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Pressure solution in rocks: focused ion beam/transmission electron microscopy study on orthogneiss from South Armorican Shear Zone, France

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Abstract

In order to characterize the μm -to-nm structures related to operation of pressure solution on phase boundaries in naturally deformed rocks, we have performed a detailed focused ion beam/transmission electron microscopy study in ultramylonite samples from South Armorican Shear Zone (France) that focused on grain boundary scale. We have studied phase boundaries between quartz, K-feldspar and white mica both in 2 and 3D and compare our evidences with theoretical dissolution-precipitation models in the current literature. The dissolution (re)precipitation processes lead to the development of different features at different phase boundaries. In both quartz-white mica and quartz-K-feldspar phase boundaries, voids were ubiquitously observed. These voids have different shapes and the development of some of them is crystallographically controlled. In addition, part of these voids might be filled with vermiculite. Amorphous leached layers with kaolinite composition were observed at the boundaries of K-feldspar-quartz and K-feldspar-white mica. The development of different features along the phase boundaries is mainly controlled by the crystallography of the phases sharing a common interface, together with the presence of fluids that either leaches or directly dissolve the mineral phases. In addition, the local dislocation density in quartz may play an important role during pressure solution. We suggest that the nano-scale observations of the quartz – white mica phase boundaries show direct evidence for operation of island-and-channel model as described in Wassmann and Stöckhert (2013), while K-feldspar – quartz phase boundaries represents amorphous layers formed via interface-coupled dissolution-reprecipitation as described by Hellmann et al. (2012).

Introduction

Pressure solution is an important mass-transfer mechanism driven by dissolution and (re)precipitation in rocks that contain intergranular fluids and may operate in a wide range of rock types and temperature, pressure conditions (e.g. Rutter 1976; Wheeler 1987; Spiers and Schutjens 1990; Stöckhert et al. 1999; Wassmann and Stöckhert 2012; 2013; Gratier et al. 2013). Dissolution precipitation is widely studied in laboratory experiments covering a broad scale of conditions. This includes studies on chemical and biochemical weathering (e.g. Hellmann et al. 2012; Daval et al. 2013; Bray 2014), archaeological findings damage (e.g. Rodriguez-Navarro and Benning 2013) or during mineral replacement (e.g. Putnis and Putnis 2007; Putnis 2009; Hövelmann et al. 2010; Putnis and John 2010) where precipitation occurs in the absence of external stresses and the process is purely chemically driven. Once dissolution precipitation occurs in presence of external stresses, such as in compaction studies (e.g. Renard et al. 2000; Miyakawa and Kawabe 2014), we refer to pressure solution. It is a well study process that can be driven not only by chemical changes but above all by deformation within the whole upper crust (e.g. Rutter 1983, Knipe and Wintsch 1985; Wintsch et al. 1991; Gratier et al. 2013). The effects of pressure solution have also been modeled in terms of shear zone initiation (e.g. Fletcher 1998) and in relation to processes connected to faulting and crack sealing during earthquake ruptures (e.g. Gratier et al. 2003). The main indicative features for operation of pressure solution at microscale are truncated grains, displacement of layering on certain planes if the contact is indented, stylolites, fibrous vein fill, fibrous overgrowth of grains in strain shadows and truncated chemical zoning patterns (e.g. Rutter 1983; Passchier and Trouw 1996; Wassmann and Stöckhert 2013). At the nanoscale strong evidence of dissolution-precipitation processes are rough surfaces of grains, where the surfaces of two adjacent grains across the partially opened phase boundary do not fit geometrically because their parts have been dissolved.

The general model for pressure solution at micro- to nanoscale suggests the presence of a thin fluid film/phase at the interface of different grains, so that these grains are completely wetted. However, pressure solution may occur as island and channel structures, where interconnected fluid pockets or films along the grain boundaries are separated by islands (e.g. Lehner 1995; Wassmann and Stöckhert 2013; and references therein). Adjacent grains within these islands form a regular grain or phase boundary. The model of island and channel flow by Wassmann and Stöckhert (2013) includes dissolution at interfaces with electrochemically rendered surface layer that is followed by short range diffusional transport in channels with diameter of 10 to 100 nm. The diffusional transport merge into fluid phase along channels connected to the grain edges by channels of 100 nm to 1 μm . The transport upscale through network of tubes and microcracks and later to large scale focused fluid flow in fractures. In this case the sinks might be represented by any free interface. During pressure solution grain and phase boundary dissolution normally occurs when

