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Compositional re-equilibration of garnet: the importance of sub-grain boundaries

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Abstract: Garnets from meta-granitoid high pressure rocks (Sesia Zone, Western Alps) show complex internal sub-grain textures in electron forescatter images. All investigated garnets consist of a large number of sub-grains with different shapes and sizes. Some garnets exhibit a sub-texture with very fine-grained (< 20 μm) sub-grains in their cores overgrown by palisade-like sub-grains in the rims. Sub-grain boundaries in these garnets have enabled diffusive element exchange between the garnet core and the surrounding matrix. Compositional mapping reveals zonation patterns of Mg that indicate modification of the garnet composition during prograde metamorphism. The extent of diffusional re-equilibration is dependent on sub-grain size and element diffusivities. Our samples show that X_{Mg} is strongly influenced by diffusion along the sub-grain boundaries, whereas apparently slow diffusing elements, such as Ca, Ti and Y preserve their original concentric zonation pattern. This differential re-equilibration leads to very complex chemical zonation that cannot be easily interpreted in terms of simple prograde growth zonation or of normally-applied spherical diffusion models. The observation that almost all garnets in the investigated samples exhibit a sub-grain pattern suggests this might be a common feature in high pressure/low temperature rocks.

Key-words: garnet, microstructure, sub-grains, EBSD, diffusional equilibration, high pressure, subduction, Sesia Zone.

Introduction

Understanding compositional re-equilibration of metamorphic minerals as a response to changing pressure (P), temperature (T) and/or chemical conditions is one of the most important aspects of metamorphic petrology. Diffusional relaxation of gradients in chemical potential within and among minerals is both a blessing and a curse for petrologists. On the one hand, relaxation of T- and P-dependent gradients in chemical compositions of minerals blurs information about previous metamorphic stages, thus complicating exact determination of P-T-paths (*e.g.* Florence & Spear, 1991) or of growth and fractionation processes during metamorphism (*e.g.* Carlson, 1989; Konrad-Schmolke *et al.*, 2005). On the other hand, as long as the mechanism of diffusional relaxation and its parameters are known, characteristic diffusion-induced chemical zonation within mineral grains can be used to constrain temperature-time histories (*e.g.* Lasaga & Jiang 1995; Perchuk & Philippot 1997; Ganguly & Tirone 1999) by calculating the effects of diffusion on pre-existing growth patterns and comparing the so-modelled profiles with those in natural samples.

It is widely accepted that transport rates differ significantly between volume and grain-boundary diffusion, the latter being up to four orders of magnitude faster (*e.g.* Joesten, 1991). Thus, compositional resetting of mineral grains along fractures and sub-grain boundaries occurs rapidly, as long as fluids are available, because chemical potential gradients are maximised between the matrix and grain interiors. In addition, the presence of sub-grains markedly increases the reactive surface area thus enhancing solution-precipitation processes. Metamorphic garnets commonly preserve strong compositional growth zoning that at higher temperatures should begin to homogenise. Such homogenisation of zoned garnet is usually assumed to occur in a spherical grain with no directional variation in diffusivity. Such an assumption is invalidated if grain boundaries, fractures or interaction with inclusions allow preferential communication between garnet interior and exterior and thus faster diffusional relaxation (*cf.* Whitney 1991; Hames & Menard, 1993; Florence & Spear 1991). Some inclusions or fractures are easily detected by optical or electron microscopy, but sub-grain boundaries or sub-microscopic lattice defects (*e.g.* Hwang *et al.*, 2003), especially in cubic minerals such as garnet, are significantly harder to detect but may be large enough to enable considerable grain boundary diffusion. Several mechanisms for sub-grain development in low temperature garnets have been proposed. These include: amalgamation of separately formed nuclei (Spiess *et al.*, 2001; Dobbs *et al.*, 2003; Okamoto & Michibayashi, 2006); brittle fracturing (Hames & Menard, 1993; Whitney, 1996; Prior 1993), and also

