Quartz grain size is 10-20 μm for all experiments, mica grain size is indicated for each on the plot. H₂O content is 0.1% wt.

VI.2. Results

Table 1 : Experimental conditions for constant shear strain experiments with only Rig stiffness correction applied on the displacement. Area correction $\cos^2$ is applied on the stress. Average displacement, strain rate, stress and viscosity are calculated for a shear strain at 2.5, for experiment at lower shear strain the final one was applied.

Experiment	Grain size μm	Mica proportion		Total displacement stiffness corrected mm	Time		Displacement rate vertical* mm/s	Shear displacement rate* mm/s	Final thickness* mm	Shear strain rate* s^{-1}	Equivalent strain rate s^{-1}	Effective viscosity		Equivalent stress	
		vol.			hours	seconds						Pa s		MPa	Pa
100Phl	63-125	1	100%	2.06	35.71	128563	1.49E-05	2.10E-05	0.47	4.48E-05	2.59E-05	1.63E+13		421	421000000
70Ph	63-125	0.7	70%	1.26	23.00	82803	1.52E-05	2.16E-05	0.98	2.20E-05	1.27E-05	4.34E+13		552	551620000
50Phl	63-125	0.5	50%	1.84	31.95	115020	1.60E-05	2.26E-05	1.18	1.91E-05	1.10E-05	4.28E+13		473	472687000
30Phl	63-125	0.3	30%	2.00	34.34	123620	1.57E-05	2.22E-05	0.82	2.71E-05	1.56E-05	2.24E+13		350	350000000
30Phl-bis	63-125	0.3	30%	2.07	36.94	132970	1.36E-05	1.93E-05	0.62	3.11E-05	1.80E-05	5.59E+13		1004	1004000000
20Phl	63-125	0.2	20%	2.00	35.45	127603	1.46E-05	2.07E-05	0.70	2.95E-05	1.71E-05	4.69E+13		800	800000000
10Phl	63-125	0.1	10%	2.04	36.26	130552	1.49E-05	2.10E-05	0.83	2.53E-05	1.46E-05	6.58E+13		961	961000000
100Qz-BR**	0-100	0	0%	2.48	44.65	160729	1.49E-05	2.10E-05	0.75	2.81E-05	1.62E-05	5.31E+13		860	860000000
F-Phl	125-200	0.3	30%	5.35	88.61	319000	1.42E-05	2.01E-05	0.71	2.84E-05	1.64E-05				
F-Phl	125-200	0.3	30%	3.22	75.82	272942	1.38E-05	1.95E-05	0.80	2.44E-05	1.41E-05				
F-Phl	125-200	0.3	30%	3.20	52.78	190015	1.55E-05	2.19E-05	0.75	2.91E-05	1.68E-05				
F-Phl	0-63	0.3	30%	2.51	53.38	192156	1.23E-05	1.74E-05	0.70	2.48E-05	1.43E-05	2.95E+13		423	423000000
Bt	125-200	0.3	30%	2.20	37.28	134193	1.57E-05	2.23E-05	0.67	3.32E-05	1.92E-05	1.73E+13		332	332000000
Bt	0-63	0.3	30%	2.21	36.70	132123	1.60E-05	2.27E-05	0.67	3.38E-05	1.95E-05	1.64E+13		321	321000000

*calculated on averaging values around a shear strain of 2.5, averaging from 2.48 to 2.54.

**Richter et al., 2018

Different shapes of the mechanical curves are observed between Bt, F-Phl and Phl bearing samples. While curves for Bt show a quasi-steady state behavior (a plateau) after peak stress, the ones for F-phlogopite show rapid differential stress decrease after peak stress (Fig. 1a). Force-time plot (Fig. 1b and 2) allows imaging the force drop in these last samples and hence discriminate whether such sharp weakening for F-phlogopite may be due to slip at forcing blocks interface over time or to mechanically and chemically-driven weakening processes within the assemblage. As detailed in chapter III, slipping at forcing blocks interface is characterized by a discontinuous and abrupt stress and force drop, and apparent slip on only one side of the sample after cutting the assembly with a gap on the other side of the shear surface (arrow pointed in number 6 on Fig. 2, for a detailed and zoomed image see chapter III). A homogeneous sample between forcing blocks indicates instead that no slip occurred between the pistons and the sample interface (number 5 on Fig. 2). It is also common to consider slipping when the sample deforms above Goetze criterion ([Mansard, 2019](#)), but it is not systematic as we do not observe slipping in Phl examples even though they deform above Goetze criterion (number 5 and 6 in Fig. 2).

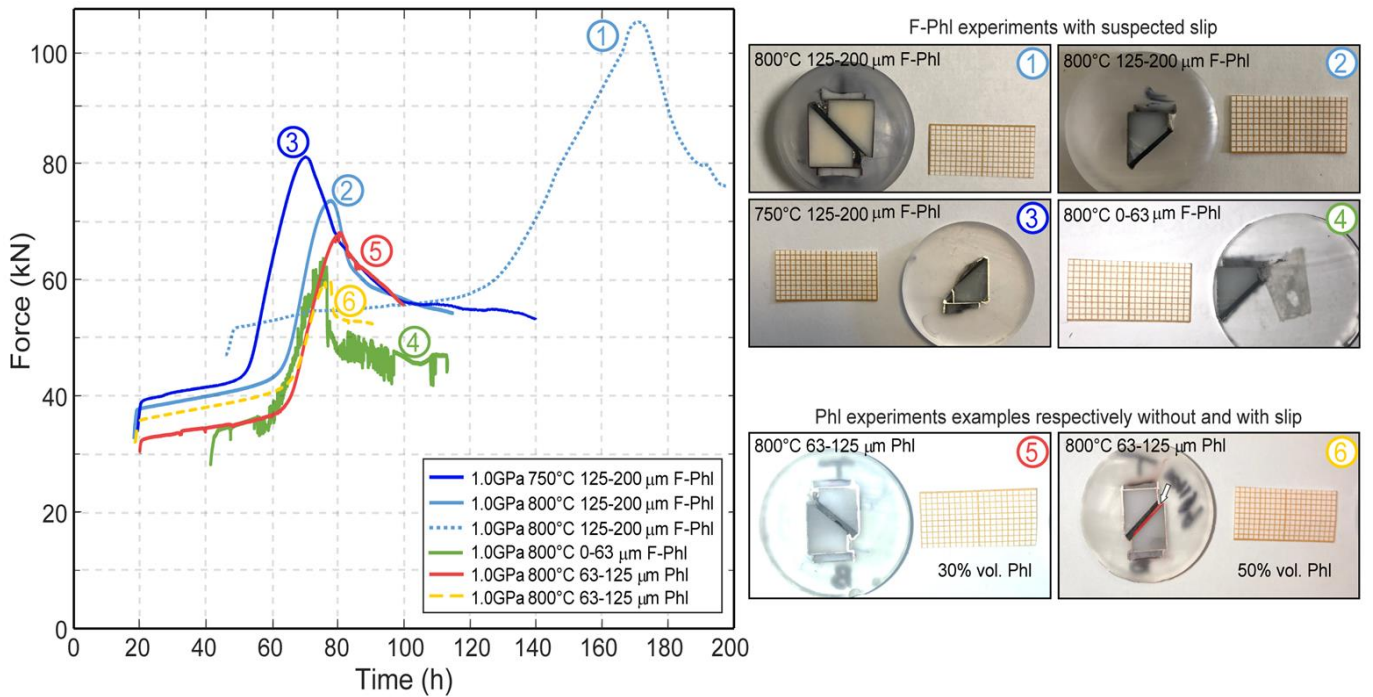


Figure 2 : Force-time evolution for mica-quartz assemblage experiments with corresponding samples cut parallel to X-Z plane, to exhibit slip and no slip. Arrow on corresponding sample 6 shows the slip interface gap. Dashed curve corresponds to an experiment performed on the Sanchez Griggs apparatus. All experiments are 30% vol. mica and 70% vol. quartz. Confining pressure is 1.0GPa, temperatures are 750°C and 800°C and strain rate is $\sim 10^{-5} \text{ s}^{-1}$. Quartz grain size is 10-20 μm for all experiments, mica grain size is precised for each on the plot. H_2O content is 0.1% wt.

Typically, results for the experiment with the fine grained (0-63 μm) F-Phl attest of the most noticeable slip, with a force decrease of 10 kN in 3 hours (for a total duration of the experiment of 75-78 hours). Other F-Phl experiments show instead a force decrease of ~ 20 kN in ~ 7 -10 hours that, along with ruptures observed in the sample assembly (Fig. 2) make us also suspect to have experienced slip. We therefore decided to not considered data after peak stress for F-phlogopite-bearing assemblages, and hence not to calculate viscosity at $\gamma \sim 2.5$ for these samples, as done in chapter III. For the other experiments, the effective viscosity (η) was calculated for $n = 3$ -5 by the following equation (Paterson and Olgaard, 2000):

$$\eta = \frac{\sigma_{eq}}{\varepsilon_{eq}} \text{ with } \sigma_{eq} = 2 \times \tau = \Delta\sigma \text{ and } \varepsilon_{eq} = \frac{\dot{\gamma}}{\sqrt{3}}$$

where σ_{eq} is the equivalent stress, ε_{eq} is the equivalent strain rate, τ is the shear stress, $\Delta\sigma$ is the differential stress and $\dot{\gamma}$ is the shear strain rate. Data from chapter IV are plotted on Fig. 1c (colored for only sample with 30% vol. of phlogopite) along with the ones for Bt-bearing experiments. The effective viscosity of Bt-bearing samples is similar to Phl-bearing ones (excluded the replica, see above; Fig. 1c).

In summary, experiments with F-phlogopite are stronger than the ones with Mg-Fe Bt and Phl since deformation begins for these samples at higher differential stresses. In the following, we document microstructures in order to investigate the causes of such differences.

VI.2.2. Microstructures

In undeformed experiments, an initial foliation made of layers of mica and quartz is observed, necessarily acquired during loading and the compaction steps (Fig. 3a, d). In all undeformed samples, this foliation is weakened in deformed micas and micrographs exhibit a fabric of quartz with interconnected layers of quartz (Fig. 3b, c, e, f). Microstructures in experiments with F-Phl, Bt and Phl are overall similar in both deformed and undeformed experiments (Fig. 3, 4). The general rock framework is constituted in all these samples by interconnected quartz layers rather than interconnected-mica layers. However, second order differences are observed. The fine-grained experiments show idiomorph mica grains and exhibits smaller individual mica grains in average than other samples (Fig. 3c, f).

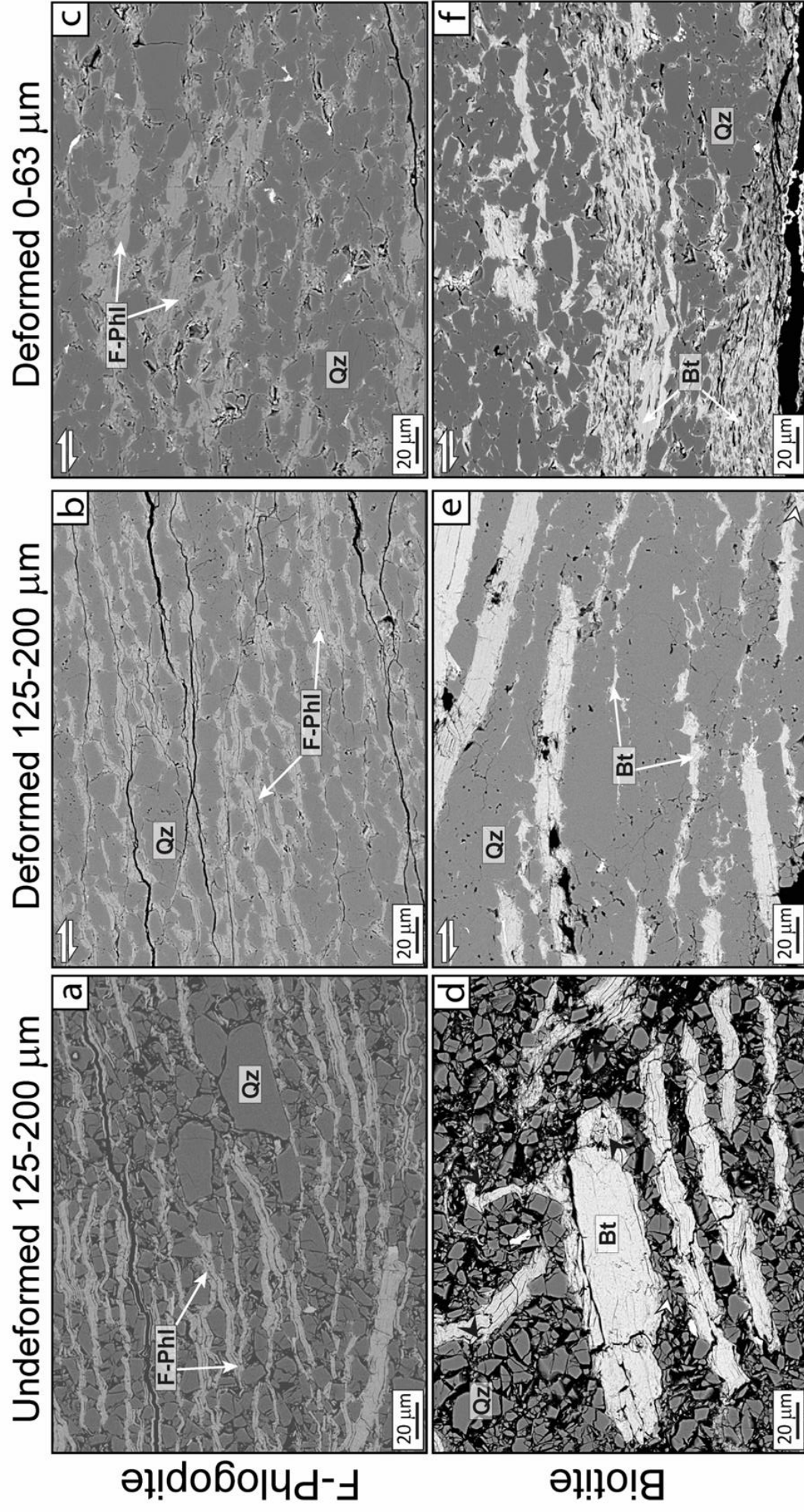


Figure 3 : Microstructural observations from mica-quartz assemblages under a scanning electron microscope (SEM). Backscattered electron (BSE) images at low magnification showing undeformed sample of a) F-Phlogopite and d) Biotite. BSE image of coarse grain F-phlogopite in b) and biotite in e) showing interconnected quartz layers. BSE image of fine grain F-phlogopite in c) showing interconnected quartz layer and biotite in f) showing interconnected mixed layer of mica and quartz. Mineral abbreviations are from [Whiney and Evans \(2010\)](#).

In all experiments, large ($> 5 \mu\text{m}$ with average size $30 \mu\text{m}$, Fig. 3) relict phlogopite grains are elongated parallel to the foliation and pinched and kinked among quartz grains resulting in apophyses (black arrows in Fig. 4). Newly formed, absent from undeformed samples (Fig. 3a, d), small mica grains ($< 5 \mu\text{m}$, Fig. 4) are located interstitially among quartz-quartz grain boundaries (Fig. 4 white arrows in all panels). Quartz forms elongated domains made of $\sim 10 \mu\text{m}$ large angular grains surrounded by fine grained quartz, (Fig. 3b, c, e, f and 4). A micro porosity, with pore diameter in average $< 1 \mu\text{m}$, is located mainly along grain boundaries of quartz (Fig. 4a, b, c, e yellow arrows).

In summary, regardless of mica composition microstructures are overall similar in all experiments even if differences are observed in mechanical behavior.