these boundaries are inclined at high angle to an instantaneous shortening direction, leading to high differential stresses in the region in the vicinity of the interface. Dissolution is followed by diffusive transport of dissolved ions within the fluid film and eventual (re)precipitation that may or may not occur locally. For the operation of such mechanism, the space for fluid transport has to be available, either in the form of porosity, partially-opened grain boundaries or fractures. In natural rocks such as granites and quartzites, the grains and phase boundaries are usually not perfectly straight, and many grain and phase boundaries might be partially opened (Kruhl et al. 2013) thus forming an interconnected network. If a fluid phase is present at the grain/phase boundary and the interface is simultaneously subject to high differential stress, dissolution occurs at the surface where solubility of a phase is enhanced and higher density of crystal lattice defects occurs.

There is a number of studies focusing on the experimental aspects of the pressure solution or dissolution-precipitation, most of them done on natural rocks. Some examples includes the importance of pressure solution on the fracture sealing (e.g. Knipe et al. 1993; Nelson et al. 1999), mylonite to ultramylonite development (e.g. Mancktelow et al. 1998; Menegon et al. 2008; Billia et al. 2013) and high pressure rocks (e.g. Wassmann and Stöckhert 2012; 2013) however, the link to the pressure solution on grain boundary scale is lacking.

In the present study we show microstructural evidences for pressure solution within the common crustal phase system of mica - quartz – feldspathic rocks via transmission electron microscopy (TEM) coupled with focused ion beam scanning electron microscopy (FIB-SEM) methods. Although there is number of papers focusing on pressure solution and dissolution-precipitation, we will mainly discuss the models of Wassmann and Stöckhert (2013) and Hellmann et al. (2012) for which we found direct evidence in our natural samples.

Geological setting and sample description

The South Armorican Shear Zone (SASZ) in European Variscides represents a major tectonic zone dividing the Central Armorican and the South Armorican Massif in France (Ballèvre et al. 2009; Fig. 1a). This subvertical shear zone has approximately WSW-ENE strike and consists of several branches spreading over hundreds of kilometers from westernmost Brittany to Vendée-Poitou region. The SASZ bears subhorizontal lineation, records right-lateral movement and is associated with development of S-C fabrics (Berthé et al. 1979).

Samples of an orthogneiss from one of the granitic plutons crosscut by the shear zone were chosen to study the interfaces of muscovite, quartz and K-feldspar. The original rock is a peraluminous leucogranite (Tartèse and Boulvais 2010) deformed syntectonically to orthogneiss. The deformation within the shear zone led to the development of S-C fabrics (Fig. 1b), that were

firstly described in this locality in the classical work of Berthé et al. (1979). The deformation evolution of these rocks includes different episodes of brittle and ductile deformation, where pressure solution plays an important role, as evidenced by quartz, K-feldspar and white micas that occur in the orthogneisses (Fig. 2). The studied S-C fabrics were formed with decrease of temperature connected to the cooling of plutons down to cataclasis (Tartèse et al. 2012). Numerous types of fluids (reaching from crustal fluids to meteoric water) were documented from the South Armorican Shear Zone. These operated during different stages of the cooling and shear zone activity (Tartèse et al. 2012).

The samples C fabric (shear bands) is parallel to the shear zone trace, where the orientation of maximum component of compressive stress on regional scale is oblique (at about 45°) to the shear zone. In order to relate the orientation of the observed pressure solution features, the orientation of the C fabric is shown in each figure and thus can be the features related to regional stress field.

The first evidences for the occurrence of pressure solution are already visible under the optical microscope, where mica grains form straight phase boundaries with quartz and K-feldspar grains (Fig. 2a and 2b, respectively). The quartz and K-feldspar phase boundaries are truncated by micas along the phase boundary, forming a new phase boundaries. The mineral assemblage that is present with the pressure solution features in the S-C fabric includes quartz, K-feldspar, white mica and chlorite.