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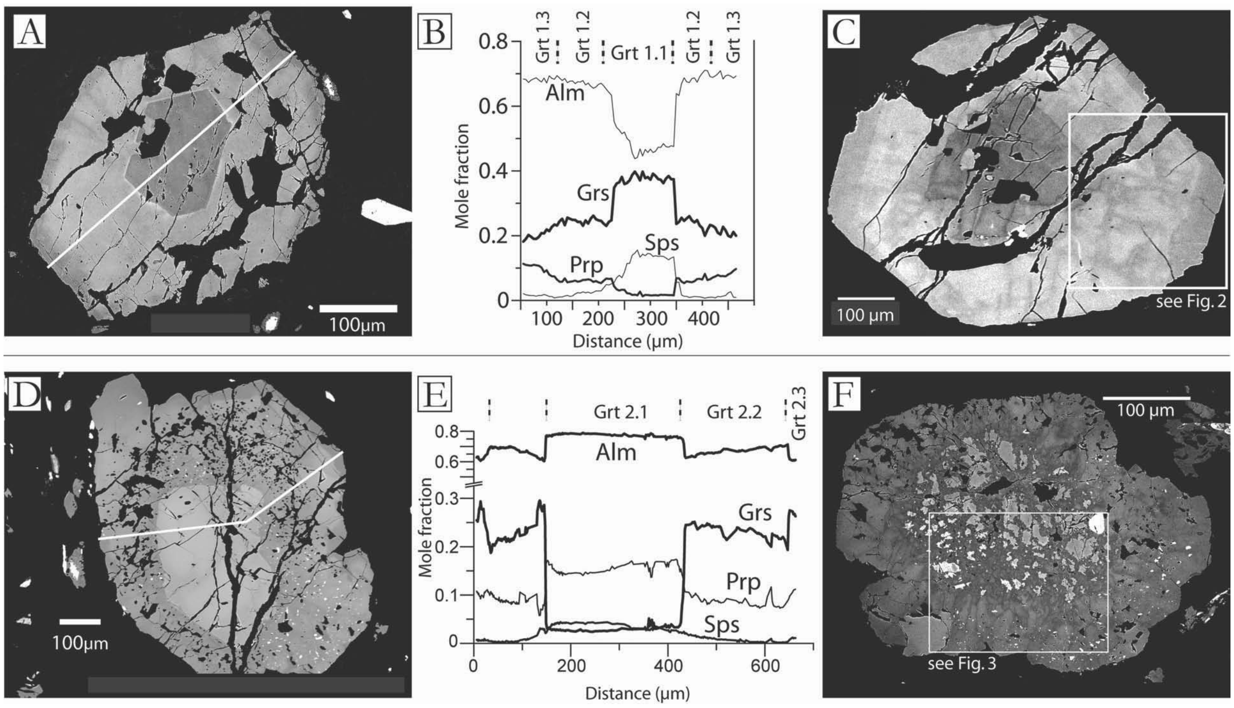

Fig. 1. Backscatter electron (BSE) images and compositional profiles of Type 1 (upper panel) and Type 2 (lower panel) garnet from meta-granitoid samples from the Sesia Zone (Western Alps). A and B: Type 1 garnet unaffected by diffusional modification has almandine-grossular-rich cores (dark in BSE image, A) and almandine-rich rims (light in BSE image) with increasing pyrope and decreasing grossular content. C: BSE image of Type 1 garnet affected by diffusional modification indicated by a complex network of "darker" garnet material in the "lighter" rim (white rectangle). D and E: BSE image (D) and compositional profile (E) of unmodified Type 2 garnet with almandine-rich, grossular-poor cores ("bright") and almandine-grossular-rich rims with increasing pyrope content. F: BSE image of a modified Type 2 garnet with radially oriented diffusion pathways ("dark" network) connecting the outer rim with the inner core.

diffusion-induced grain boundary migration (*e.g.* Hwang *et al.*, 2003), or even dislocation creep (*e.g.* Prior *et al.*, 2000; Storey & Prior, 2005) at high metamorphic temperatures. However, the influence of these sub-textures on the diffusional re-equilibration of elements during metamorphism has not yet been investigated and is the focus of this study.

We investigated high-pressure garnets with electron microprobe and electron backscatter diffraction and found that many garnets consist of a large number of sub-grains with irregular boundaries as well as different grain sizes and shapes. In order to test whether this sub-grain network has enhanced element mobility and compositional relaxation we also undertook detailed compositional mapping of the same garnets. The effects on resetting of zoning patterns and consequences for geothermobarometry and geochronology will be discussed.