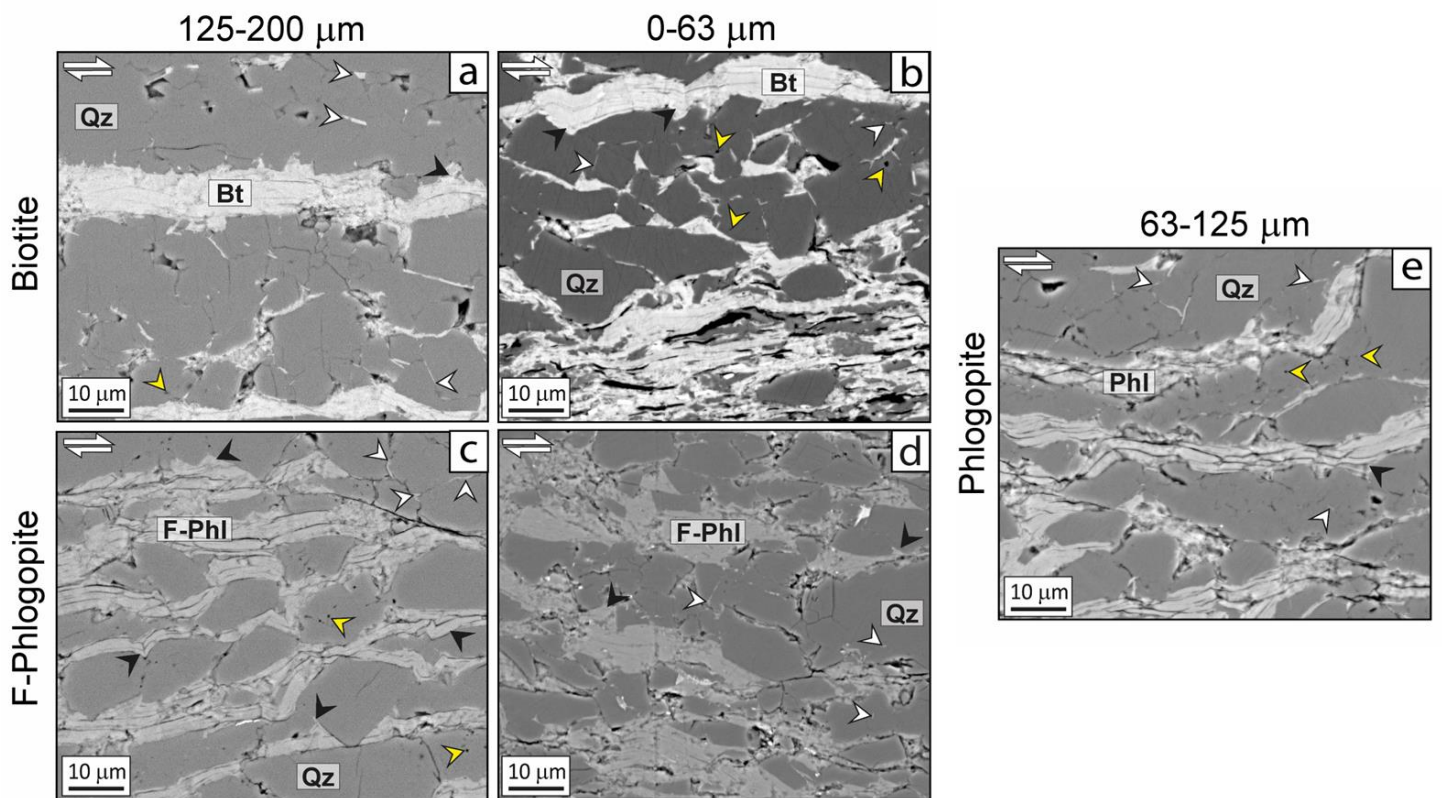


Figure 4 : Microstructural observations from deformed mica-quartz assemblages under a scanning electron microscope (SEM). Backscattered electron (BSE) images at high magnification showing elongated, kinked and pinched relict grains of mica surrounded by angular relict clasts of quartz, with few micropores for a) biotite coarse grain, b) biotite fine grain, c) F-phlogopite coarse grain, d) F-phlogopite fine grain and e) phlogopite intermediate grain. White arrows: tiny mica grains ($< 5 \mu\text{m}$). Yellow arrows: micropores. Black arrows: mica pinched and kinked microstructures. Mineral abbreviations are from Whitney and Evans (2010).

VI.2.3. Cathodoluminescence imaging of quartz

SEM-CL imaging of quartz show non-luminescent grains corresponding to phlogopite, rare Fe-oxides and fractures, and luminescent grains of quartz.

In general, in F-Phl-bearing samples, quartz is defined by a significantly lower luminescence compared to Bt and Phl samples which show a higher amount of high luminescence (light) for similar threshold values (Fig. 5), despite both Bt-bearing and F-Phl-bearing samples are made with the same quartz starting powder (La Gardette quartz). Conversely changing the grain size does not change the luminescence of quartz as seen in F-Phl samples (Fig. 5a, b). While in the undeformed F-Phl-bearing sample parent quartz grains are angular and exhibit variable luminescence but homogeneous within a grain (Fig. 6a), in the deformed coarse-grained sample parent quartz grains are elongated and S-shaped and exhibit variable luminescence within the grain (Fig. 6b).

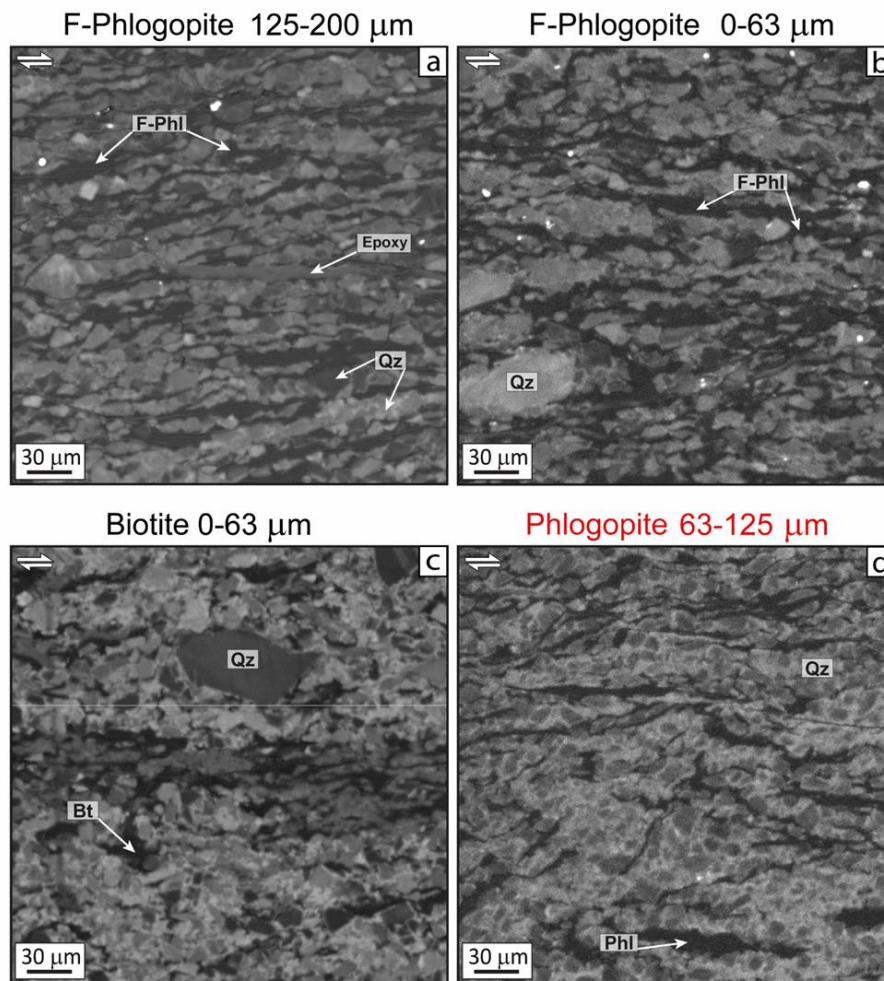


Figure 5 : SEM-CL maps of a) F-phlogopite coarse grain size, b) F-phlogopite fine grain size, c) Biotite fine grain size and d) Phlogopite intermediate grain size. Phlogopite is colored in red to highlight a different quartz material.

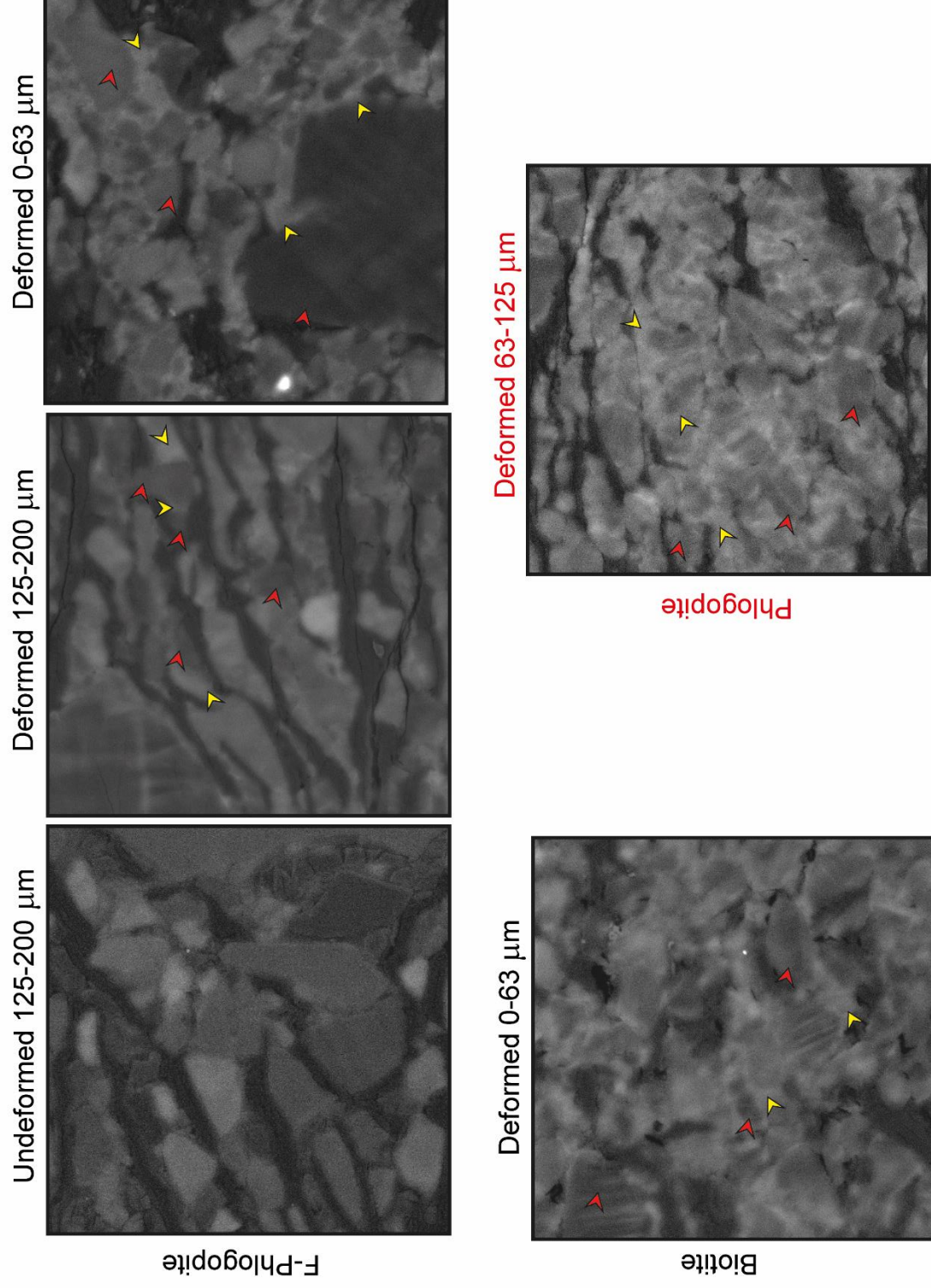


Figure 6 : SEM-CL maps at high magnification of a) undeformed F-phlogopite coarse grain size, b) F-phlogopite coarse grain size, c) F-phlogopite fine grain size and e) Phlogopite intermediate grain size. Phlogopite is colored in red to highlight a different quartz material.

In the finer grained F-Phl sample a quartz cement is observed to surround parent quartz grains. It exhibits lighter luminescence than angularly shaped parent quartz (yellow arrows in Fig. 6c).

Bt and Phl-bearing samples are characterized by large domains of quartz matrix with high luminescence (lighter pointed by yellow arrows on Fig. 6) that surround quartz that are angularly shaped and have lower luminescence (darker pointed by red arrows on Fig. 6). Zooming in high magnification micrographs we see the variation of high luminescence quartz between undeformed and deformed F-Phl samples (Fig. 6a-c):

As in SEM-CL imaging, hyperspectral cathodoluminescence (CL) images exposes non-luminescent grains corresponding to mica, rare Fe-oxides and fracture, and luminescent grains exhibiting quartz (Fig. 7). The spectra are identical among all samples except the intensity of the luminescence at each peak, despite Phl- and Bt/F-Phl-bearing samples are made with different quartz starting materials. The highest peak is observed at 653 nm (1.9 eV) (yellow quartz) while a secondary, less intense peak at 427 nm (2.9 eV, blue quartz). Intensity differences translate in slightly different colors or color intensity in the CL maps between samples (Fig. 7). Quartz showing yellow CL color is named Quartz 1 and recognized from its shaped to be formed by the parent quartz grains. Quartz showing blue CL color is named Quartz 2 are recognized from its smaller grain size and microstructural position (surrounding Quartz 1) to be secondary quartz.

Yellow and blue luminescence grains correlate with the dark and light luminescence respectively in SEM-CL. The intensity of the peak at ~2.9 eV is higher in Bt-bearing samples than in F-Phl- and Phl-bearing ones (Fig. 7e). Mineral phase maps computed from hyperspectral CL maps show that the proportion of Quartz 2 is higher in Bt samples compared to F-Phl and Phl samples (Fig. 7f).

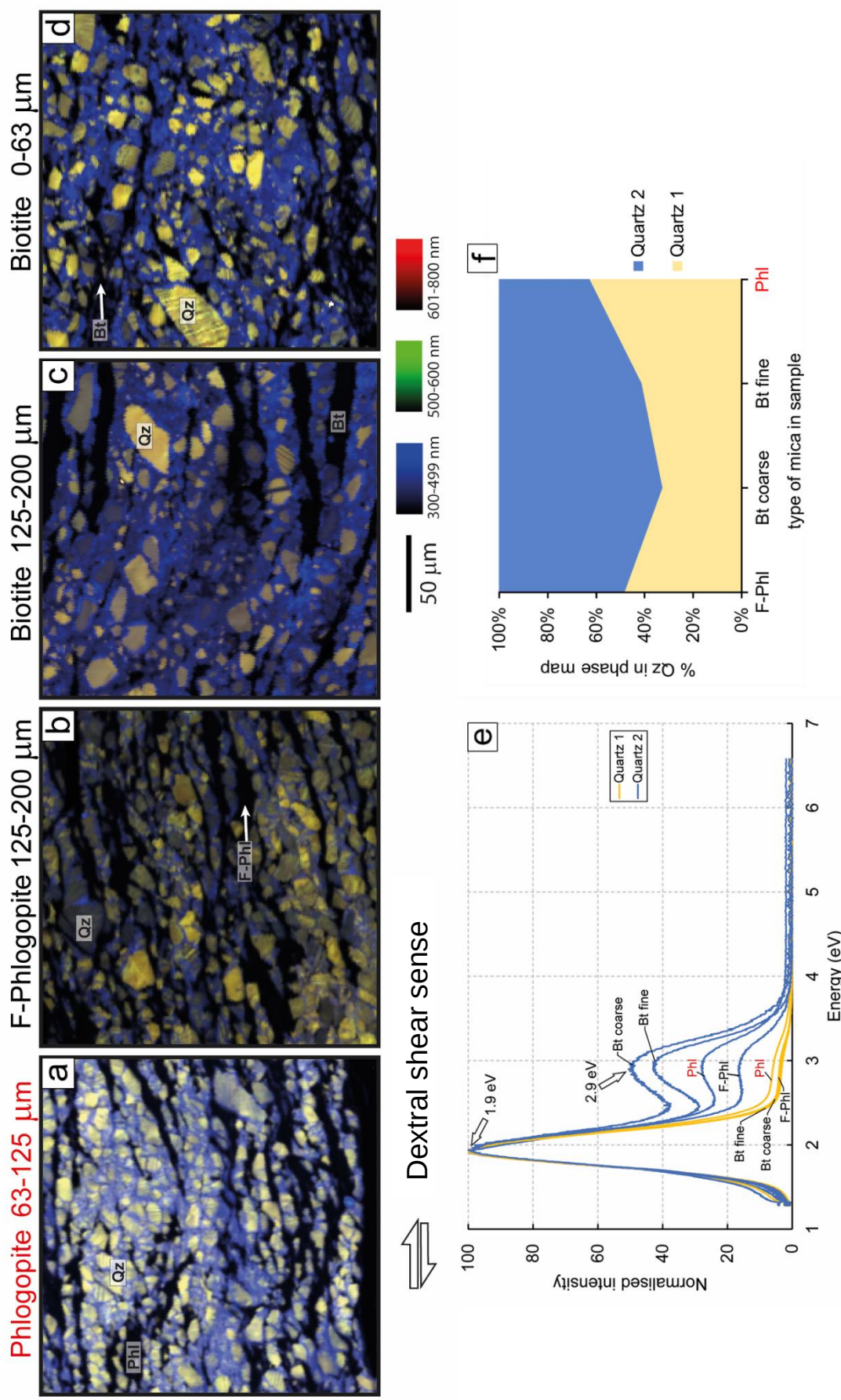


Figure 7 : Hyperspectral cathodoluminescence (CL) imaging from 70% vol. Qz and 30% vol. mica samples. a) Phlogopite sample b) F-Phlogopite coarse grain sample c) Biotite coarse grain sample d) Biotite fine grain sample. e) Spectrum of energy (eV) normalised to the intensity of the highest peak. For Phl-bearing samples for example (red), the peak at ~ 2.9 eV is 30% of the intensity of the one at 1.9 eV. f) Proportion of quartz types, Qz1 and Qz2, in phase map-type of mica in phase map evolution. Phlogopite is colored in red to highlight a different quartz material.

VI.2.4. Mineral composition

Compositional maps for aluminum content measured for similar counts per second scale intensities (1-500 cps) and analytical conditions (15 kV, a beam current of 150 nA, a dwell time of 0.1 seconds and a step size of 0.5 μm), indicates a similar Al pattern in Bt and F-Phl samples (Fig. 8). Quartz 2 exhibits a high Al content than Quartz 1. In fine-grained Bt-bearing samples, secondary quartz shows in average higher number of counts than in other samples, suggesting higher Al content in Quartz 2 (Fig. 8c).

The chemical composition of mica varies within a sample (showing a trend, Fig. 9), but the magnitude of variations is similar between undeformed samples and deformed ones and among deformed samples, both for F-Phl and for Bt-bearing experiments. Such variations mainly concern Al^{VI} and Al^{IV} in Bt (0.05-0.57 a.p.f.u. and 0.8-1.4 a.p.f.u., respectively, Fig. 10c, d). Minor variations are observed for the Si content, mainly comprised in 2.8-3.2 a.p.f.u. for F-Phl and 2.6-3.2 a.p.f.u. for Bt (Fig. 9a). The alkaline content of mica poorly evolves between ~ 0.8 -1 a.p.f.u. (Fig. 9b).