Analytical techniques and sample preparation

TEM foils (0.15 µm thick) were prepared directly from preselected areas in the thin-section via the focused ion beam technique (FIB) in a FEI FIB200 instrument following the procedure described by Wirth (2009). We have studied TEM foils containing quartz – white mica and K-feldspar – white mica phase boundaries. The foils were cut both normal to the thin section surface and the phase boundaries. The TEM analyses were performed in a FEI Tecnai F20 X-Twin TEM operating at 200 kV. Both FIB and TEM are operating at the German Research Centre for Geosciences (GFZ Potsdam). Initial imaging and phase characterization via high angle annular dark field (HAADF) imaging coupled with energy-dispersive X-ray spectroscopy (EDS) was followed by high resolution TEM along the phase boundaries. We also performed FIB-SEM nanotomography along a quartz-muscovite phase boundary to observe its three-dimensional aspect via the Slice-and-View® approach. Through this technique, the 3D imaging of a certain volume of the target material is achieved by repeating the sputtering process with the focused ion beam intercalated with electron beam imaging in a Dual-Beam machine® (FIB/SEM), with the sample normally tilted at 52°. For this study we selected an area of 20 x 20 µm, where a protective layer of 1.5µm of Pt was first deposited to prevent FIB damage. We then sputtered material out from the frontal and lateral sides

of this region with the FIB operating at accelerating voltage of 30 kV and beam current of 30 nA. The frontal surface was then “polished” with the FIB operating at 3 nA beam current, and the slice-and-view process was conducted at the same conditions. The selected region was sliced normal to the grain boundaries. Electron imaging was obtained via the back-scattered detector via accelerating voltage of 20 kV and beam current of 8 nA, and each slice of sputtered material had 100 nm. All this procedure was performed in a FEI Quanta 3D FEG dual beam machine, also operating at GFZ Potsdam.

Results

Quartz – white mica phase boundary

The phase boundaries of white mica – quartz were studied in a foil where a muscovite grain neighbours two quartz grains (Fig. 3b). We selected a region affected by pressure solution, due to the straight phase boundary between mica and the quartz – K-feldspar matrix (Fig. 2, 3a), where the quartz and K-feldspar grains are crosscut by mica.

The mica grain has its c-axis perpendicular to the foliation and the phase boundary runs parallel to the white mica basal plane. Quartz has highly irregular phase boundary with the mica grain (Fig. 3b). Along this boundary, voids are opened towards the quartz grains (Fig. 3b) and in some locations muscovite interpenetrates quartz. Another important observation is that the white mica and the quartz crystal are rarely in direct contact to each other along the phase boundary due to the numerous voids along this boundary. These voids vary in size from few nanometers up to approx. 100 nm (Fig. 4) and have different shapes. They might have been filled with a fluid that has escaped into the vacuum during sample preparation or might be filled with vermiculite as revealed by EDS.

The roughness of the quartz grain surface is pronounced, and is visualized by the dark contrast of quartz imaged with a low indexed zone axis parallel to the electron beam (Fig. 4). The locations in quartz where voids penetrate deeper into quartz grain are characterized by higher dislocation density, with the dislocation lines directly penetrating through to the surface of the grain (Fig. 4b, 5a, 5b).

In this foil (Fig. 3b), the dissolution of quartz produces very different features in two neighbouring quartz grains. In the upper grain, the quartz surface is extremely irregular (Fig. 4) and voids vary from trapezium to triangle shapes, with smaller embayments into the quartz. The lower grain on the other hand is triangular-shaped with straight and oriented crystal surfaces (Fig. 5). The void spaces are filled with nanocrystalline, needle-like vermiculite. The surface of quartz in the lower grain with triangular voids (Fig. 5a) is smooth. Determination of the crystallographic orientation of the phase boundary planes was carried out via high resolution TEM (HRTEM, Fig.

5b) and fast Fourier transform (FFT) of HRTEM lattice fringe images. Fig. 5c is a dark-field image (01-10 reflection) of the same location as in Fig. 5a. The respective planes of a triangular void represent crystallographic positive and negative rhombs (01-11) and (10-11) (as depicted at Fig. 5a, b).