Results

Investigated samples

The investigated samples are meta-granitoid high pressure rocks from two different outcrops from the western part of the central Sesia Zone (see Konrad-Schmolke *et al.*, 2006), Western Alps: an area famous for its well-preserved Alpine eclogite facies mineral assemblages in continentally derived rocks (*e.g.* Dal Piaz *et al.*, 1978). Metamorphic peak conditions of 500–600 °C, 1.5–2.0 GPa are deduced for the partly-preserved HP assemblage rutile+garnet+epidote/zoisite+sodic amphibole+omphacite+phengite+quartz (Koons 1986; Zucali *et al.*, 2002). Sodic amphibole and omphacite are often strongly retrogressed to chlorite, albite +/−paragonite and epidote. Garnet forms clasts in a well-developed, blueschist- to greenschist-facies, retrograde foliation defined by phengite and/or sodic amphibole and/or chlorite, and is strongly zoned with respect to major and trace elements. In back-scattered electron (BSE) images (Fig. 1) some grains show domains with well-preserved concentric zoning whereas other parts of the same grains show an irregular, mesh-like zonation pattern.

Generally, garnet in our samples shows at least three growth zones, indicated by brightness variation in BSE images (*cf.* Konrad-Schmolke *et al.*, 2006) and exhibits two different types of zonation pattern. Type 1 garnets have almandine-grossular-rich cores that appear dark in the BSE image (Grt 1.1), surrounded by a "lighter" mantle (Grt 1.2) with significantly lower Ca and higher Fe, and a "darker" outer rim (Grt 1.3; Fig. 1A, B) where Mg increases at the expense of Ca. Type 2 garnets have "bright" almandine-rich, grossular-poor cores (Grt 2.1), overgrown by an almandine-grossular-rich mantle characterised by decreasing grossular and increasing almandine content (Grt 2.2). In some regions Grt 2.2 is overgrown by a small "darker" rim (Grt 2.3) with higher grossular and pyrope and lower almandine content (Fig. 1D, E). Konrad-Schmolke *et al*., (2006) interpreted both types of zonation patterns to represent prograde growth, with the Type 2 patterns interpreted to result from water undersaturation of the host rock prior to and during prograde metamorphism.

High contrast BSE images reveal that both types of garnets often show internal modifications of the concentric zonation pattern. The right part of the mantle region of the Type 1 garnet in Fig. 1C shows a very patchy zonation, caused by a contrast between irregular "bright" fragments separated by a network of "darker" material, a feature frequently observed in garnets from HP and UHP rocks (*e.g.* Pennacchioni 1996; Austrheim *et al*., 1996; Inui & Toriumi, 2002; Zack *et al*., 2002; Janak *et al*., 2006; Hoschek 2007). The large Type 2 garnet crystal in Fig. 1F has "bright" Grt 2.1 fragments in a "darker" core, which is surrounded by a "lighter" mantle and a "darker" rim. The entire garnet grain is truncated by a complex, "darker", in this case fracture-like radial pattern visible in the BSE image. The internal pattern reaches well into the garnet interior where it forms a dense network of "darker" garnet material.

Correlation of internal garnet texture and chemistry

An orientation contrast (OC) image (Fig. 2A) of the "cloudy" area in the garnet shown in Fig. 1C reveals a complex internal sub-texture. Different shades of grey in the image indicate a difference in crystallographic orientation of neighbouring areas (see *e.g.* Prior *et al*., 1999). The image shows that the outer part of the garnet crystal clearly consists of several crystallographically mis-oriented segments separated by sharp boundaries (arrows). Towards the more internal part of the garnet the crystallographic boundaries between these areas become more diffuse and differences in the mis-orientation of neighbouring segments, although still evident in some areas, become less pronounced. Comparison of OC images with major and trace element compositional maps (Fig. 2B–F respectively) shows that chemical modification of the originally concentric growth zonation correlates with the position of boundaries between crystallographically mis-oriented areas, if these are present (*cf.* arrows in A, B and C). In the upper left part of the crystal the lack of sub-structure boundaries hinders a correlation of the compositional mappings with the sub-structure pattern. Further, the compositional mappings show that the extent of chemical modification is element dependent.

The compositional patterns of Mg and X_{Mg} (=Mg/(Mg+Fe)) match the "dark-light" pattern visible in the BSE image and correlate with the sub-structure pattern in the OC image, if developed. The concentration of Mg and the X_{Mg} value is similar along the entire sub-structure boundary network as well as at the outermost rim of the entire garnet grain (Fig. 2B and C) suggesting similar P-T conditions during the equilibration of the garnet rim and the modification of garnet interior along the sub-structure boundaries. In contrast, Ca, Ti and Y concentrations (Fig. 2D, E and F respectively) clearly still preserve a concentric pattern that is independent of the radially-oriented sub-structure boundary network. This observation suggests decoupling of relaxation of compositional gradients of relatively fast-diffusing elements (such as Mg) from that of slow-diffusing elements (such as Ca, Ti and Y). Further, the preservation of the original concentric zonation pattern demonstrates that the development of the sub-structure pattern postdates initial growth and that diffusional modification of the garnet interior occurs as a result of the presence of the sub-structure boundary network.