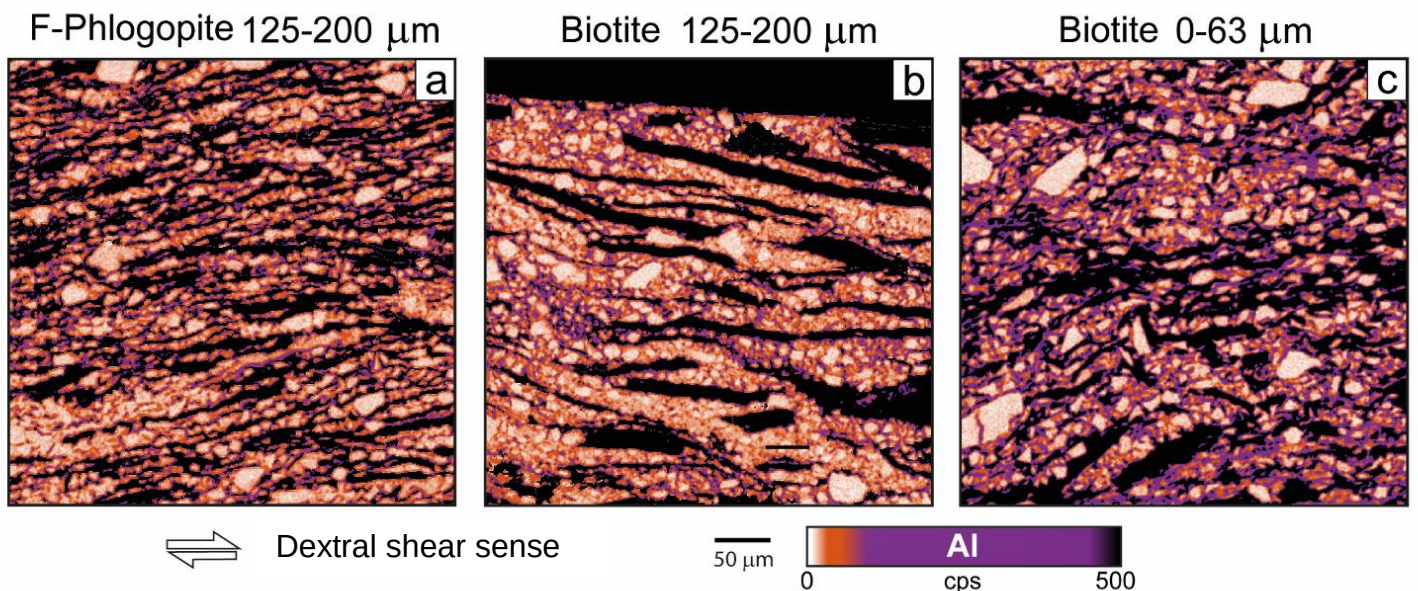


Figure 8 : Elemental maps for Al in quartz in a) coarse grain fluorphlogopite, b) coarse grain biotite and c) fine grain biotite experiments. Analytical conditions are 15 kV, a beam current of 150 nA, a dwell time of 0.1 seconds and a step size of 0.5 μm . Scale is 1 to 500 counts per second (cps).

Since the composition of biotite in the undeformed samples span the entire range of composition, such variability may be related to the initial compositional heterogeneities present in mica grains of the starting material (e.g. zoning). In Bt samples, larger biotite grains show higher Al and Fe contents than smaller ones, as shown by X-ray maps (Fig. 10b, c, h, i). Conversely, in F-Phl-bearing sample mica exhibits homogeneous content in Al, Mg and Fe, but is richer in Mg than mica in Bt-bearing samples (Fig. 10a, d, g). Slight variations of F content are observed between core and mantle of F-Phl grains, especially in the largest ones (between 1 and 100 cps, Fig. 10i).

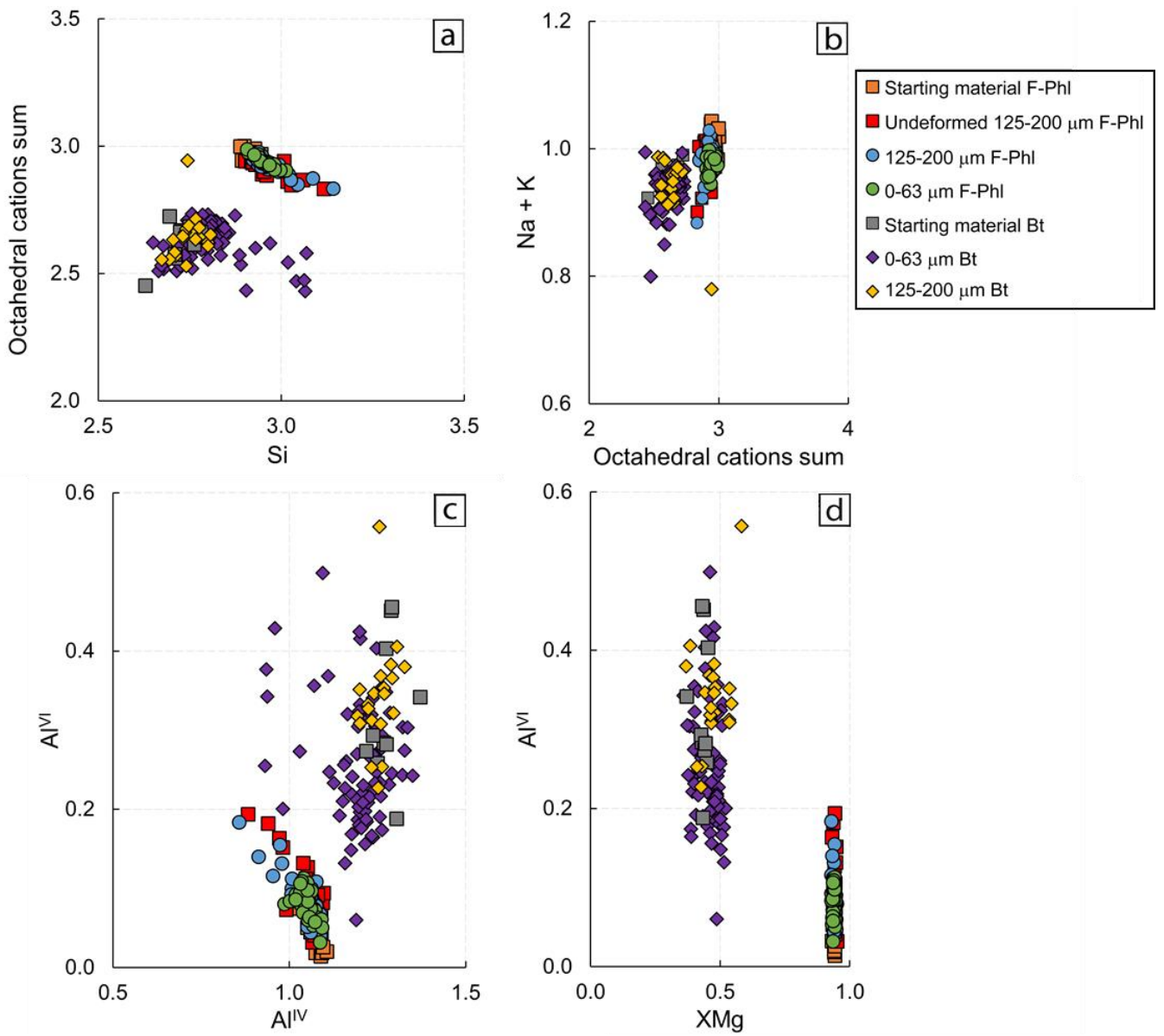


Figure 9 : Plots of EPMA point analyses for phlogopite in a.p.f.u. a) Na+K versus Octahedral cations sum. b) Octahedral cations sum versus Si. c) Al^{VI} versus XMg. d) Al^{VI} versus Al^{IV} .

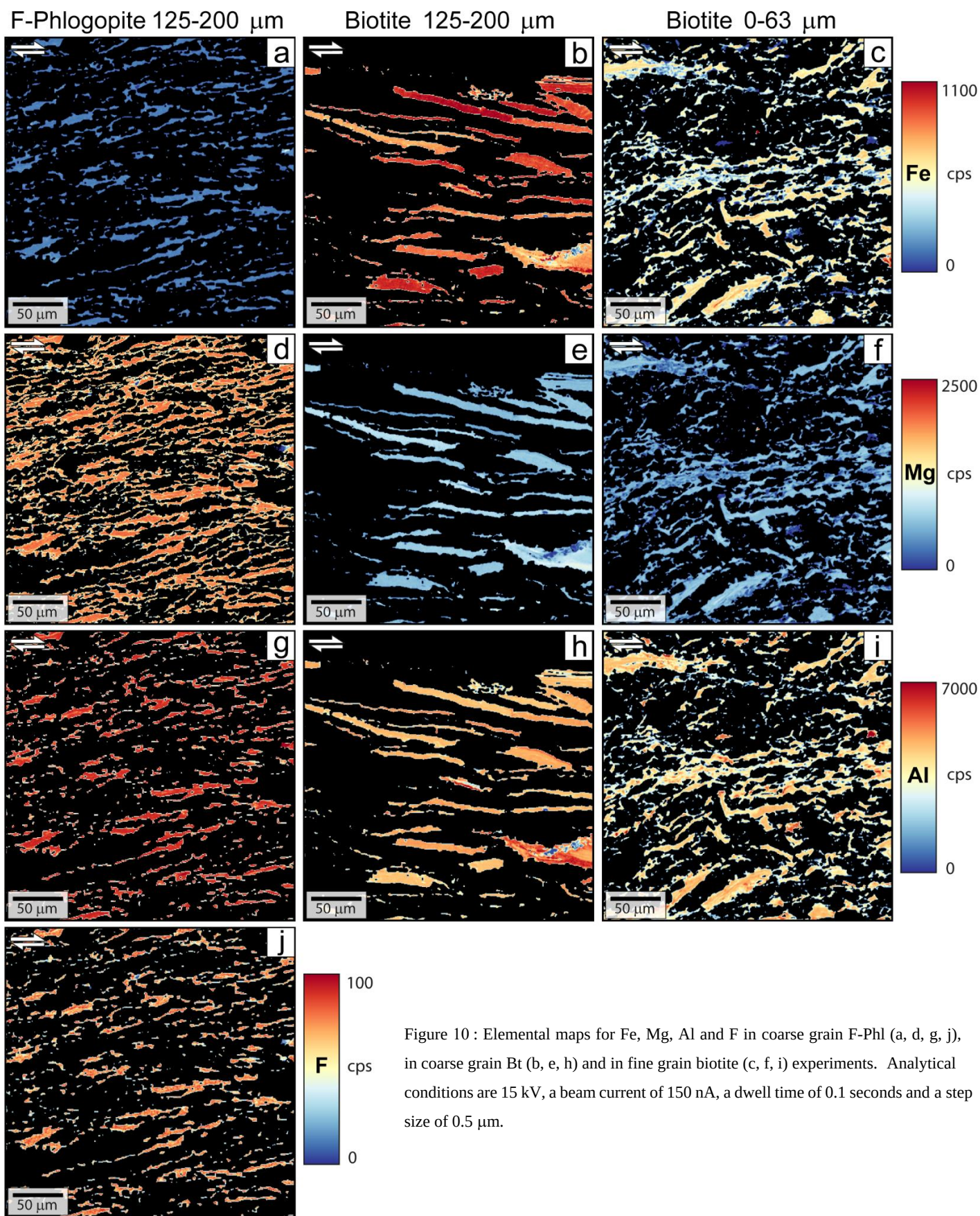


Figure 10 : Elemental maps for Fe, Mg, Al and F in coarse grain F-Phl (a, d, g, j), in coarse grain Bt (b, e, h) and in fine grain biotite (c, f, i) experiments. Analytical conditions are 15 kV, a beam current of 150 nA, a dwell time of 0.1 seconds and a step size of 0.5 μm .

VI.3. Discussion

VI.3.1. Strength of mica-quartz aggregates with different compositions of mica

Mechanical results show that the bulk strength of the quartz-F-Phl samples is higher than the quartz-Bt samples. Independently from the grain size, all Bt-bearing experiments share the same weak rheology, while F-Phl samples exhibit stronger rheology above or close to the Goetze criterion, except the fine grain (0-63 μm) experiment that is weaker, but where a slip at piston interface occurred.

By comparing the strength of all F-Phl assemblages for different experiments, results suggest that for F-Phl samples, only the assemblage with finer grains show a weaker behavior as it has a higher amount of reworked quartz, this possible explanation is more detailed later in the discussion.

Some additional differences between experiments need to be recalled such as the duration of the experiments. F-Phl experiments lasted in average 68 hours whereas Bt experiments lasted 37 hours, which could be either linked to the strength of F-Phl experiments or to the lead independently of the type of mica. As a consequence, as longer experiments will have a higher shear strain (γ) and less overlap, this would affect the sample geometry with higher chances of jacket breaking and making data correction trickier. Therefore, when the shear strain (γ) is higher than 4 the area corrections is overestimated (Heilbronner et al., 2020). Indeed, when the overlap area between the two forcing blocks gets smaller we calculate the area correction assuming that the stress is only acting on the area, the confining medium (salt) also compresses and the force distribution is getting less and less clear (Fig. 11). Shear strain are higher for F-Phl mixtures with an average of 7 compared to 5 for Bt experiments, which leads to an exponential hardening behavior and could explain the fact that platinum jackets ruptured for F-Phl samples

Furthermore, different duration of experiments may favor the completion of reaction to achieve equilibrium allowing more grain growth in longer experiments.

Phl experiments with 30% vol. Phl show a stronger behavior relative to the first experiment (Fig. 1a) in agreement with more brittle deformation of quartz as attested by the net of intragrain fractures (Fig. 3-4). A possible loss of water during the experiment might be a reason for this strength difference, although we have no data yet available in this sense.

Note that the similar strength and effective viscosity between Bt and Phl experiments indicate that the type of quartz (milkyquartz for Phl, La Gardette quartz for Bt mixtures respectively) do not play a major role in the strength of the assemblage as soon as they are both hydrated and sieved at the same grain size (10 μm).

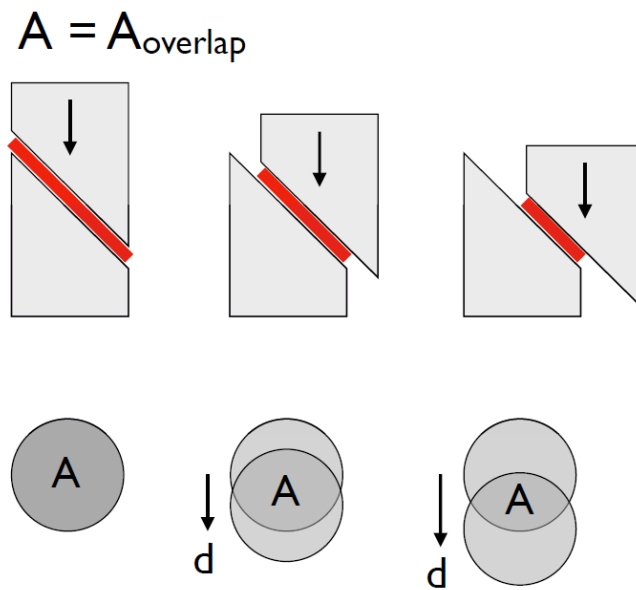


Figure 11 : Sketch describing the area of overlap of the forcing blocks (A) that decreases as the forcing blocks are offset with increasing shear. This area is corrected using the area correction in shear experiments (after [Heilbronner et al., 2020](#)).

VI.3.2. Mica deformation mechanisms

Previously (chapters IV and V), we concluded that in Phl samples we found no evidence of basal slip as there are no basal dislocations observed, and suggesting that ripplifications lead to kink bands and conjugate expansion arrays. Instead, the microstructural position of small phlogopite grains and composition changes suggest that phlogopite undergoes dissolution and precipitation with varying transport distances from nanometric to micrometric scales. This process, rather than dynamic recrystallization or mechanical reduction from kinking, appears to be the primary mechanism for phlogopite grain size reduction. Usually, dissolution-precipitations involves a compositional change between the parent and product grains, showing two populations of micas. While this was observed in Phl samples, data for F-Phl and Bt assemblage are not yet available. However, given the similarity of microstructural positions of small mica grains in all these experiments, we assume that the small mica grains in F-Phl and Bt samples are also newly-grown micas by dissolution-precipitation processes.

It is documented that different types of mica may have different mechanical and elastic properties. This is the case of white mica relative to biotite, or of F-phlogopite relative to phlogopite (Wilson and Bell, 1979; Kronenberg et al., 1990; Mares and Kronenberg, 1993; Dahl and Dorais, 1996). This is partly due to the different crystal structure and cations fill. For example, margarite that include a divalent cation (Ca^{2+}) in the interlayer is more resistant than white mica where the interlayer is filled with univalent cations (as K^+ and Na^+) so that it is often named “brittle mica” (Takeuchi, 1964). The enhanced chemical bonding of these divalent cations, coupled with the simultaneous substitution of Al for Si, alters the mechanical characteristics of the mica, shifting it from viscous to brittle (Guggenheim and Bailey, 1978). In the case of F-Phl, $\text{F}(\text{OH})_{-1}$ substitution coupled with the concomitant removal of hydroxyl H^+ towards the interlayer space, can increase the mechanical strength of biotite. Indeed, elimination of K^+ - H^+ repulsion, reinforces the interlayer bonds, and allows K^+ to penetrate further into the interlayer space, resulting in a geometry less favorable to basal slip (Dahl and Dorais, 1996).