In order to document the phenomena of dissolution of quartz boundaries at the contact to mica in three dimensions, we performed a nanotomography study (Fig. 6 a-d and Supplementary material) via FIB/SEM in the same location where the foils were extracted (Fig. 3a). The phase boundary separates quartz and mica grains (Fig. 6a - d, left and right respectively) with a small K-feldspar grain that is grown in between quartz grains (Fig. 6b, c, d). The voids are shown as these appear along the phase boundary causing increase in phase boundary porosity. The movie documents the phenomena of partially opened phase boundaries, as well as opening and closing of the dissolution sinks (voids; Fig. 6), detail illustrated in the Figs. 4 and 5.

Quartz – mica dissolution features were also found in quartz enclosed in micas (Fig. 7a, d) that are crosscutting the quartz – K-feldspar (Fig. 2b) matrix. Quartz has highly irregular grain shape (Fig. 7a, d), that is better visualized in the dark field image (Fig. 7c). Quartz grains document different dissolution features that depend on the orientation relationship between adjacent mica and quartz grains. Partially opened grain boundaries represented by pores along the phase boundary of mica and quartz are also commonly observed, depending on the grain orientation (Fig. 7a).

Small dissolved voids are found along the quartz phase boundary neighbouring with mica basal plane (Fig. 7b, e) with dislocations connected to the tips of dissolved voids (Fig. 7b). In those locations where the basal plane of mica is (approximately) perpendicular to the quartz grain, mica grew over quartz and penetrated into its structure (Fig. 7e). In other parts of the phase boundary, no voids are observed, but amorphous material occurs (Fig. 7e). The mica basal plane is inclined at an angle of ca. 45° with respect to the quartz surface, as documented by HRTEM by the presence of fringes of both grains (Fig. 7f) and measured with FFT of HRTEM (Fig. 7g). The original thickness of the amorphous layer is approximately 15 nm, but the irradiation damage of quartz during imaging caused the thickness to increase to 29 nm as shown on Fig. 7f. The observed dissolution features are closely connected to the preferred orientation of the mica.

K-feldspar – white mica phase boundary

In the orthogneiss matrix, quartz is neighbouring K-feldspar (Fig. 2b). The evidence for pressure solution was therefore also expected to occur along the K-feldspar – white mica grain boundaries because the K-feldspar grains are truncated at the contact to mica in a same way as quartz. The partially opened phase boundaries of both phases are rough and do not correspond in shape to each other, suggesting partial dissolution of one or both phases. The phase boundaries contain numerous

Kommentiert [1]: Supplementary material

pores (Fig. 8a), especially when the basal plane of mica is parallel to the phase boundary with K-feldspar (Fig. 8a, d). When the mica basal plane is inclined at high angle to the phase boundary with feldspar, the former tends to grow over the latter (Fig. 8b, c). The mica initially grows over the K-feldspar grains, which at more evolved stages leads to disaggregation and isolation of smaller K-feldspar or quartz grains, such as the smaller K-feldspar grain in Fig. 8c (in the upper part of the image) isolated from the larger K-feldspar grain in the same image. This process also affected the smaller quartz grain nearby.

Another related feature found in direct contact with the phase boundaries affected by dissolution are low-angle grain boundaries. In a bulged K-feldspar grain, a low-angle grain boundary evolves in continuation of mica – K-feldspar interface (Fig. 8b). Often these are connected to the voids at the opened K-feldspar – mica interfaces (Fig. 8d). Higher dislocation density is present close to the interface (Fig. 8e).

Amorphous phase may also be developed along parts of the partially open phase boundaries between mica and K-feldspar (Fig. 8f). The phase is localized directly at the partially opened K-feldspar phase boundary and may contain some porosity (Fig. 8f, 9a, 9b). The interface of K-feldspar and amorphous phase is straight and very well visible because the diffraction contrast of K-feldspar ends abruptly at the interface (Fig. 9c) with non-crystalline material. Chemical composition of this amorphous phase resembles that of kaolinite. Even though the K-feldspar is dissolved in a different way in comparison to quartz, the dislocations are still connected to the phase boundary and proceed into the K-feldspar grain (Fig. 8f, 9b).

Discussion

Observations of grain boundaries within quartz / K-feldspar and mica within crustal rocks show complex pressure solution features, such as truncated grains and phase boundaries. The studied grain and phase boundaries show presence of amorphous material, numerous partially opened grain boundaries and voids, resulting in the development of porosity.