The OC image (Fig. 3A) of the large garnet grain shown in Fig. 1F reveals that the garnet crystal shown in Fig. 1F exhibits a different, much more complex internal sub-structure pattern than the previous example: Whereas the core of the garnet crystal consists of a large number of small, mostly roundish sub-grains, the rim is made of larger elongate, sometimes palisade-like sub-grains. The close-up in Fig. 3B shows that the size of sub-grains in the fine-grained areas is between 1 and 10 μm, whereas the palisade-like areas in the rim have diameters of at least 20 to 30, sometimes up to 100 μm. As demonstrated by the electron backscatter diffraction (EBSD) mapping (Fig. 3C), the colour-coded pole plots (Fig. 3D) and the mis-orientation-angle diagram in Fig. 3E, the mis-orientation angle between most sub-grains is less than 5°, indicating the predominance of low angle grain boundaries among most of the crystallographic segments.

Due to the different diffusivities of elements in garnet, the size of the sub-grains is a critical factor with respect to the degree of re-equilibration of metastable interiors, as can be demonstrated with a comparison of the sub-grain pattern with compositional X-ray mappings. The compositional variations of Mg and the X_{Mg} value (Fig. 4A and B) correspond well with the sub-grain boundary pattern in the garnet crystal, *i.e.*, it shows high values at the garnet rim and along the sub-grain boundaries throughout the interior. As a result of the small grain-size of sub-grains and thus high grain-boundary density, the core is almost entirely equilibrated at the same X_{Mg} level as the garnet rims. Compositional maps of Ca (Fig. 4C) and Fe (Fig. 4D) show markedly different patterns. Whereas the Fe pattern roughly corresponds with the grain boundaries of the palisade-like sub-grains in the rim of the large garnet, there is almost no correlation between Ca-content and the sub-grain pattern in this domain. In the core, Fe and Ca exhibit a completely different compositional pattern compared to that of Mg and X_{Mg}. Apart from the numerous Garnet 2.1 fragments Ca and Fe patterns allow recognition of two core regions with slightly higher Ca and lower Fe values (high grossular garnet 2.2), one in the upper left side and one in the lower left side of the map. This suggests the existence of two garnet nuclei that amalgamated during the growth process, a structure that is obviously older than the Mg and X_{Mg} patterns, which equilibrated at or near peak metamorphic conditions. Differences in the re-equilibration between major elements are also evident in the composition of Grt 2.1 fragments. Whereas some of