In our experiments, the higher mechanical strength of F-phlogopite relative to phlogopite may therefore partly explain the increased resistance of the bulk assemblage. However, in experiments made of 30% vol. of mica, the general sample fabric is made of interconnected layers of quartz that therefore

accommodate the most of the deformation, and where mica is the weak phase. Thus, it is difficult to conclude that the intrinsic strength of different-phlogopite may can control the bulk strength of the assemblage. What is different between experiments with mica with different compositions is instead the amount and spatial distribution of secondary quartz. Hence, the behavior of quartz or the effect of mica on quartz reworking may constitute an alternative explanation to the variations of the bulk strength.

VI.3.3. Quartz reworking and implication on rock strength

Quartz exhibits significant grain size reduction in all samples independently from mica composition and from the type of quartz. Angularly shaped parent quartz grains suggest poor plastic deformation. Nevertheless, quartz plastic deformation cannot be excluded as further crystallographic orientation and microstructural analysis. As seen previously (chapters IV and V) dynamic recrystallization by SGR and solution-transfer diffusion creep are the primary quartz reworking mechanisms, influenced by mica content and resulting grain boundary interactions.

Hence, similarly to Phl-bearing samples, CL peak spectrum for F-Phl and Bt bearing samples correlate with two main peaks at 1.9 eV and 2.9 eV, as well as similar luminescence. However, CL energy peaks show different intensities, with normalized intensity at 100, between Phl-bearing samples and F-Phl and Bt bearing samples. CL emission band at 1.9 eV are linked to structural defects in oxygen in deficient quartz, such as the presence of fewer non-bridging oxygen hole center defects or less OH-groups (Stevens-Kalceff, and Phillips, 1995; Hamers et al., 2023; Götze et al., 2024). Conversely, the emission band at 2.9 eV could be related with Ti (up to 130 ppm) and is responsible for contrasting of growth zoning (Müller, 2000). Gorton et al, 1997 distinguishes the emission band around 2.9 eV as intrinsic emission at 2.96 eV and intrinsic defect at 2.8 eV. The most significant impurity in the quartz lattice, besides Al, is bonded water in the forms of H^+ , OH^- , and H_2O , yet the solubility of water in the quartz lattice is dependent on the Al content (Kronenberg et al., 1986).

As in chapter IV, Quartz 2 seen as blue in CL images is interpreted as the result of the dissolution-precipitation of quartz (quartz 2) forming around relicts of parent Quartz 1 grains, in cracks. CL variations between F-Phl, Bt and Phl experiments could be linked to the quartz composition as the milky quartz in Phl samples is poor in Li compared to the quartz from La Gardette rich in Li. However, the

amount of Quartz 2 is overall higher in Bt-bearing samples than F-Phl and Phl-bearing samples, suggesting increased reactivity of quartz.

Quartz reworking processes are mainly produced at grain boundaries as suggested by CL, chemical composition and microstructures. Quartz reworking seems to vary with the type of mica, being more abundant in Phl and Bt samples and less in F-Phl samples. The amount of quartz reworked can therefore be recognized as one of controlling feature of the sample weakness. One possible explication for weakening is that quartz reworking induces grain size reduction and phase mixing (as witnessed by interstitial mica).

VI.3.4. Effect of mica composition on deformation processes initiated at grain boundaries

As seen in previous chapters, several studies suggest that the occurrence of phyllosilicates enhances the dissolution of quartz because of the formation of a fluid film at the interface (Becker, 1995; Schwarz and Stockhert, 1996; Farver and Yund, 2000; Rutter and Wanten, 2000; Renard et al., 2001, Meyer et al., 2006, Greene et al., 2009). Dissolution at the mica-quartz interface is facilitated in comparison to other interfaces, such as quartz-quartz, due to the increased thickness of the fluid film at grain boundaries caused by water (Farver and Yund, 2000, Anzalone et al., 2006; Meyer et al., 2006; Greene et al., 2009). These interactions predominantly occur at grain boundaries where solution-transfer or effective diffusion (dissolution-precipitation) take place. Interface-coupled dissolution-precipitation facilitates chemical exchange between the reaction front and bulk solution through transient fluid pathways (Aldree-Williams et al., 2015; Duan et al 2023). The suspected catalyst for solution-transfer diffusion creep at grain boundaries may lie in the chemical potential difference within the two-phase aggregate. Dissolution is indeed activated by a stress-induced difference in chemical potential of the solid between the dissolution and precipitation zones, leading to mass transfer through a fluid (Paterson, 1973; Weyl, 1959). The chemical potential gradient between mica-quartz could contribute to accelerated dissolution. It is assumed that quartz dissolution depends first on temperature and local stress, in our experiments the temperature is the same but the local stress might evolve depending on the amount and the arrangement of mica. Therefore, we can suppose that mica has a mechanical effect on the sample such

as samples with poor amount of mica or with strong mica (F-Phl), the local stress is high and would increase quartz dissolution.

However, the microstructures show that F-Phl sample grains deform in a similar way than mica grains in Phl-bearing sample. In fact, in the 30 vol. % phlogopite sample quartz is essential to the fabric, accommodating strain as interconnected layers. This suggest that instead of being mainly driven by strain partitioning between quartz and mica, the dissolution potential of quartz may be primarily controlled by the composition of the fluid that changes with the changing chemical composition of recrystallizing micas.

As micas have a different composition (F-Phl, Phl and Bt), they have a different solubility as well, which would also influence the dissolution potential of each and influence the thickness of the film fluid.

Additionally, liberating OH in the system during Bt dissolution could increase H₂O amount keeping the pH close to 6-7 and therefore increasing thickness of the fluid film. Conversely, for F-Phl dissolution liberating F, a hard ligand (Pearson, 1963), often result into HF complexes making the solution more acid and dissolving more quartz (Keppler, 1993; McPhie et al., 2011; Xing et al., 2019) while preventing quartz from precipitating. F can be a complexing ligand for Li (Sallet et al., 2023) and influence the quartz recrystallization. All this could be an explanation for the lower amount of reworked quartz in F-Phl samples.

As a conclusion F-Phl samples are stronger than Bt because of (1) lower amount of quartz precipitated probably linked to the F content in the solution making less weak phase in the fabric, (2) the crystal structure of F-Phl is stronger than Bt. A competition between dissolution-precipitation process and underlying chemical interactions at grain boundaries, as well as the intrinsic strength of the mica, leads to stronger samples with F-Phl than Bt.

VI.4. Conclusion

The effect of mica composition on rock strengths was investigated in two-phase (quartz and mica) mixtures by the means of shear experiments in the Griggs apparatus, at similar conditions (750°C and 800°C, 1.0 GPa and bulk strain rate $\sim 10^{-5} \text{ s}^{-1}$) but with different types of mica.

The main result from these experiments is that fluorophlogopite (F-Phl)-bearing assemblages are significantly ($\Delta\sigma \sim 1000 \text{ MPa}$) mechanically stronger than phlogopite (Phl)-bearing and Mg-Fe biotite-bearing (Bt) assemblages. Similar microstructures in all samples show that the intrinsic strength of mica is likely not the controlling factor of the resistance of the assemblage since quartz is the phase forming the framework and interconnecting layers. The type (chemistry) of quartz also does not explain the strength contrast between F-Phl and Bt experiments.

The contrasted mechanical behavior may result instead from quartz grain size reduction achieved by dissolution-precipitation of quartz. This process is promoted by the interactions at grain boundaries between mica and quartz and is influenced by the mica composition. We propose that, depending on the composition of dissolving mica under stress, the thickness and composition of the fluid-film at mica-quartz interface will be different influencing solution pH, phases solubility and therefore dissolution-precipitation.

Chapter VII – General discussion, conclusion and perspective

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VII.1. Discussion

In this discussion the characteristics of worldwide upper to mid crustal natural shear zone rocks were reported in Table 1 for a selection of 36 papers, in order to highlight the differences and contrasts in natural samples and to be compared with our experimental samples. Phase distribution was constrained following the classification of [Heilbronner and Barrett, 2014](#) (Fig. 1).

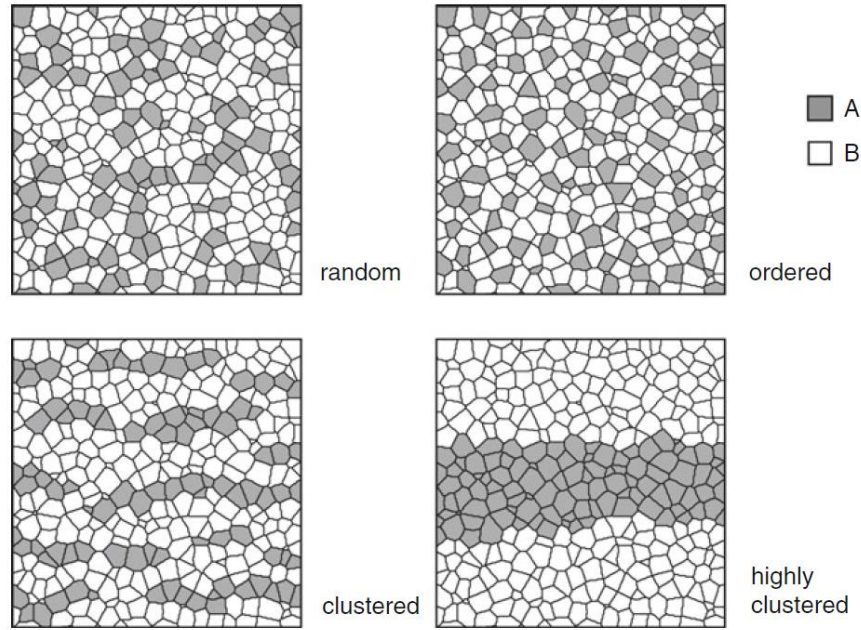


Figure 1 : Four examples of spatial distributions. In each case, the ratio of phase A: phase B $\sim 0.3:0.7$ ([Heilbronner and Barrett, 2014](#)).

V.3.1. Mica deformation

In natural shear zones, the main deformation microstructures observed in phyllosilicates (biotite, muscovite, chlorite) are: kinking, bending, grains (re)crystallization and mica fish (Table 1 and references therein).

A granitic mylonite from the Pyrenees ([Airaghi et al., 2020b](#)) exhibits kinks of chlorite and white mica (red arrows in Fig. 2a), similarly to phlogopite kinks in our experimental samples (red arrows in Fig. 2c) suggesting intracrystalline deformation processes. Along with kinking, mica apophyses in quartz can also be observed in both natural sample and this study (black arrows in Fig. 2b, c), attesting to the strength contrast between stronger quartz and weaker mica.

In addition, in Fig. 2b, large porphyroclasts are cut by fracture networks, evolving into microshear zones with the increase in strain (Goncalves et al. 2016). Along these fracture networks, mass transfer led to the precipitation of secondary phases, including a large proportion of mica. A similar process was observed in our experiments, as small grains of mica, with a composition different from the original mica porphyroclasts, were precipitated in the core of quartz aggregates (Fig. 2). Dissolution-precipitation was identified in our experiments as the main mechanism of mica grain size reduction (see chapter V). Indeed, we do not observe any evidence of dynamic recrystallization of mica in our experiments, as well as in natural samples, as a result of the single slip-plane of mica hindering this mechanism (Shea and Kronenberg, 1992; Berger et al., 2017).

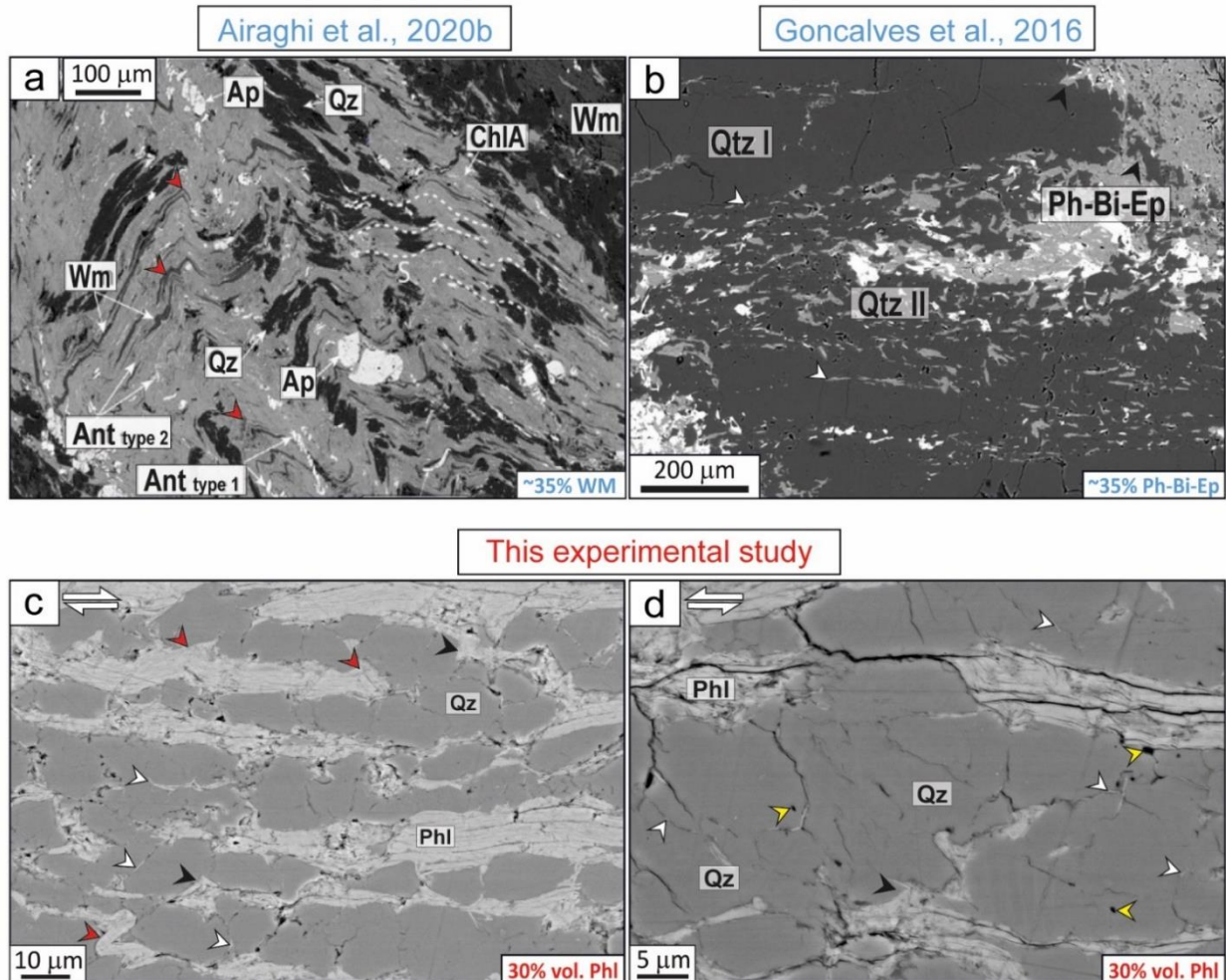


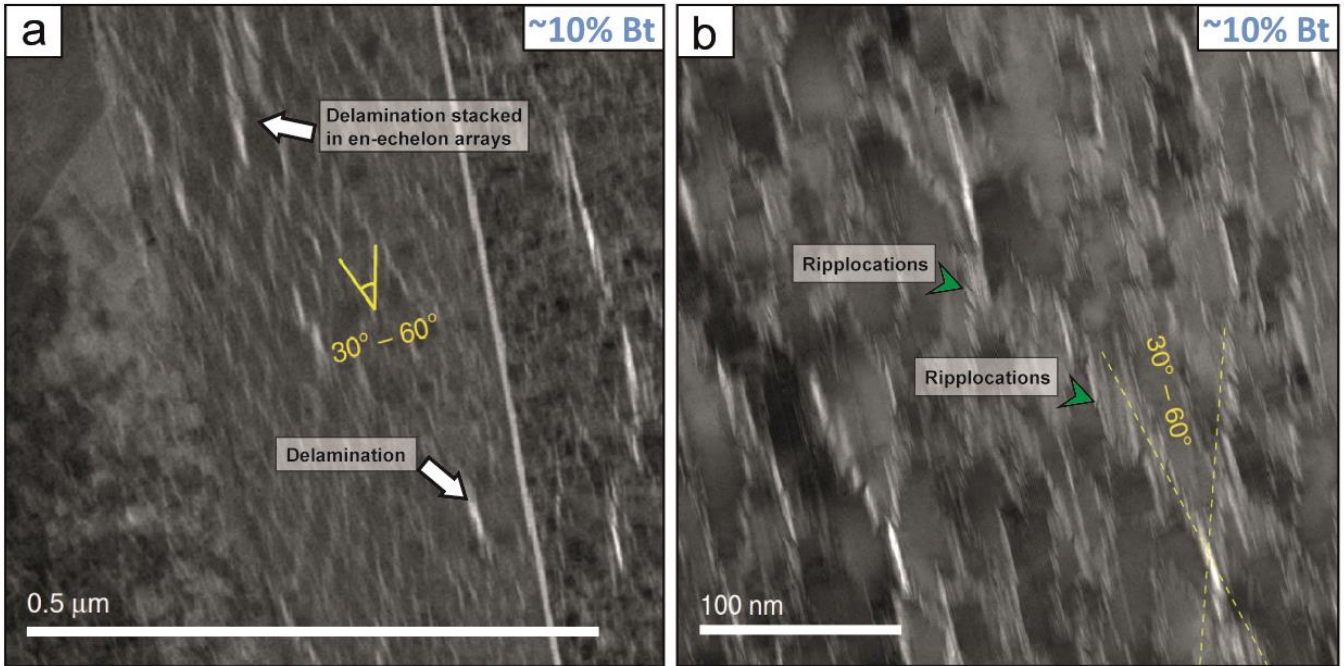
Figure 2 : Backscattered electron micrographs of natural shear zone mylonites a) from the Pyrenees (Airaghi et al., 2020b) for 35% of white mica content, b) from the Alps (Goncalves et al., 2016) for 35% of hydrated minerals (phengite-biotite-epidote) and of our experimental samples of phlogopite-quartz mixture for 30% vol. phlogopite c) in low magnification and d) high magnification. Red arrows show kinking, white arrows show small mica, yellow arrows show micropore and black arrows show mica apophyses. Abbreviations: Qz or Qtz: quartz, Wm: white mica, Ap: apatite, Chl: chlorite, Ant: anatase, Phl: phlogopite, Ph-Bi-Ep: phengite-biotite-epidote.