Although the pressure solution processes are normally explained in terms of the effect of local high and low stresses acting in rocks in the presence of fluids, for certain rock types this might not be the case. Recently, Kruhl et al. (2013) has shown that partially opened grain and phase boundaries are commonly observed in different types of rocks, such as quartzites, granitoids and marbles. Our observations support this study and document a number of partially opened interfaces which serve as fluid pathways especially at the late stage of the rock evolution at temperature < 300°C, which is the temperature for the brittle-ductile transition in quartz (Voll 1976). The presence

of partially open grain and phase boundaries with highly irregular surfaces, where the geometries of the boundaries of opposite neighbour crystals do not match, is a clear evidence of partial dissolution along these interfaces.

The orientation of maximum compressive stress at the grain scale is complicated to assume therefore we can only make an estimate. The maximum compressive stress at the regional scale is oriented approximately at 45° to the shear zone (Fig. 10a). If we extrapolate the stress orientation towards the microscale, the maximum stress would be at 45° to the shear band (C fabric orientation) in the thin section plane (Fig. 10a). These shear bands contain mica grains that are aligned within the fabric. Thus the orientation of the quartz – mica interfaces is parallel with the shear zone. Looking at the TEM foils the stress (σ_1) extrapolation is not that straight forward (Fig. 10b). Thin foils that were prepared for this study were cut from the thin sections perpendicular to the C fabric and at the same time mica basal planes as shown at Fig. 10. Therefore the orientation of maximum compressive stress on the TEM foil is not oriented at 45° to the mica C fabric. Assuming the foil as a plane we need to project the maximum compressive stress orientation which is thus perpendicular to most of the observed interfaces and mica basal planes. However this should be taken into account as an assumption since we do not have any control on the local stresses.

If we assume the maximum compressive stress orientation to 45° to the interface the effect of the maximum compressive stress to the interface with respect to the pressure solution might be questionable. Experimental works mostly reveal the maximum compressive stress being perpendicular to the interface however, observed white mica – quartz interfaces with newly formed porosity have the maximum compressive stress oriented at 45° .

The dissolution of quartz when in contact with the white mica appears to be crystallographically controlled, as evidenced by the positive and negative rhomb planes of quartz in contact with the (001) plane of mica. At the same interface, irregular voids with trapezoid and triangle shapes are formed in quartz crystals of different orientations. These features are also observed in the nanotomography method, confirming the extent of this phenomena in 3D. As discussed by Putnis and John (2010), the newly established porosity generated by dissolution allows fluid to be transported and precipitated with no need for solid-state diffusion. Similar voids were observed by Mancktelow et al. (1998) and Billia et al. (2013) in quartz or quartzo-feldspathic mylonites to ultramylonites, where grain boundary pores are expected to represent transient porosity evolved through dislocation-localized dissolution during dislocation creep of the ultramylonite. Similar features were also observed by Gratier et al. (2005) as experimental microstylolites in quartz where the maximum compressive stress was also perpendicular to the microstylolite interface.

At the white mica – quartz interfaces can be found both dissolution interfaces represented by

amorphous or leached material and open voids in the same orientation. This suggests that there is strong effect of stress which can locally either cause leaching or open voids.

The model of island and channel structure based on the presence of a thin fluid film at the interface boundary explaining pressure solution operation on micro – nanoscale described by Wassmann and Stöckhert (2013) appears to be valid for the observations in the orthogneiss samples reported here. According to their model, the dissolution precipitation creep requires at least one fluid and one solid phase, chemically open system and association of dissolution precipitation with chemical changes. In case of the studied samples, the parts of the grain not dissolved represent the islands in the island and channel structure model, while the voids represent the channels of this model. The dissolved areas are commonly filled with vermiculite, which represents residual precipitated material from dissolved phases. The assumed orientation of compressive stress is also preferable for this model.

The model for dissolution precipitation creep in the sense of Wassmann and Stöckhert (2013) further comprise: (i) interphase dissolution that might be restricted to load-bearing islands with thin fluid film; (ii) diffusional transport into network of channels surrounding islands, (iii) diffusional transport through fluid along the channels; (iv) transport through 3D network of tubes and microcracks and (v) large scale focused fluid flow in fractures. The observed combination of voids with partially opened boundaries present numerous ways where fluid flow may operate within the system.