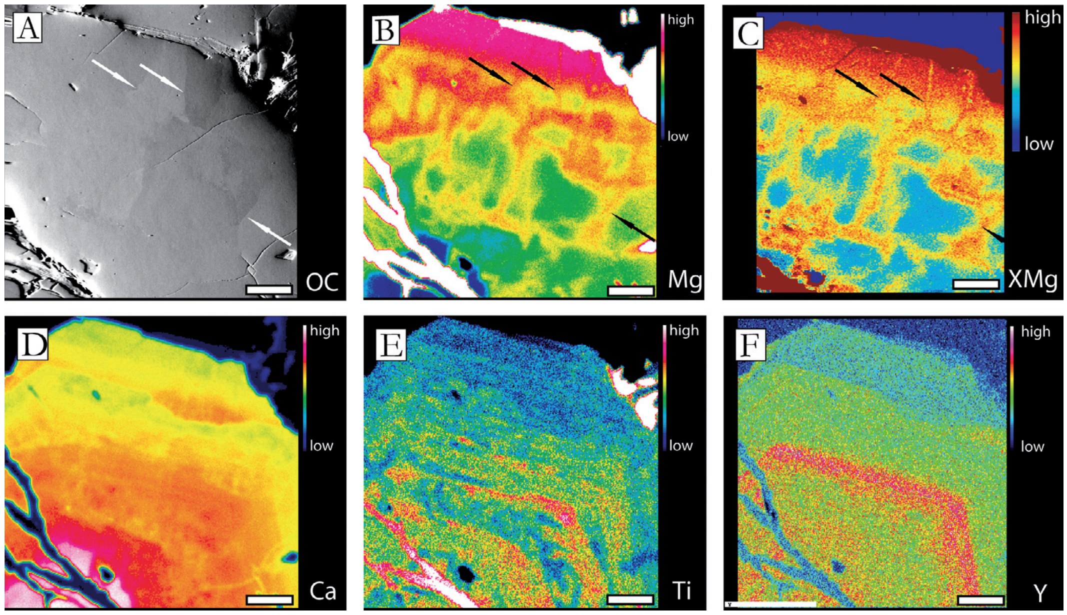

Fig. 2. Orientation contrast (OC) image (A) and compositional mappings (B-F) of the area marked in Fig. 1 C. A: Different grey-shades in the OC image indicate a sub-grain pattern (traced by black lines) in the garnet. The arrows show that the compositional pattern of Mg and X_{Mg} largely correlate with the sub-grain pattern (B, D), whereas Ca, Ti and Y still preserve a concentric zonation (C, E, F).

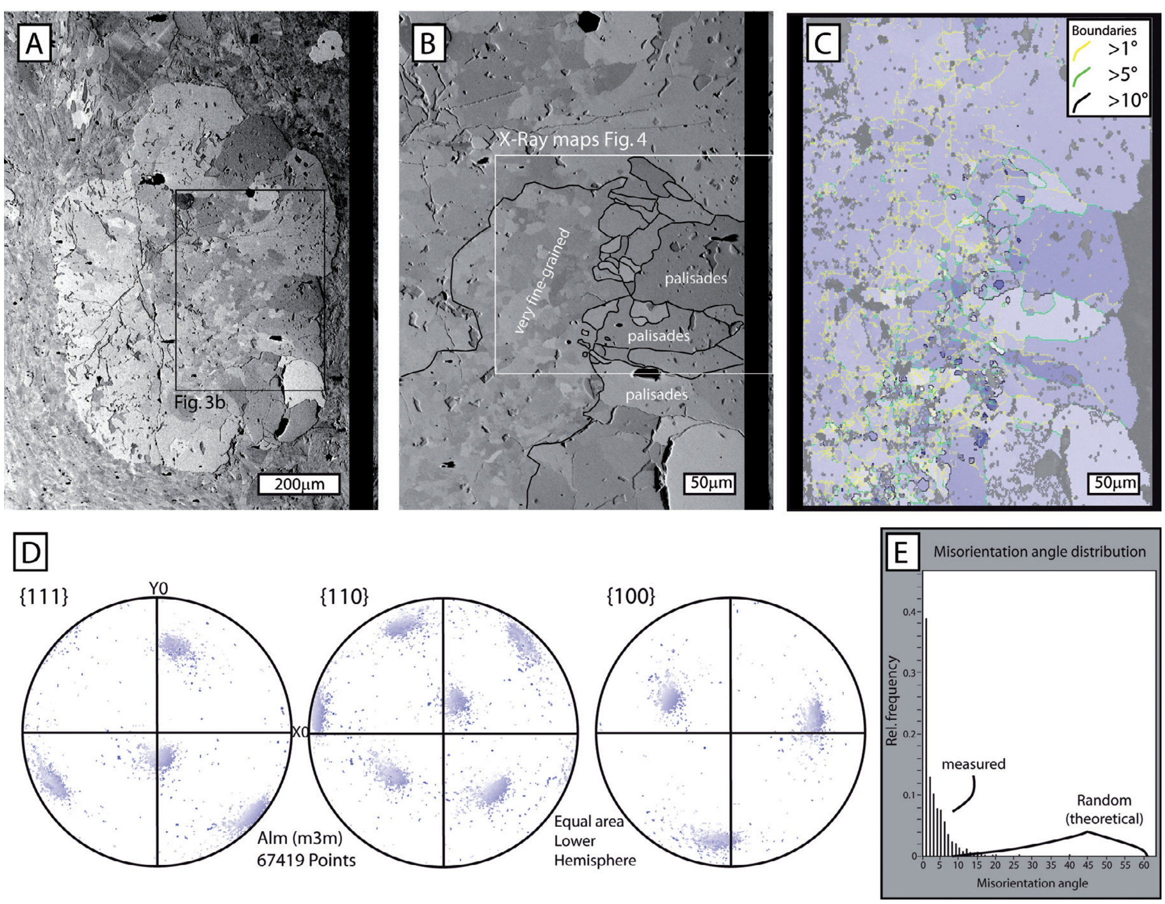

Fig. 3. (A and B) The OC images of the garnet crystal shown in Fig. 1F reveal a complex sub-grain patterns with a very fine-grained (< 10 μm) core and a palisade-like, Coarsegrained (20–150 μm) overgrowth pattern. (C–E) The electron backscatter diffraction (EBSD) mapping (C) of the area shown in (A) as well as the colour-coded pole plots (D) and the mis orientation-angle diagram (E) for that area indicate the predominance of low-angle grain boundaries among most of the crystallographic segments.