Hence, in mica intracrystalline deformation, kinking and folding typically accommodate the deformation, and it is often linked as a result of basal slip ([Etheridge et al 1973](#); [Bell et al., 1986](#); [Meike 1989](#); [Kronenberg et al., 1990](#); [Mares and Kronenberg, 1993](#)). In our case, we observe kinks, but no basal dislocation consistent with basal slip.

On the other hand, we observe other nanostructures such as amorphous films at low angles to the basal plane and ripplocations, which may also allow kinks to be formed macroscopically. At the nanoscale, the only similarity with natural sample are lattice expansion structures or delamination structures, which trigger some layers to form ripples (ripplocation structures, [Aslin et al., 2019](#)) deflecting around regions of lattice expansion (green arrows in Fig. 3b, d). Delamination structures often occur in en-echelon stacks, and are aligned in two principal directions, 30°–60° apart and bisected by the basal plane (Fig. 3a-c). This produces a diamond-shaped expansion structure array throughout biotite grains. Conjugate delamination arrays are the result of elastic strain energy release due to the accumulation of layer-normal strain in ripplocations ([Aslin et al., 2019](#)). Indeed, it has been proposed that ripplocations lead to (1) kink bands or (2) conjugate expansion structure arrays (see chapter V). This suggest that kinking can be either a result of basal glide or ripplocations, in our case, as illustrated in chapter V, we see microstructures following more the model proposed by [Aslin et al. \(2019\)](#).

The major defects observed in mica in our study—dislocations, triangular defects, amorphous layers, lattice expansion, and ripplocations—indicate no significant intracrystalline plastic deformation except local kinking, and instead, grain size reduction primarily occurs through dissolution and precipitation processes rather than basal slip or dynamic recrystallization.

Aslin et al., 2019



This experimental study

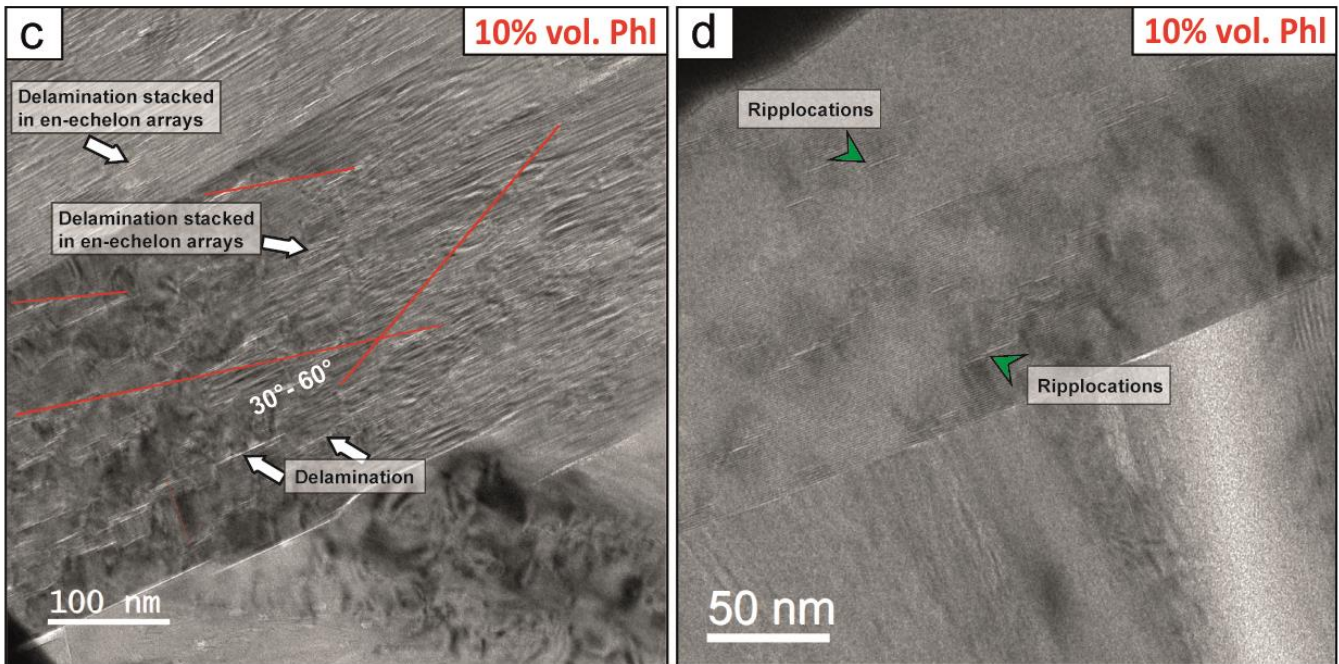


Figure 3 : Brightfield transmission electron microscope micrographs in naturally deformed biotite for 10% biotite content with a) showing delamination structures and b) ripplocations, similarly to experimentally deformed samples of 10% vol. phlogopite sample with c) delamination and d) ripplocations. White arrows point to delamination, green arrows show ripplocations.

V.3.2. Quartz plastic deformation

In natural shear zones studied here (Table 1 and references therein), the main deformation process for quartz is plasticity (accommodated by dynamic recrystallization), while in less frequent cases the operation of dissolution-precipitation is observed. Shear zones in the Pyrenees for example ([Alaoui et al., 2022](#)) exhibit dynamic recrystallization of quartz by subgrain rotation (SGR) in polymineralic mylonites with 28% vol. of white mica (Fig. 4a), while polymineralic mylonites with 42% vol. of white mica exhibit no evidence of dynamic recrystallization, the only microstructures observed are deformation bands (narrow and wide, in white and yellow arrows, respectively in Fig. 4b).

In our biminerale experiments, samples exhibiting quartz dynamic recrystallization by subgrain rotation are characterized by 10% vol. Phl (Fig. 4c), whereas for a higher mica content (50% vol. Phl) no evidence of dynamic recrystallization is observed (Fig. 4d). As a result, the presence and the absence of quartz recrystallization microstructures is similar between natural polymineralic samples and biminerale experiments for similar proportions, despite different temperatures (Fig. 4, Table 1). Indeed, samples from [Alaoui et al., 2023](#) exhibit SGR for $T=350-380^{\circ}\text{C}$, while our experimental study indicates SGR for $T=800^{\circ}\text{C}$ which is highly above conditions corresponding to SGR in nature ([Stipp et al., 2002](#)) but correlates with other experimental conditions suggesting SGR ([Hirth and Tullis, 1992](#); [Stipp et al., 2003](#)). This means that a correlation between natural and experimental sample is legitimate, independent of differences in temperatures and strain rates, as microstructures are similar.

When looking into quartz slip-systems, in natural samples the dominant slip-systems are basal $\langle a \rangle$ and rhomb $\langle a \rangle$ for greenschist- to lower amphibolite-facies conditions of metamorphism (Table 1, Fig. 5b, [Stipp et al., 2002](#)). Conversely, in our experimental samples it is often prism $\langle a \rangle$, as attested by the distribution of misorientation axes of subgrain boundaries (Fig. 5d), with very weak CPO (Fig. 5c) which is also the case in rare natural samples (Fig. 5a, [Mansard et al., 2018](#), Fig. 4 [Alaoui et al., 2023](#)), but a weak fabric does not prevent quartz grains to be deformed (Fig. 4).

Therefore, we suggest that our experimental samples are mostly compatible with natural samples that underwent amphibolite facies conditions (Hippertt, 1994; Mansard et al., 2018; Alaoui et al., 2023). Especially that, according to strain geometry (Schmid and Casey, 1986), temperature (Kruhl, 1996), strain intensity (Lister and Hobbs, 1980), and the presence of fluids (Blacic, 1975), certain slip slip-systems are activated (Hunter et al., 2016).

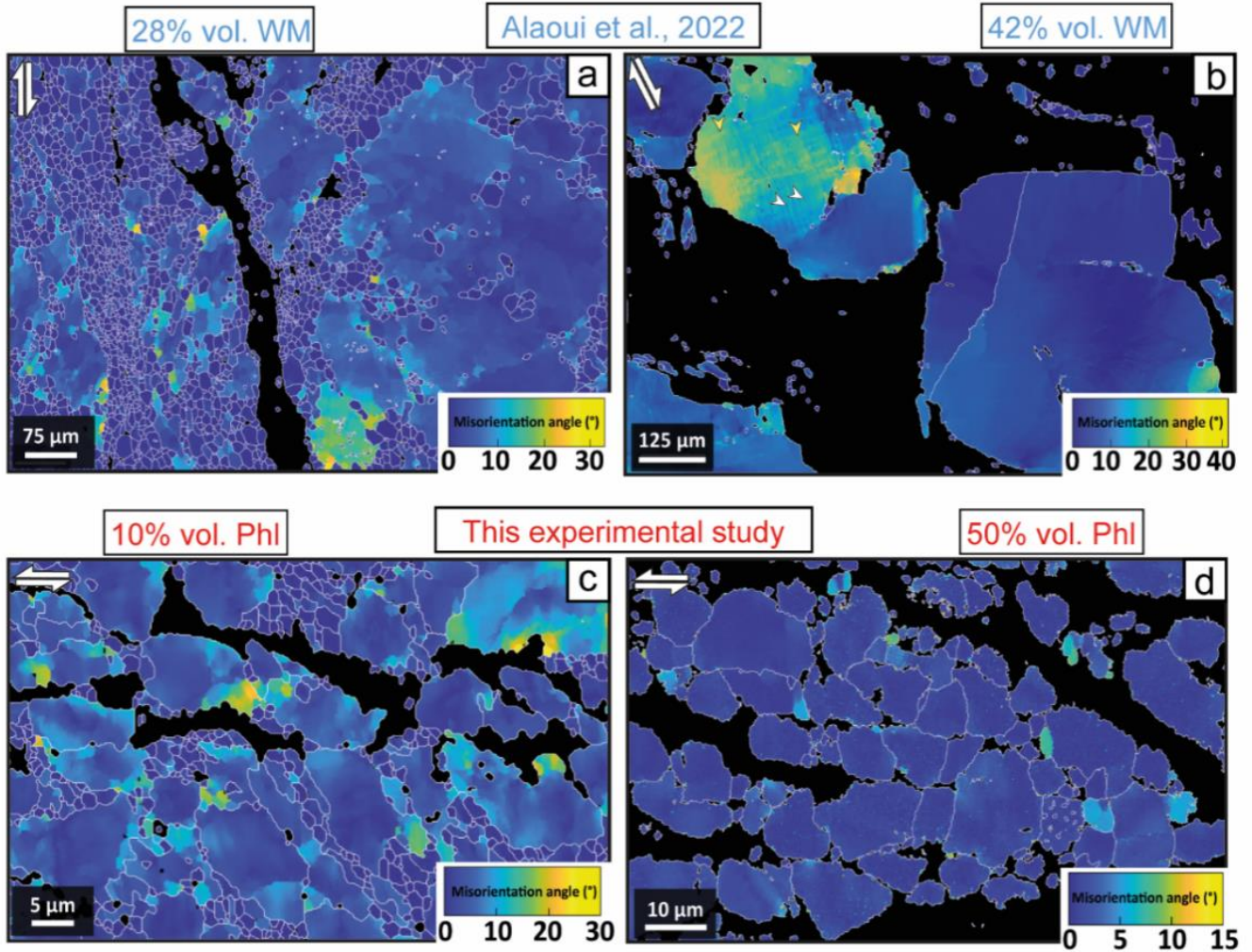


Figure 4 : Intragranular misorientation maps of quartz acquired with EBSD data for natural samples of Alaoui et al., 2023 for sample with a) 28% vol. white mica and b) 42% vol. white mica and from this experimental study with c) 10% vol. phlogopite sample and d) 50% vol. phlogopite sample.

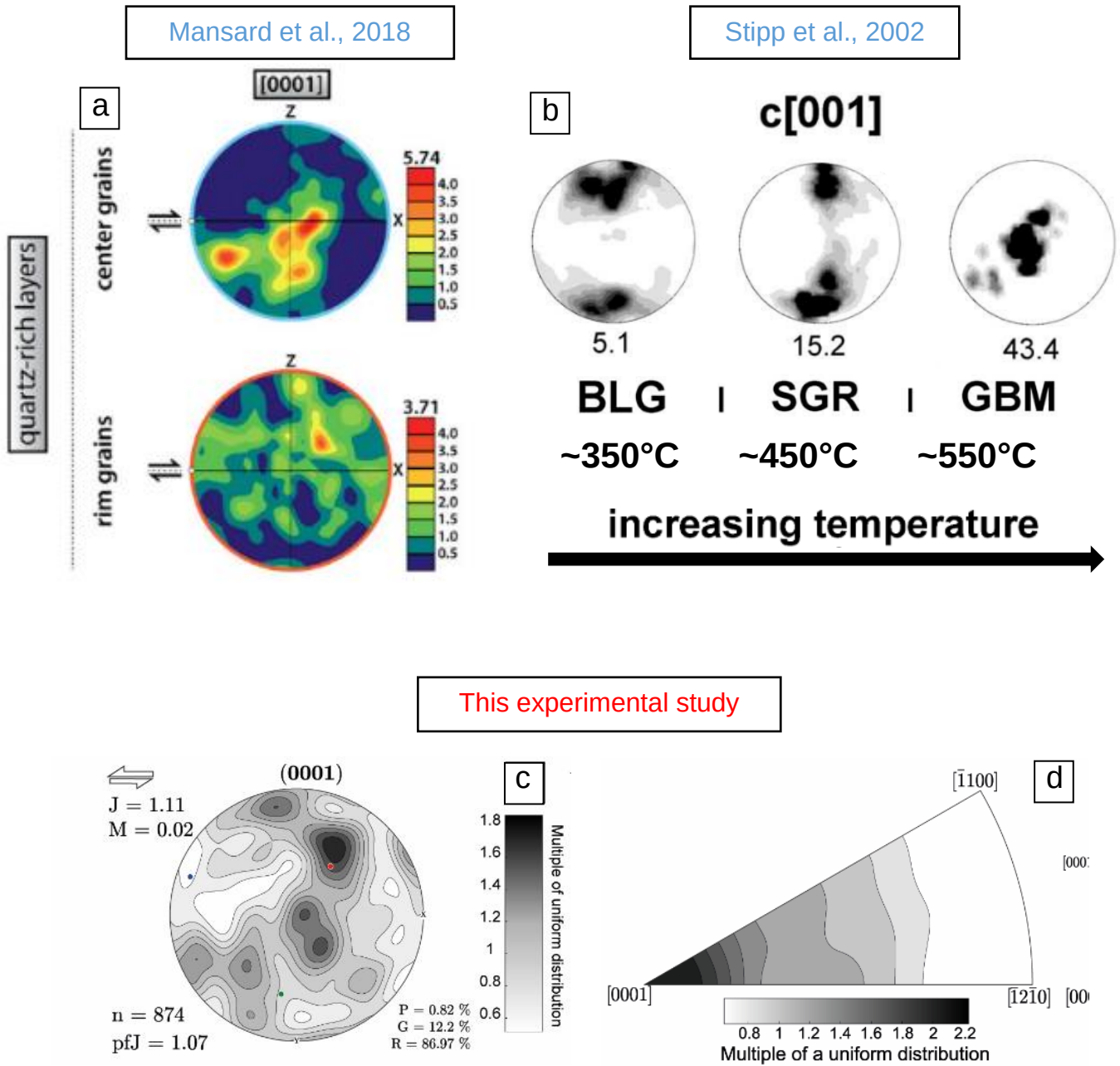


Figure 5 : Pole figures of quartz from natural samples of a) [Mansard et al., 2018](#) for mylonites with quartz rich layers, b) [Stipp et al., 2002](#) for quartzite samples with corresponding dynamic recrystallization microstructure and temperature (BLG: bulging, SGR: subgrain rotation, GBM: grain boundary migration), and from our experimental samples for 10% vol. phlogopite with c) pole figure and d) inverse pole figure misorientation axis. All pole figures in the upper hemisphere.

V.3.3. Polymineralic assemblage deformation

Phase distribution

Phase distribution estimated qualitatively in natural samples does not depend on phase proportion neither on quartz CPO (Table 1 and references therein).