The chemical composition of K-feldspar and its boundary layer has been evaluated by EDS. The amorphous material layer observed in the K-feldspar interface represents K-feldspar leached by fluids operating at this interface, most probably at very low temperatures. In the leached layers, K is removed out of the structure of K-feldspar and an amorphous layer rich in silica and aluminum is preserved with a similar composition of kaolinite. The presence of amorphous layers formed via interface-coupled dissolution-reprecipitation was also observed by Hellmann et al. (2003) in experimentally altered plagioclase and Hellmann et al. (2012) in chemical weathering studies in multiple silicates. In both cases, the development of the leached layer was interpreted by the activation of dissolution reprecipitation mechanisms along the minerals interfaces (Hellmann et al. 2012) although without the presence of external stress and in free fluid. The main arguments of Hellmann et al. (2012) that unify laboratory and field chemical weathering were: (i) sharp chemical interfaces in the form of crystalline-amorphous boundary; (ii) cation depletion depths; (iii) fast rates providing the surface layers significantly exceeding diffusion rates. According to Hellmann et al. (2012) the main important processes include dissolution in a thin fluid film with pristine mineral lattice, coupled precipitation of porous amorphous phase and precipitation of amorphous silica even in undersaturated solution. Our observations show nanometer-sharp boundaries at the crystalline-

amorphous interfaces, which seem to be formed at a single reaction front. Presented observations therefore support the conclusion of Hellmann et al. (2012) that interfacial dissolution reprecipitation is universal mineral-water alteration mechanism also in the presence of external stresses. Moreover the alteration of feldspars leading to its breakdown, growth of muscovite and the loss of K into fluid was shown to be significantly important in mylonites (Knipe and Wintsch 1985). The grains of quartz and K-feldspar might be dissolved preferentially because of its high dislocation density that may increase their solubility (Wintsch and Dunning 1985).

The leached layer formation is extensively studied mainly with respect to weathering and building materials damage either through solid-state transformation (e.g. Casey et al. 1993) or through interface-coupled dissolution and subsequent precipitation (e.g. Hellmann et al. 2012; Ruiz-Agudo et al. 2012). We have observed amorphous phase with evolving porosity (Fig. 8f) that is assumed to be formed through interface-coupled dissolution-precipitation. The presence of newly formed porosity represent another possibility for fluid transport. Amorphous material is found at the surfaces which are parallel or sub-parallel to the shear zone and perpendicular to the maximum compressive stress. There is only a small amount of the amorphous material thus we do not assume that it will effect the rheology of the rock. Similar silica gel material was observed in experimentally deformed granite by Di Toro et al. (2004) who documented dynamic weakening of granite with silica gel formation during sliding on fault.

Conclusions

The evidence for activity and progress of pressure solution mechanism is shown on an example of a quartz – mica – K-feldspar grain boundaries in orthogneiss from South Armorican Shear Zone. The partially open phase boundaries allow fluids to wet the interface and therefore allow the pressure solution to occur at a broad scale of conditions.

The process of pressure solution appears to be fairly complex and driven by more than the stress concentration at the interface or chemical composition of fluid. The observations confirm the validity of island and channel model of Wassmann and Stöckhert (2013) and model of leached layers presented by Hellmann et al. (2012). Our data show that within the presence of external stresses the respective crystallographic orientation of phases plays a major role, as well as the fluid presence that directly dissolve the mineral phases, and the local dislocation density or more general the defect density. As shown from the dissolved quartz grains at the interface to mica, the dissolution appears in different locations, where preferentially quartz is being dissolved preferentially along crystallographic surfaces that represent rhomb planes. The crystallographic orientation of mica with respect to the dissolved phase is important, where the dissolution can form

the void dissolved sinks, amorphous layers or leached layers even at the same orientation of maximum compressive stress. At the same time the mica might be overgrowing the dissolved phases and thus causing grain size reduction That is significantly important because of the rock rheology and deformation mechanism change as well as the increase of fluid path that is being induced. On a larger scale the grain size reduction is important mechanism allowing plate tectonic processes (Platt and Behr 2011). Moreover the stress effect in the shear zone can cause both opening of voids and dissolution at the same time.

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Figure captions

Fig. 1 a) Geological map of Armorican domain in Brittany, France with location of South Armorican Shear Zone and sample location (white star). b) Sample of orthogneiss with S-C fabric and given orientation of maximum compressive stress (σ_1) with respect to the S and C surfaces

Fig. 2 a) Optical micrograph (crossed polarizers) of orthogneiss composed mainly of quartz, K-feldspar and muscovite. The arrows point at locations with pressure solution features such as sheet silicates in contact with quartz or K-feldspar and flattened grain shape. Orientation of maximum compressive stress (σ_1) is given with respect to the S-C fabric. b) BSE image of orthogneiss with matrix formed by quartz and K-feldspar with plagioclase porphyroclasts that is crosscut by muscovite. The white arrows indicate quartz – muscovite interfaces and black arrows K-feldspar – muscovite interfaces where pressure solution process was studied. Abbreviations: kfs – K-feldspar, ms – muscovite, pl – plagioclase, qz – quartz

Fig. 3 a) Optical micrograph (crossed polarizers) of orthogneiss sample with quartz – muscovite grain boundaries where the TEM foil was prepared and FIB 3D slice-and-view produced. The phase boundary area is marked with white line over the hole that was sputtered during FIB slicing. b) The high-angle annular dark field (HAADF) image of the foil cut across the quartz – muscovite phase boundary with location of figures 3 a, 3 d and 4 a along the phase boundary indicated. Porosity along the grain boundaries is visible as dark spots or elongated contrasts. The underlying carbon grid the foil rests on forms the bubble structure below the foil. Assumed orientation of maximum compressive stress (σ_1) is given to each figure. Abbreviations as in Fig. 1

Fig. 4 TEM bright-field images of a quartz – muscovite phase boundary. Quartz grains are dissolved and voids have been formed. Newly formed vermiculite is found within the opened phase boundary and the voids. The dislocation density increases towards the quartz phase boundary. The shadow of the underlying perforated carbon film is visible in some locations of the images. a) Quartz grain with high dislocation density and the location of the detailed images 3 b and 3 c indicated as boxes. b) Detailed image of the interface quartz-muscovite showing very rough surface of quartz with many dissolved embayments on a different scale. White arrows show the dislocation connected to the end of voids thus indicating preferential solution of the quartz in the vicinity of the dislocation core. c) Protruding embayment into the quartz grain partially filled with vermiculite growing on the dissolved quartz surfaces. d) Pronounced almost triangular voids with straight quartz surfaces. The voids are filled with vermiculite. Assumed orientation of maximum compressive stress (σ_1) is given to each figure. Abbreviations: ms – muscovite, qz – quartz, vrm – vermiculite

Fig. 5 a) Composite TEM bright-field image of dissolved quartz interface quartz-muscovite with triangular voids. Note the straight surfaces of quartz correspond to (01-10) and (10-11) crystal faces. The now opened phase boundary is filled with vermiculite. b) Diffraction pattern (FFT of HREM image) of the partially dissolved quartz grain. c) Corresponding dark-field image of quartz grain with dislocations connected to the tips of triangular voids marked by white arrows. Assumed orientation of maximum compressive stress (σ_1) is given to each TEM figure. Abbreviations as in Fig. 3

Fig. 6 a) - d) FIB images from 3D slice-and-view of the quartz (left) and muscovite (right). The opening and closing of larger voids along the grain boundaries (white arrows) is demonstrated in the movie (supplementary material). The precipitation of small K-feldspar grain (white) is possible because of presence of partially opened grain boundaries (grey arrows) which allows the fluid flow