Connectivity of mica

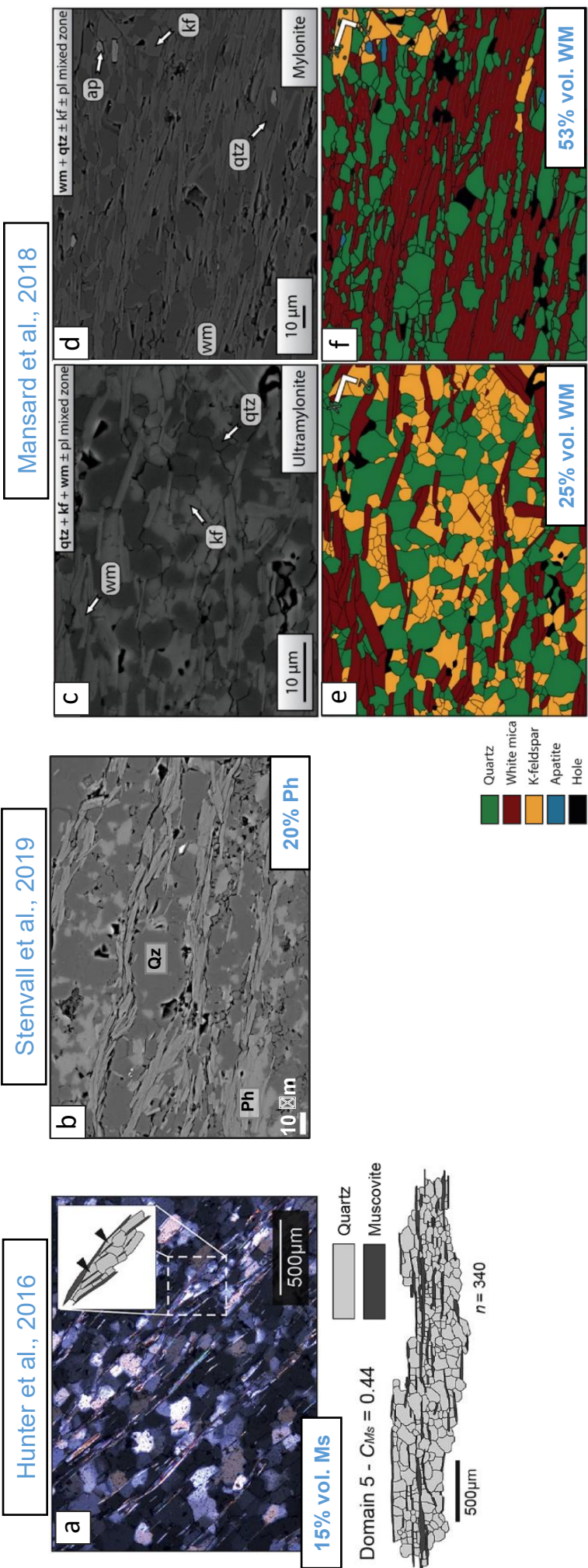
In natural samples, connectivity of mica starts at least for a 10% mica proportion and is common around 25% mica in the bulk rock (Table 1, [Song and Ree, 2007](#), [Hunter et al., 2016](#), [Wehrens et al., 2017](#), [Mansard et al., 2018](#), [Stenvall et al., 2019](#)). Indeed, for bimineralic mylonites ([Hunter et al., 2016](#)) connectivity of muscovite increases progressively with mica content and interconnected layers start to appear at 15% vol. muscovite (Fig. 6a). Conversely, for polymineralic mylonites, interconnectivity of mica starts either around 20% mica (Fig. 6b, [Stenvall et al., 2019](#)) either between 25% and 53% mica (Fig. 6c-f, [Mansard et al., 2018](#)).

For our bimineralic experiments, phlogopite interconnectivity starts between a content of 30% and 50% vol. Phl. (Fig. 6g, h), which is similar to polymineralic samples of [Mansard et al., 2018](#).

Quartz CPO strength as a function of mica proportion and connectivity

Quartz CPO decreases progressively with the increase in mica proportion, till random or weak CPO with large (~18%) mica content, in natural samples (Fig. 7a, b, [Graziani et al., 2020](#)), as well as in our experimental samples even if, the latter exhibit very weak fabric (J-index ~1 and maximum m.u.d < 3) compared to natural samples (Fig. 7c-e). One difference is that the proportion of mica defining the progressive loss of quartz CPO is around ~18% of hydrated minerals in natural samples (Fig. 7b, [Graziani et al., 2020](#)), conversely to our experimental samples where there is no fabric loss as there is hardly any fabric all, whatever the proportions. (Fig. 7d, e).

The influence of mica proportion on quartz CPO correlates with the proportions where mica interconnectivity appears both in natural and experimental samples (see section above and Fig. 6).



Mica interconnectivity
Mica proportion

This experimental study

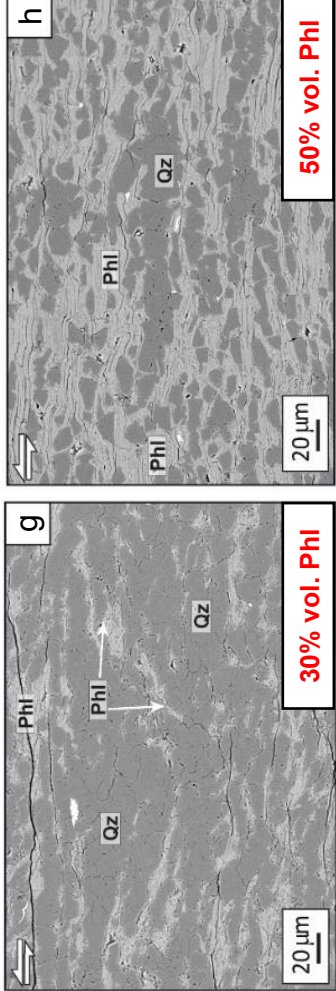


Figure 6: a) Close-up of a fabric domain with higher muscovite connectivity. Here, quartz is variably pinned by muscovite (inset, black arrows) from Hunter et al., 2016. SEM-BSE images representative of b) 20% phengite sample from Stenvall et al., 2019, and from Mansard et al., 2018 in c) of an ultramylonite with 25% vol. white mica, d) mylonite with 53% vol. white mica and their respective manually digitized maps in e) and f). SEM-BSE of our experimental study of g) 30% and h) 50% vol. phlogopite samples. Qz or qtz: quartz, kf: K-feldspar, wm: white mica, ap: apatite, ph: phengite, phl: phlogopite.

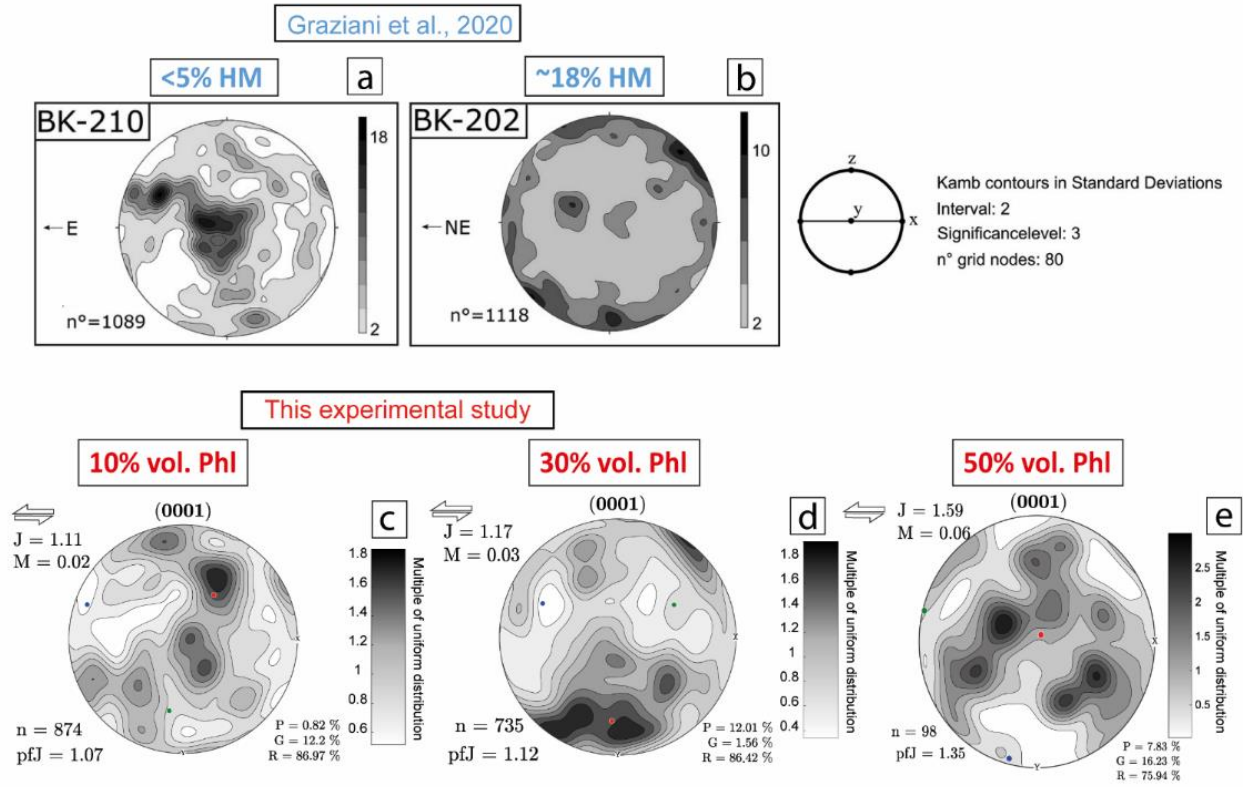


Figure 7 : Pole figures of quartz from natural samples of [Graziani et al., 2020](#) for mylonites with a) less than 5% of hydrated minerals (HM) and b) ~18% of hydrated minerals (HM), and from our experimental samples for c) 10% vol. phlogopite, d) 30% vol. phlogopite and e) 50% vol. phlogopite. All pole figures in the upper hemisphere.

Grain size

The grain size of quartz and biotite in natural samples varies depending on the size of the parent grains which varies depending on the nature of the protolith and pressure-temperature conditions (Table 1 and references therein). We could not directly compare grain sizes as it is difficult to discriminate from the references in Table 1 if the new grains result from dynamic recrystallization or metamorphic reactions, and as various grain sizes are often estimated only optically.

We can only underline that the grain size of our experimental samples is much smaller than the ones in natural samples (starting material: Quartz 10-20 μm and Mica 63-125 μm), which is due to the size of the experimental shear zone in the sample assembly (~1 mm), as we could use similar grain sizes to nature but would have only 2-3 grains. Despite of grain size differences between nature and experiments we observed similar microstructures, therefore the initial grain size does not affect microstructures and samples are comparable.

Piezometry

As first seen in chapter IV, adding a second phase as mica questions the use of piezometers is appropriate in or if other methods are necessary to evaluate stresses in these conditions.

When we plot our experiments (phlogopite + quartz) and the experiment from [Tokle \(2019\)](#), (muscovite + quartz) on the plot of [Stipp and Tullis \(2003\)](#) (Fig. 8) we see that two-phase system results are either higher or lower than the theoretical values predicted by the curve of [Stipp and Tullis \(2003\)](#). In our case, depending on how we discriminate our recrystallized grains (using the GOS as in [Cross et al., 2017](#) or using the grain size as a criteria) a non-neglectable uncertainty appears, and strong uncertainties are observed as well by [Tokle \(2019\)](#). Uncertainties and deviation to the theoretical values could be linked to the contribution of grain-size sensitive process (GSS) which participate to lower grain size.

The use of piezometers of quartz is not in favor of two-phase systems certainly, our results support conclusion of [Tokle et al. \(2023\)](#) and [Gottardi et al. \(2024\)](#) demonstrating that not accounting for phyllosilicate content can lead to incorrect results. Additionally, it is not usable for samples where diffusion creep is dominant as it was mainly constructed on the basis of dislocation creep dominant samples.

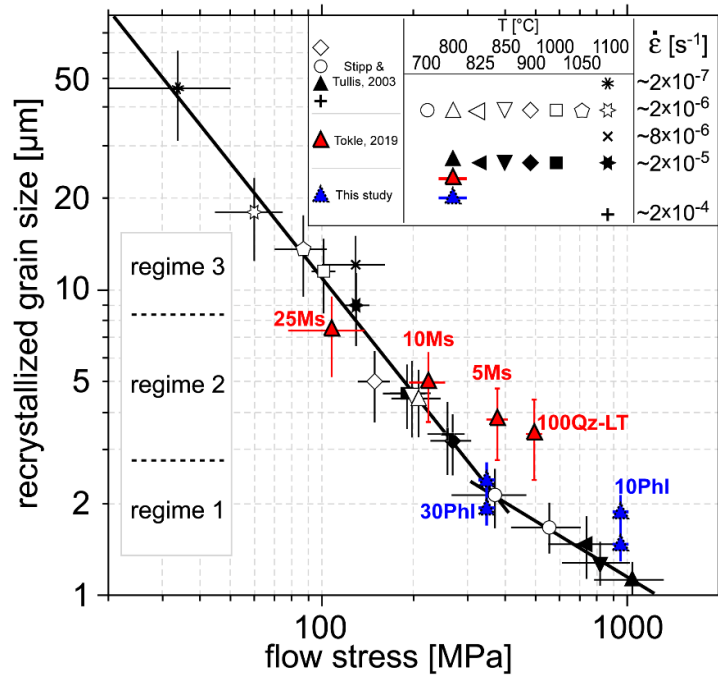


Figure 8: Recrystallized grain sizes for this experimental study and previous one of [Tokle \(2019\)](#) are plotted with the corresponding flow stress values on the diagram of recrystallized grain size paleopiezometer of quartz from [Stipp and Tullis \(2003\)](#). The two curves represent the relationships estimated by the paleopiezometer for the regimes proposed by [Hirth and Tullis \(1992\)](#). All experiments confining pressure is 1.5 GPa except our experimental ones with dashed contours where it is 1.0 GPa.

V.3.4. Evidence for dissolution-precipitation in quartz

Several studies in natural shear zones suggest that dissolution-precipitation in quartz is evidenced by random CPO of new quartz grains (Table 1 and references therein). In the Swiss Alps for instance, [Goncalves et al. \(2016\)](#) highlighted that precipitated new quartz grains (Qtz IIb here) have a different orientation from the parent quartz grains (Qtz I), whereas new quartz grains that originate from dynamic recrystallization preserve the heritage of parent grain orientation (Qtz IIa) (Fig. 9a). But other processes than dissolution-precipitation tend to randomize the CPO: in mature shear zones, the original CPO of Qtz I is progressively lost by the activation of grain boundary sliding along with diffusional processes (Fig. 9b-d, in this case the mica + epidote content is 30-35%).

In our experiments, as the fabric is weak and CPO is poorly defined, dissolution-precipitation in quartz is mainly evidence by overgrowths of Qz2 resulting from inherited grains Qz1 (Fig. 9e). Another evidence is the fact that blue luminescence Qz2 does not correlate only with perfectly with quartz grains observed on EBSD maps. Indeed, a crystallographic continuity is observed in the misorientations axis distribution of each grain and does not correlate only to yellow luminescence Qz1 grains, but to both yellow Qz1 and blue Qz2 grains (white arrows in Fig. 9f)

As a consequence, this implies in both natural and experimental samples that not only dynamic recrystallization microstructures characterize quartz deformation but dissolution-precipitation as well and depending on hydrated minerals content the influence of each will vary. In fact, it is suggested that mica dissolution-precipitation triggers and causes quartz dissolution ([Graziani et al., 2020](#); [Alaoui et al., 2023](#)).

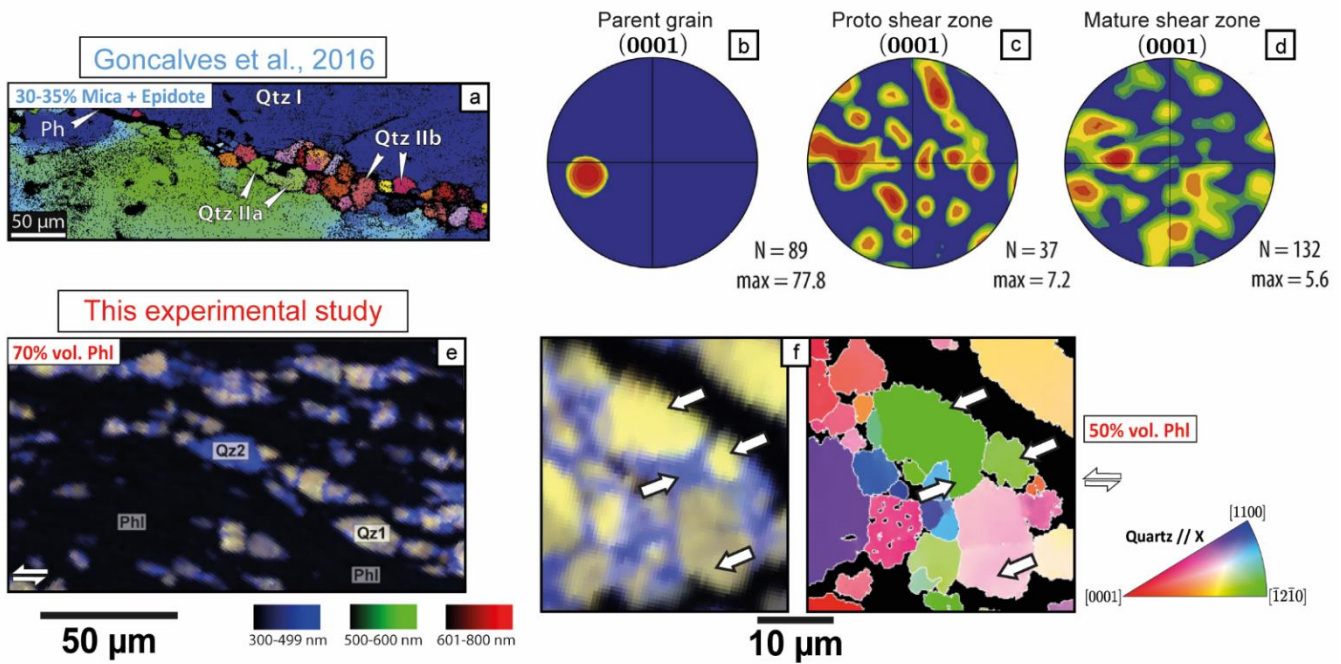


Figure 9 : EBSD map of quartz orientations parallel to the X direction a) in natural sample with 30-35% of Mica-Epidote from [Goncalves et al., 2016](#) with corresponding pole figures in natural sample b-d) from parent grain to mature shear zone. e) CL image of our experimental 70% vol. Phl sample and f) 50% vol. Phl with corresponding EBSD map of quartz orientations parallel to the X direction.

V.3.5. Evidence for dissolution-precipitation at mica-quartz interface

In the South Armorican shear zone ([Bukovská et al., 2019](#)), a brightfield transmission electron microscope (TEM) micrograph of focused ion beam (FIB) foil of orthogneiss sample exhibit white mica precipitation, at the interface of white mica and quartz, causing the dissolution of quartz and the formation of porosity (Fig. 10a). Another similar evidence for dissolution is provided by the dissolving margins of quartz grains in contact with mica folia ([Hippertt, 1994](#), sketched in Fig. 10b). The formation of dissolution interfaces can be influenced by subgrain boundaries. According to [Hobbs et al. \(1976\)](#), subgrain walls are planar structures with a high density of tilted dislocations that can serve as a preferred interface for fluid infiltration, precipitation, microfracturing, and dissolution.

In quartz porphyroclasts with dissolution interfaces, well-developed subgrain microstructures are rarely seen ([Hippertt, 1994](#)), suggesting that recovery has not resulted in a discernible decrease in the stored strain energy in these quartz porphyroclasts ([Meike, 1990](#)). This is consistent with the theory that porphyroclasts oriented with basal planes at low angles to the foliation are subjected to a single slip tend to be uniformly deformed, which increases the difficulty of recovery by dynamic recrystallization ([Urai et al., 1986](#)).

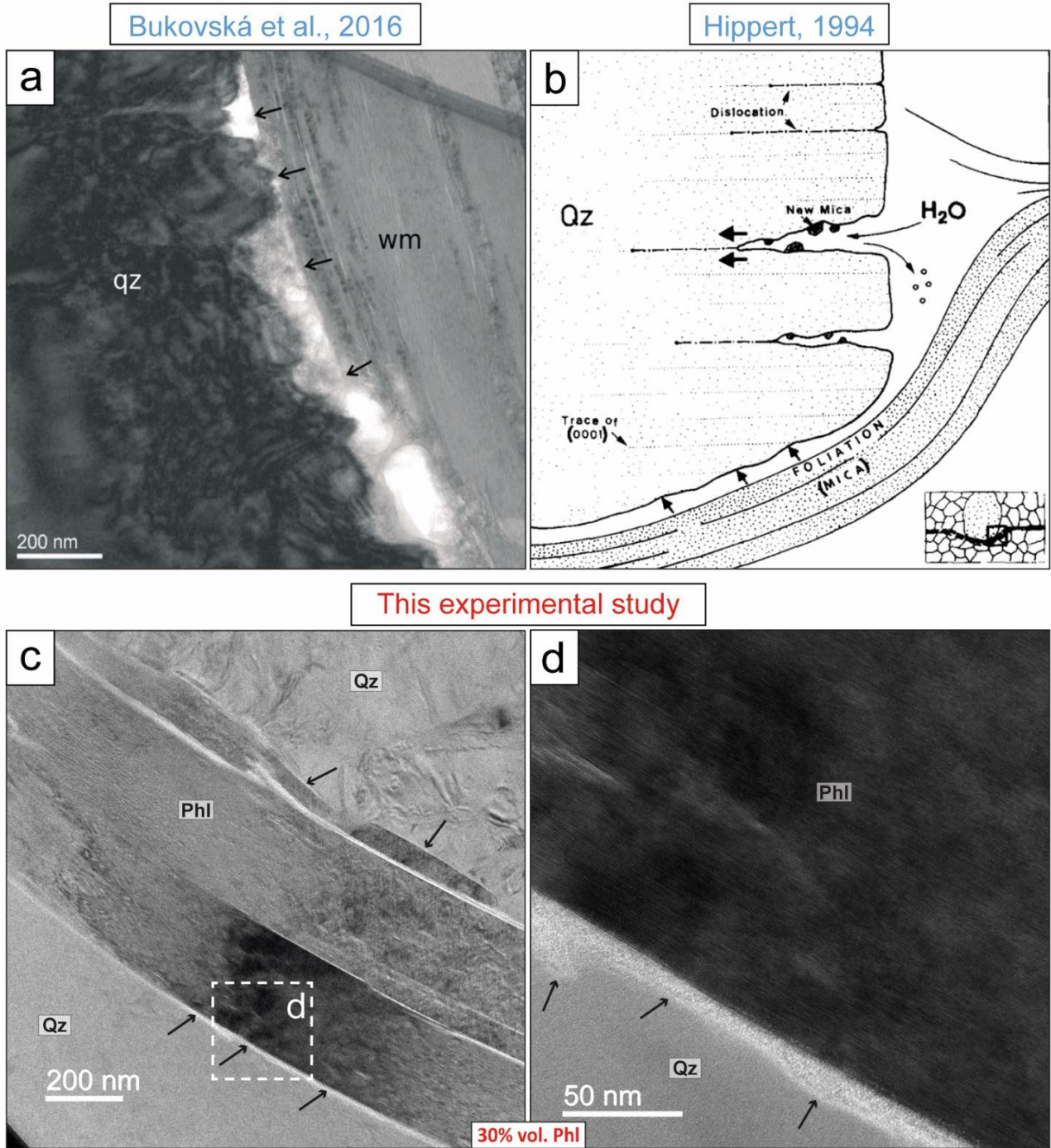


Figure 10 : Brightfield transmission electron microscope micrographs of a) natural sample from Bukovska et al., 2016 of interface of white mica an quartz, with black arrows showing dissolution of quartz. b) Conceptual model of dissolution process of quartz at interface with mica folia, with the sketch in the lower right corner showing the location of the figure (Hippert, 1994). Brightfield transmission electorn microscope micrographs of experimental 30% vol. phlogopite sample c) of phlogopite and quartz intefaces with black arrows showing amorphous layers and corresponding zoom in d) highlighting the undulating shape of this contour. Qz: quartz, wm: white mica, Phl: phlogopite.

As a result, one may anticipate that these grains dislocation density would rise, leading to strain hardening and possibly preferred disintegration (Wintsch and Dunning, 1985; Meike, 1990). It is suggested that the development of the dissolution preferentially interfaces along basal directions is driven by such a concentration of dislocation on basal planes (Hippertt, 1994, Fig. 10b). Hippertt (1994) proposed a model of formation of internal dissolution interfaces in quartz grains that deflect an adjacent mica-rich foliation. Dissolution interfaces are restricted to quartz porphyroclasts oriented with their basal glide planes at low angles to the foliation. Starting at the point where the dislocations cross the external surface, dissolution moves along the lines of dislocation. Dissolution releases the strain energy associated with the dislocation, facilitating pit development, therefore it should preferably occur where a dislocation line intercepts the external surface (Lasaga, 1983). The opening of basal interfaces, where fluid progressively infiltrates and allows dissolution to spread laterally and where new phase precipitation might occur, can be explained by the concentration of dissolution pits progressing along basal directions (Hippertt, 1994, Fig. 10b). The advancement of the internal dissolution front (big arrows in Fig. 10b) and the recrystallization of phyllosilicates are made possible by the percolation of H₂O in the opening interface. In the quartz-mica interface, an external dissolution front also acts (Bell and Cuff, 1989), producing an undulating shape (small arrows in Fig. 10b). The internal and external dissolution fronts should compete to each other (Hippertt, 1994). Beyond merely accommodating the progressive shearing component of deformation, phyllosilicates as highly anisotropic minerals play a more important role during deformation by improving the solubilities, diffusion rates, or rates of transfer of ions from nearby non-phyllosilicate minerals during dissolution and solution transfer (Kerrick, 1977; Bell and Cuff, 1986; Hippertt, 1994).

In the case of our experimental study, brightfield TEM micrographs of FIB foil of 30% vol. phlogopite sample exhibit contours composed of Si only (100%), lacking of diffraction pattern, which is interpreted as silica amorphous layer (Fig. 10c, d, see chapter V for details). These amorphous layers are parallel to sub-parallel to the basal plane (Fig. 10c), and are characterized by an undulating shape such as in the model proposed by Hippertt (1994) (Fig. 10b and d). However, the amorphous layer we observe are extremely thin and well defined compared to one observed by Bukovská et al. (2019). As a consequence,

aside from dynamic recrystallization, quartz deformation is also the result of diffusive mass transfer process (dissolution-precipitation) wherein ions move through the grain boundaries. The rate of this process depends on ion flow velocity, dissolution rate, and precipitation rate. The presence of fluids wetting the grain boundaries (increased with mica content) enhances silica transport, dissolution, and precipitation (Tullis and Yund, 1982). Therefore, mica enhances fluid flow, leading to creep and easier mineral dissolution.

VII.2. Conclusion

In this study, we aimed to investigate the role of mica in strain accommodation and development of shear zones in the upper to middle crust, from the scale of crystal defects to the polycrystalline aggregate. Several previous studies investigated the role of mica in strain accommodation in shear zones, however without detailing the deformation processes of mica in the case of natural samples, and without two-phase biotite-quartz system for experimental ones. Indeed, experimental studies on mica either used single crystals (Etheridge et al., 1973; Bell et al., 1986; Meike, 1989; Kronenberg et al., 1990; Mares and Kronenberg, 1993), polyphased systems (Holyoke and Tullis, 2006a, b) or natural samples as schist, gneiss, pegmatite (Shea and Kronenberg, 1992, 1993; Mariani et al., 2006; Misra et al., 2012). In the case of two-phase system with mica the amount of studies is poor and used muscovite only (Tullis and Wenk, 1994; Niemeijer and Spiers, 2005; Tokle et al., 2023).

Hence, we used a biotite-quartz two-phase system as upper and mid crustal rocks analogue, and studied in detail deformation and reaction mechanisms.

We identified dissolution-precipitation as weakening process in biotite-rich systems with solution-transfer diffusion creep localized at grain boundaries, which is enhanced by the presence of phyllosilicates, nevertheless accompanied by dynamic recrystallization in quartz.

Concerning the different deformation mechanisms in biotite we conclude on no significant intracrystalline plastic deformation except local kinking, and instead, grain size reduction primarily occurring through dissolution-precipitation processes rather than basal slip or dynamic recrystallization.

When increasing the proportion of mica to quartz aggregates, dissolution-precipitation mechanisms are particularly prominent, and reactivity of quartz, strongly contributes to rock weakening. Indeed, mechanical weakening is visible even for low fractions of micas, where the quartz is still the interconnected, load-bearing framework mineral.

The chemical composition of mica in the mineral assemblage influences interactions at grain boundaries between mica and quartz. Depending on the composition of dissolving mica under stress, the thickness and composition of the fluid-film at mica-quartz interface will be different influencing solution pH, phases solubility and therefore dissolution-precipitation.

VII.3. Perspective

Throughout this study, several questions have been revealed and still remain either entirely or partially unanswered, 4 main points appear and need further future work.

Experiments: repeating and supplementary

First and foremost, various experiments must be repeated and new ones must be added. For a better comparison, F-Phl and Bt experiments should be repeated at the same grain size than Phl experiments and with the same quartz (milky quartz). Phl experiments could be repeated at 30% vol. mica with Li-rich La Gardette quartz, in order to observe Li-rich quartz reworking with this type of mica.

Furthermore Phl-Qz experiments with various content shall be repeated to have at least a double of each for reproducibility, as it was done for the 30% vol. Phl ending up with a diverging unexplained mechanical behavior.

Concerning new added experiments, stepping experiments for the Phl-Qz assemblage would complete the set and finally give an n-value for biotite experiments as none were obtained yet experimentally, which is necessary to compare with the work done on muscovite by [Tokle et al. \(2023\)](#). This could also give more evidence on the necessity to obtain piezometer for two-phase system and not only pure quartz system applied to polyphase natural samples. Finally, experiments with muscovite instead of biotite with exactly similar conditions and similar quartz powder, grain sizes and proportions as our study would

give a direct comparison on how mechanical behavior, micro and nanostructures and associated deformation processes evolve with a different composition. Knowing overall that pure biotite and pure muscovite deform differently ([Meike, 1989](#); [Mares and Kronenberg, 1993](#)).

Supplementary characterization

As mentioned in the discussion above, no evidence in dynamic recrystallization of mica was observed it would be interesting to explore this point with EBSD on biotite and compare it with the results from [Aslin \(2019\)](#). Another lacking characterization, is TEM analysis on F-Phl samples in order to characterize nanostructures in F-Phl and eventually finding better evidence explaining the strength of F-Phl samples.

Image analysis

A question rising from grain size variation, mentioned in the discussion above, is what link it has with the strength of the assembly: (1) if the size of parent grains directly influences the strength of the assembly, or (2) the size of new grains or (3) the parent/new grain size ratio in one phase or (4) the grain size ratio between the two phases? (see BSE images chapter VI). As well as another point left to resolve is if quartz is mainly reworked in quartz levels, far from quartz-mica interfaces. One way to explore and eventually answer these questions is detailed image analyses (as done by [Mansard, 2017](#) using CIP or ImageJ) in order to give better exposure of grain boundary, grain interconnectivity, shape of phases and quantify differences between quartz-quartz, mica-mica and quartz-mica domains.

Water

Finally, characterization of water content in our experiments is lacking. The use of FTIR is considered to explore the water content between a cold undeformed sample, a hydrostatic sample and a deformed sample. Another technique considered is the nano-SIMS as our experimental sample are small and tedious to prepare for FTIR, and with this technique not only water amount would be characterized but also Ti content to better constrain the signals obtained in CL. As mentioned in chapter IV, CL signal might not be chemical signal and be neither a variation of Al nor Ti, but just a defect in oxygen.

Table 1: Review of papers on natural shear zones with hydrated minerals and the properties comparable to our experiments (Part 1).

Reference	Temperature (°C)	Pressure (GPa)	Stress (MPa)	Strain rate (s ⁻¹)	Water content	Metamorphic-facies conditions of deformation	Metamorphic reaction coeval with deformation	Grain size	
								Parent grains	New grains
Airaghi et al., 2020b	270-350 and 200-280	0.1-0.5	??	??	??	Greenschist	Mica and chlorite forming reaction (chloritization, sericitization)	Quartz: ~1.5 mm	Quartz: 20-200 mm; White mica: 10-30 mm (out of feldspar) and 20-100 mm (out of biotite)
Alaoui et al., 2023	350- 380	0.1-0.5	??	??	L.O.I.: 1.03-4.86 wt. %	Greenschist	Mica and chlorite forming reaction (chloritization, sericitization)	Quartz : > 50 mm; Biotite and Amphibole <1 to 4 mm (Bielsa)	Quartz: < 50 mm (Bielsa); 9-12 mm (Maladeta)
								Quartz: 17-26 mm (Maladeta)	White mica: 10-100 mm; Chlorite: 100-500 mm
Aslin et al., 2019	??	??	??	??	??	Amphibolite to upper greenschist	??	??	??
Berger et al., 2017	~450°C	0.6	??	??	??	Greenschist	White mica and chlorite forming reactions	White mica and chlorite: 100-400 mm	White mica: 3 mm
Berthé et al., 1979	400-450	0.5	??	1E-12	??	Greenschist	Yes	Medium grained granite of 2-5 mm	Quartz and Mica in C surfaces: 10-30 mm; Quartz

Bestmann et al., 2011	600	0.1	??	??	??	??	high-P amphibolite to greenschist	Yes	Quartz: up to 500 mm in quartzite	coarse grains: 150-300 mm
Bistacchi et al., 2012	250-300	??	??	??	??	??	Greenschist	??	??	Quartz: 1-2 mm (quartzite) and 0.5-2 mm (pseudotachylite)
Bukovska et al., 2016	300-350 (C fabric); >550 (S fabric)	0.1-0.4	??	3.8E-12-1.8E-14	??	??	Greenschist	Mica and chlorite forming reaction (chloritization, sericitization)	S fabric white mica: 0.5-4 mm; Quartz: 46 mm stage I	Quartz: 8-81 mm stage II
Fliervoet et al., 1997	350-450 (GS); 550-650 (Am)	??	??	??	??	??	Greenschist (GS) and Amphibolite (Am)	Mica forming reaction (sericitization)	Quartz: 60 mm (Amph); 10-200 mm (GS)	Quartz: 11 mm (Am), 0.5 mm (GS) Biotite: 5 mm (Am)
Goncalves et al., 2016	480 ± 50	0.85 ± 0.01	??	??	??	??	Greenschist	Yes	Quartz: up to 5 mm	Quartz: 10-30 mm
Goodwin and Wenk 1995	580-600	??	??	??	??	??	upper Greenschist to epidote amphibolite	Myrmekitization of feldspar and mica forming by sericitization	??	Overall average grain size 5 mm
Gottardi et al., 2024	345-485	??	32-69	2.4E-13-1.1E-14	??	??	Greenschist	??	Quartz: >500 µm	Quartz: 20-100 µm
Graziani et al., 2020	540-790 with cluster 720 ± 40	0.8 ± 0.2	??	??	??	??	Amphibolite to Granulite	Myrmekitization of feldspar and mica forming by sericitization	Quartz: 200-500 mm ; Biotite: 50-200 mm	Quartz ribbons: 10-500 mm; Biotite: >~50 mm