Fig. 7 Quartz – muscovite grain boundaries of small quartz grains enclosed in a muscovite matrix. a) HAADF image of two quartz grains within muscovite with numerous voids along the quartz-muscovite interface. The bright contrast surrounding the pore between quartz and muscovite is caused by redeposited gallium during FIB foil preparation thus indicating an open pore space prior to FIB milling. The location of Fig. 6b and 6c is indicated by white rectangles. b) TEM bright-field image of quartz grain with dark diffraction contrast together with small voids (black arrows) along the phase boundary quartz muscovite at the basal plane of muscovite. The voids are filled with non-crystalline material. The tips of voids mark the endpoints of dislocation lines (white arrows) that protrude out of the quartz grain surface. c) TEM dark-field image of a quartz grain with dissolved surfaces imaged at different scale. Note the in form of small embayments or much more altered grain boundaries resulting in an irregular shape of the grain (some of them marked by white arrows). d) HAADF image of quartz grains within muscovite with location of Fig. 4 e. e) TEM bright-field image of quartz surrounded by muscovite. Pressure solution is different depending on the orientation relationship of quartz and muscovite. The quartz grain with small embayments (marked by black arrows) at the interface parallel to the basal plane of muscovite. At those locations where the muscovite basal planes of muscovite are approximately perpendicular to the quartz surface, the muscovite grows into the quartz (white arrows). Other quartz-muscovite interfaces display amorphous material at the quartz- muscovite phase boundary (Fig. 6f). f) TEM lattice fringe image of the quartz-mica interface. In the lower left corner and in the upper right corner lattice fringes from quartz and muscovite are visible. The width of the amorphous material is 29 nm, however, the original extent measured was only 15 nm. During the exposure time of the sample to electron irradiation during HREM imaging quartz and muscovite are damaged resulting in disappearing lattice fringes of both minerals. Quartz is more affected by irradiation damage than

muscovite. g) Diffraction pattern (FFT of HREM Fig. 6 f) of the interface region. Assumed orientation of maximum compressive stress (σ_1) is given to each figure

Fig. 8 a) HAADF image of a K-feldspar – muscovite interface which is partially opened. The partially opened phase boundary is displayed by dark contrasts indicated by black arrows. b)-f) TEM bright-field images of K-feldspar – muscovite phase boundaries. b) K-feldspar – muscovite phase boundary. The K-feldspar bulges into the muscovite. The bulged area of K-feldspar is separated from the K-feldspar by a low-angle grain boundary approximately at the position of the original interface K-feldspar - muscovite. Note the porosity at the tip of the bulge extending parallel to the basal planes of muscovite. c) K-feldspar grain being disintegrated by growth of muscovite (white arrows). d) Partially opened phase boundary of muscovite and K-feldspar (black arrows). Low-angle grain boundary in K-feldspar indicated by white arrows. e) Dislocations (white arrows) protruding from the muscovite – K-feldspar phase boundary into the K-feldspar grain. f) Amorphous material (black arrows) at the K-feldspar - muscovite phase boundary forming patches on the K-feldspar surface. White spots present epoxy from sample preparation. Assumed orientation of maximum compressive stress (σ_1) is given to each figure

Fig. 9 a) HAADF image of a K-feldspar – muscovite interface with amorphous material at the K-feldspar surface with location of Fig. 8b and 8c indicated. b) Bright-field image of the interface with amorphous material at the K-feldspar surface (white arrows) and dislocations connected to the surface (black arrows). Amorphous material is represented by the absence of diffraction contrast. White areas at the interface represents epoxy from sample preparation, white spots within K-feldspars represent inclusions. c) HREM image of the interface of K-feldspar (crystalline, upper part) and amorphous material with approximate kaolinite composition (lower part). Assumed orientation of maximum compressive stress (σ_1) is given to each figure

Fig. 10 Extrapolation of stress orientation from regional scale to the mineral interfaces. a) Orientation of maximum compressive stress (σ_1 , black arrow) at the regional scale applicable to the shear zone, at microscale within the thin section with respect to the S-C fabric and on nanoscale in the case of mineral interfaces of mica and quartz. b) Extrapolation of the σ_1 orientation from thin section to the perpendicular thin foil prepared for TEM. In order to assume the stress at the phase interface the foil is assumed to be planar and the σ_1 is suitably projected.

Supplementary material captions

3D slice-and-view movie along the quartz – white mica interface. The movie shows an interface of quartz (left) and white mica (right). The dissolution processes formed a numerous voids along the

phase boundary of the two mineral phases, which are opening and closing – documenting 3D character of the voids. Many of the voids are formed at lower left part, where the protruding voids move into the quartz from the interface of mica. Small grain of K-feldspar (white) occurs along the grain boundary of two quartz grains and in its vicinity another mica grain is grown. Just at the beginning of K-feldspar slicing the pronounced opened grain boundary in between two quartz grains is present. With the growth of mica numerous grain boundaries are more or less opened. Some of the voids are filled with vermiculite (as shown on Fig. 4, 5)

Fig.1

Fig.2

Fig.3

Fig.4

Fig.5

Fig.6

Fig.7

Fig.8

Fig.9

Fig.10