Gueydan et al., 2003	400	0.9	210	2.2E-14	1 wt. % in protolith	Greenschist and Blueschist	Mica forming reaction from feldspar	Quartz: ~5mm	Quartz: 100 mm
Herwegh and Jenni, 2001	340-400	??	??	??	??	Greenschist	??	Calcitic the matrix: 18-50 mm (from N to S)	Phyllosilicates in phyllosilicates-rich layers: 30-150 mm (from N to S)
Hippertt, 1994	300-450 and 500-800	??	??	??	??	Greenschist and Amphibolite	Mica and chlorite forming reaction (chloritization, sericitization)	??	Quartz: 30-200 mm
Hunter et al., 2016	490-530	??	??	??	??	Lower greenschist		??	Quartz: 60-350 mm (polyminerally), 61-86 mm (bimineralic); Muscovite: 40-84 mm
Ingles et al., 1999	??	??	??	??	??	Greenschist	Chloritization, sericitization, saussuritization and epidotization	Overall 1-2 cm	Quartz: 100 mm
Kanaori et al, 1991	??	??	??	??	??	Greenschist	Chloritization, illitization	Biotite: ~100-500 mm	??
Killian et al., 2011	500-550 ± 50	0.6-1.6	??	??	??	Greenschist	Myrmekitization of feldspar and K-feldspar and biotite forming reactions	Quartz: 110 mm	Quartz: 19-55 mm; Mica: <20 mm

Mancktelow and Pennachioni 2013	550-600	0.5-0.7	1-10	1E-11-1E-14	??	upper Amphibolite	??	??	Biotite trails a few mm
Mansard et al., 2018	300-350	0.1-0.4	??	??	??	Greenschist to lower amphibolite	Yes	Quartz: 120-170 mm (Protolith)	Quartz: 114 mm (mylonites domain 3) and 29-38 mm (mylonites domain 4)
Menegon et al., 2006	500-670	??	??	??	1 wt. % of aqueous fluid during mylonitization	Greenschist	Myrmekitization, perthite, oligoclase and chlorite formation	??	Quartz: 100-400 mm
Menegeon et al., 2008a	450-500	??	??	??	1 wt. % of aqueous fluid during mylonitization	Greenschist	Mica, epidote, myrmekite, oligoclase, perthite and chlorite formation	??	Quartz: 100-400 mm
Menegeon et al., 2008b	300-450	??	??	??	1 wt. % of aqueous fluid during mylonitization	Greenschist	Mica, epidote and chlorite forming reaction (chloritization, sericitization)	??	Quartz: 20-100 mm
O'Brien et al., 1987	450-500	??	??	??	??	upper Greenschist	Yes	??	Biotite: < 0.02 mm
Papeschi et al., 2018	650	0.18-0.2	??	??	??	blueschist to Greenschist	Mica, sericite and chlorite formation	Quartz: ~500 mm	Quartz: 10-100 mm; Sericite-biotite-chlorite aggregates: < 10 mm

Pennacchioni and Mancktelow 2007	550-600	0.5-0.7	1-10	1E-11-1E-14	??	upper Amphibolite	Fractures filled with epidote, chlorite or biotite developing bleached halos	??	Biotite and chlorite trails a few mm
Simpson, 1985	580-600	??	??	??	??	upper Greenschist to epidote amphibolite	Myrmekitization of feldspar and mica forming by sericitization	Quartz in ribbons: 100-300 mm (Amphibolite)	Quartz: 30 mm (Amphibolite); 5-15 mm (Greenschist)
Song and Ree, 2007	355-450	??	34-35 (paleostress); 22-25 (flow stress)	1E-16	??	middle Greenschist	Mica, chlorite formation	??	Muscovite: 2-98 mm (boundary), 1-25 mm (inclusions); Quartz: ~33 mm
Stenvall et al., 2019	450-480	0.27-0.42	54 (mylonite), 39 (ultramylonite)	1E-11-1E-12	??	Greenschist and Amphibolite	Mica, chlorite formation	Quartz: a few mm to >100 mm; Quartz ribbons: 47 mm; Fine-grained phyllosilicates: 50 mm (protomylonite)	Quartz ribbons: 29 mm; Fine-grained phyllosilicates: 50 mm
Stipp et al., 2002	250-700	0.25-0.3	up to 200 (flow stress)	1E-11-1E-14	??	lower Greenschist	Mica, aluminosilicate formation, chloritization	??	Quartz: 6-500 mm
Wehrens et al., 2017	250-450	0.65	??	??	??	Greenschist	Chloritization, epidote forming filling fractures	Quartz: <0.5-1 cm; Biotite: <0.3-0.5 cm	Quartz <180 mm; Biotite <0.5 mm
Wibberley, 1999	300	0.18	??	??	??	Greenschist	Mica and chlorite forming reaction	??	Quartz: 0.25-0.5 mm; Muscovite: 1 mm-10 mm

									(chloritization, sericitization)		
Wintsch and Yi., 1995	425-600	6	??		1E-14	??		Greenschist	Mymerkitization, epidote formation	Quartz: 3-5 mm	Quartz: 200-400 mm; Biotite: 300-600 mm

Table 1 : Review of papers on natural shear zones with hydrated minerals and the properties comparable to our experiments (Part 2).

Reference	Phase proportion in mylonite (if not it is indicated)		Rock Fabric	Phase distribution (random vs. clustered)	Quartz Microstructures			Phyllosilicate microstructures
	Quartz	Phyllosilicate or Hydrated minerals (HM)			CPO	Recrystallization microstructures	Evidence for dissolution-precipitation	
Airaghi et al., 2020b	??	??	S-C mylonite + fractures	Random	??	??	??	Recrystallization and kinking
Alaoui et al., 2023	30% (Bielsa)	40-50% (Bielsa); 20-28% (Maladeta)	S-C mylonite + fractures	Random	Weak, prism <a> (Bielsa), rhomb <a> and basal <a> (Maladeta)	BLG, SGR, undulose extinction, deformation bands	Quartz grains aligned along intragrain fractures, subgrain boundaries and zones of high dislocation densities	??
	38% (Maladeta)						new quartz grains isolated in matrix mica with random CPO	
Aslin et al., 2019	??	5-10% (protolith); 15-	Mylonite	??	??	??	??	Kinking, Ripplactions,

		20% (deformed samples)							Delamination structures
Berger et al., 2017	??	??	Mylonites to fault gouges	Random	??		BLG, SGR, undulose extinction, deformation bands	??	Bending into strain localization planes; new grains precipitated in quartz matrix; Mica recrystallization and shearing along (001) plane
Berthé et al., 1979	??	??	S-C mylonite + fractures	Random	Clear girdle type suggesting plastic deformation, prism <c>		Quartz recrystallization	??	Undulose extinction
Bestmann et al., 2011	60-40%	~10%	Pseudotachylyte veins	Random	Significant CPO in host grains		Undulose extinction, deformation bands and lamellae	random CPO of new small grains in intergranular microshear zones	Kinking of muscovite
Bistacchi et al., 2012	~60% mixed with Feldspar	20-25% (Orthogneiss); 40-45% (Paragneiss and micaschist)	Mylonites	Random to Clustered	??		??	??	Crystallization in shear fractures
Bukovska et al., 2016	??	??	S-C mylonite + fractures	Random	Significant CPO in S fabric lost progressively, mainly basal <a> some contribution of		Quartz recrystallization	random CPO in shear bands; white mica precipitation causing quartz dissolution at nanoscale	Interconnected layers; slip along mica bands

Fliervoet et al., 1997	40% (Am); 35% (GS)	10% (Am), 20% (GS)	Mylonite	Random	prism <c> and rhomb <a>	Weak, rhomb <a>	Undulose extinction, subgrain formation, polygonal new grains	??	??
Goncalves et al., 2016	??	30-35% in mature shear zone, 10-15% in transitional shear zone	Fractures evolving into shear zones	Random	Significant CPO in mature shear zones, basal <a> and rhomb <a>	SGR on the rim of inherited large grains	random CPO of new small grains in proto shear zones total CPO loss in mature shear zones	Crystallization in shear fractures precursor	
Goodwin and Wenk 1995	??	~10%	S-C mylonite	Random	??	Undulose extinction and occasional subgrain formation	??	Folded biotite but kinking is rare, intracrystalline deformation in relict grain, fine grain and grain boundaries. Mica fish. Subsequent cataclasis.	
Gottardi et al., 2024	??	4-18%	Mylonite	Random to Clustered	Type I cross girdles with basal, prisms and rhomb <a> slip	Dynamic recrystallization by SGR	??	??	
Graziani et al., 2020	~35%	>~10% (footwall); <~10% (hanging wall)	S-C mylonite	Random to Clustered	Overall weak; strong for samples poor in HM	GBM in HM poor samples	Biotite and amphibole crystals in interstitial	Biotite crystals in interstitial areas in feldspars aggregates, in pressure shadows	

								areas in feldspars aggregates	and matrix, no preferred orientation
Gueydan et al., 2003	40%	10% (west); 35-50% (east)	S-C mylonite	Random	??	dynamic recrystallization with deformation bands	??	??	??
Herwegh and Jenni, 2001	less than 3%	less than 7%	Mylonite	Random	??	??	Phyllosilicate grains coarsen suggesting mass transfer along grain boundaries	Bending. Embedded in calcitic matrix. Grains coarsen with increasing temperature.	
Hippertt, 1994	??	3-35% (quartzites); 60-90% (phyllonite)	Mylonite	Random	Significant CPO, basal <a> (quartzite), prism <a> to basal <a> (during phyllonitization)	Recrystallization in planar zones oriented parallel to the basal planes and oblique to the adjacent foliation	Quartz porphyroclasts with rim enriched in fluid inclusions, pressure shadows,	Bending and kinking associated with chloritization of biotite	
Hunter et al., 2016	~85% (Bimineralic); 74% (Polymineralic biotite-rich domain)	~15% (Bimineralic); 23% (Polymineralic biotite-rich domain)	Mylonites	Random	Decreases from quartz-dominated domains to micaceous domains; rhomb <a> and prism <a> (Quartz rich domain), minor	dynamic recrystallization	Mica causing pinning and reduction of recrystallized quartz	Mica fish, interconnected muscovite layers	

						basal <a> (Mica rich domains)				
Ingles et al., 1999	<30% (small-scale shear zones)	15% (small-scale shear zones)	S-C mylonite	Random	??	??	Limited to very high strain bands	??	Pinched and swelled	
Kanaori et al., 1991	??	??	Cataclasite in granite	Random	??	??	??	??	Kink bands, microboudinage and biotite fish	
Killian et al., 2011	30% ± 1.5	??	Mylonite	Random to Clustered	Strong CPO with basal <a> slip system	SGR, GBM, grain boundaries are pinned	random CPO in ultramylonite, grain size decrease during extension of polyminerallc aggregates	Fine grained biotite, no kinking or interconnected layers in the ultramylonite biotite precipitated between quartz grains		
Mancktelow and Pennachioni 2013	??	??	Mylonite + dykes and veins	Clustered	??	??	??	Biotite-rich trails		
Mansard et al., 2018	35-40% (mylonite); 46% (ultramylonite)	12-53% (mylonite); 25% (ultramylonite)	Mylonite	Random	Significant in quartz-layers prism <a> and rhomb <a>, weak in mixed layers	undulose extinction, subgrains, new small grains of quartz recrystallize within parent quartz, SGR	??	Interconnected white mica layers in high strain mylonite		
Menegon et al., 2006	27% (protolith)	13% (protolith)	Mylonite	Random	??	SGR and GBM	??	Bent and kinked		
Menegeon et al., 2008a	27% (protolith)	13% (protolith)	Mylonite	Random	type II crossed girdle dominant basal <a> slip	GBM in HIM poor samples	??	Local mica trails		

							with minor rhomb $\langle a \rangle$ and prism $\langle a \rangle$ slip during recrystallization	BLG, SGR, deformation bands and lamellae	random CPO of recrystallized grains in some samples	Muscovite and chlorite layers are locally present along the foliation and along recrystallized shear bands
Menegeon et al., 2008b	31% (protolith)	12% (protolith)	Mylonite + veins	Clustered	CPO varies strongly depending on % grain strain; basal $\langle a \rangle$ and rhomb $\langle a \rangle$ with minor prism $\langle a \rangle$	??	??	syntectonic recrystallization	??	Grain size reduction by cataclasis and syntectonic recrystallization
O'Brien et al., 1987	28% (protolith)	6% (protolith)	S-C mylonite	Clustered	Significant CPO	??	??	BLG, SGR, undulose extinction, core-and-mantle structures	??	Kinks and wide extinction bands perpendicular to basal cleavage plans
Papeschi et al., 2018	??	??	S-C mylonite	Clustered	??	??	??	??	??	Biotite-rich trails
Pennacchioni and Mancktelow 2007	??	??	Mylonite + fractures and veins	Clustered	??	??	??	Deformation bands. Subgrains and recrystallized new grains in irregular patches and	??	Kink bands. Recrystallization of very fine-grained new biotite. Biotite ribbons.
Simpson, 1985	??	??	Mylonite	Random	??	??	??	Deformation bands. Subgrains and recrystallized new grains in irregular patches and	??	Kink bands. Recrystallization of very fine-grained new biotite. Biotite ribbons.

Song and Ree, 2007	3-4% (mylonite matrix)	>95% (mylonite matrix)	Mylonite	Random to Clustered	Strong CPO with a type I crossed girdle (small mica fraction) basal <a>; Weak CPO (>3% mica fraction)	along ribbon boundaries. SGR, GBM, grain boundaries are pinned	??	Mica-rich matrix with interconnected layers, kink bands in muscovite
Stenvall et al., 2019	30% (protomylonite)	10% (protomylonite); 20% (mylonite)	Mylonite	Random	Significant CPO in protomylonite and mylonite, Lack of CPO in ultramylonite	Undulose extinction, minor subgrains and irregular grain boundaries	Strain-free nature of the grains and misorientation angles distributions close to the theoretical random distribution curve (ultramylonite)	Interconnected layers in protomylonite and mylonite, absent in ultramylonite.
Stipp et al., 2002	??	??	Mylonite + veins	Clustered	Stronger CPO with increasing temperature; Basal, rhomb and prism (BLG to SGR), Prism <a> (SGR to GBM) to Prism <c> (630°C)	undulose extinction, BLG,, SGR, GBM	biotite causing pinning and reduction of recrystallized quartz	??

Wehrens et al., 2017	20-30% (granodiorite); 32-35% (granite)	10-15% (granodiorite); <8% (granite)	Mylonite + dykes, veins, cataclasite	Clustered	??	intracrystalline plasticity and SGR	Grain size reduction accompanied by mica content increase, pinning of quartz	Biotite aligned subparallel to shear plane, recrystallized grains; white mica interconnected bands
Wibberley, 1999	33% (cemented ultracataclasite); 22% (phyllonite)	29% (cemented ultracataclasite); 52% (phyllonite)	S-C mylonite	Highly clustered	??	no evidence of dynamic recrystallization, dislocated quartz at nanoscale	??	Muscovite exhibits relatively low strain. Dislocated clasts exist within the cement
Wintsch and Yi., 1995	??	??	S-C mylonite	Clustered	??	Dislocation creep evolving in quartz layers	Solution transfer processes are present but not explicated for quartz	??

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La déformation des phyllosilicates et son effet sur la formation des zones de cisaillement dans la croûte moyenne et supérieure : une étude expérimentale des mécanismes de déformation de l'assemblage mica-quartz

Les mécanismes de localisation de la déformation dans les zones de cisaillement ductiles dans la croûte terrestre contrôlent fortement les dynamiques des orogènes, la nucléation de séismes et la formation de structures lithosphériques. La percolation de fluides lors de la déformation joue un rôle majeur dans le développement de ces zones de cisaillement par le biais de transformations minéralogiques comme la croissance de phyllosilicates par exemple les micas). L'objectif de ce projet est d'évaluer le rôle des micas dans l'accommodation de la déformation et le développement des zones de cisaillement dans la croûte supérieure à la croûte moyenne, de l'échelle de défauts cristallins aux agrégats polycristallins. Ce projet combine une étude microstructurale et pétrologique détaillée d'assemblages synthétiques mica-quartz déformés expérimentalement sous différentes conditions de température et de taux de déformation avec l'observation d'échantillon naturellement déformés. Un composant majeur de ce projet sera la caractérisation des mécanismes de déformation des phyllosilicates (intra-cristallin, polycristallin, agrégat de minéral biphasé), leur dépendance à l'égard de la composition chimique ainsi que le rôle des réactions minérales dans l'accommodation de la déformation. Le projet comprend l'utilisation de plusieurs techniques, telles que la presse de Griggs de nouvelle génération, le MEB et l'EBSD, la microsonde et le TEM.

Mots clés : Déformation Expérimentale – Réactions métamorphiques – Zone de cisaillement – Phyllosilicate.

Phyllosilicate deformation and its effect on shear zone formation in the upper and middle crust: an experimental study of the deformation mechanisms of mica-quartz assemblage

The mechanisms of strain localization within ductile shear zones in the Earth crust strongly control the dynamics of mountain belts, the nucleation of earthquakes and the formation of lithospheric structures. Fluid influxes during deformation play a major role in the development of these shear zones by means of mineralogical transformations such as the growth of phyllosilicates (as micas). The aim of this project is to investigate the role of micas in strain accommodation and development of shear zones within the upper to middle crust, from the scale of crystal defects to the polycrystalline aggregate. The project combines detailed microstructural and petrological study of the mica-quartz samples experimentally deformed under different temperature and strain rate conditions with the observation of naturally deformed samples. A major component of the project will be the characterization of the deformation mechanisms of phyllosilicates (intra-crystalline, polycrystalline, two-phased mineral aggregate), their dependency on chemical composition as well as the role of mineral reactions in strain accommodation. The project include the use of multiple techniques, such as the new generation Griggs apparatus, SEM and EBSD, microprobe and TEM.

Keywords: Phyllosilicate – Shear zone – Metamorphic reactions – Experimental deformation



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