the grains (arrow 1 in Fig. 4) are nearly unchanged with respect to their original composition, other, smaller sub-grains show a complete re-equilibration with respect to Mg (although the Ca and Fe content is not exactly the same as at the rim, arrow 2, Fig. 4), which leads to a complex X_{Mg} pattern that cannot be simply interpreted in terms of a P and T evolution.

Interpretation and discussion

The garnet crystals in this study exhibit different sub-grain textures both of which are responsible for internal compositional variations that significantly deviate from originally concentric or near-concentric growth zonations that are partly preserved in the samples. The results derived from Type 1 garnets show that the patchy compositional variations visible in BSE images of many HP and UHP garnets (*e.g.* Matthews *et al*., 1992; Austrheim *et al*., 1996; Zack *et al*., 2002) (Fig. 1C and F) are most likely the result of differential diffusive chemical re-equilibration of pre-existing growth zonations along sub-grain boundaries. Peak X_{Mg} values along grain and sub-grain boundaries in our samples indicate compositional re-equilibration during the prograde evolution or near peak metamorphic conditions. Further, there is a decoupling of chemical resetting between apparently fast diffusing elements, such as Mg, and slow diffusing elements such as Ca, Ti and Y during the re-equilibration process (Fig. 2). The importance of garnet fracturing associated with a chemical modification of garnet interiors during metamorphism has been addressed by various workers (*e.g.* Whitney 1996; Hames & Menard 1993; Hwang *et al*., 2003). It is obvious that diffusive modification of the zoned garnet has important implications for petrologic investigations, such that P-T estimations and postulated P-T trajectories might be misinterpreted (*e.g.* Chakraborty & Ganguly 1991; Florence & Spear 1991). Interestingly, the OC images and the compositional mappings in Fig. 2 show that the sub-grain boundary pattern always coincides with chemical modifications of the growth zonation pattern, but that the pathways along which fast elemental exchange occurs not necessarily cause mis-orientations between neighbouring areas of the garnet crystal. This observation allows an interpretation of the sub-texture development. Obviously, the crystallographic anisotropies enabling directed element exchange allow, but not generally cause, neighbouring crystal segments to slightly rotate with respect to each other. Sub-grain rotation might only be enabled if the anisotropies form a three-dimensionally interconnected network but is hindered if the neighbouring segments are still crystallographically connected. Although several works (*e.g.* Vollbrecht *et al*., 2006) postulate dislocation mobility in terms of viscous deformation in garnet at relatively low temperatures around 650 °C, we doubt a significant contribution of dislocation creep as a mechanism for the development of the sub-grain structure, because of the extremely low peak metamorphic temperatures (500−550 °C) of our samples (*cf.* Zhang & Green 2007). Further, none of the investigated crystals in our sample shows significant elongation resulting from dynamic recrystallisation or cataclastic fragmentation as a response to host rock deformation. Thus, it is questionable whether dislocation creep and recovery (*e.g.* Prior *et al*., 2000), cataclastic deformation (*e.g.* Trepmann & Stöckhert 2002) or grain boundary sliding (*e.g.* Terry & Heidelbach 2004) can be responsible for the sub-texture development. This observation is important because deformation-controlled cation diffusion has been observed in plastically deformed tourmaline (Büttner 2005) and might also occur in garnet. Due to the absence of any signs of crystal-plastic deformation in our samples we favour the assumption that a diffusion induced dislocation migration and/or diffusion induced recrystallisation process (Hwang *et al*., 2003) is responsible for the development of the sub-grain texture. The preferred exchange of cations, as observed in our samples, and a resulting distortion of the crystal lattice might cause dislocation generation and facilitate dislocation migration. But to doubtlessly clarify the nature and development of the crystallograhic anisotropies that enable fast and selective elemental exchange, further investigations utilising transmission electron microscopy are necessary but are beyond the scope of this paper.

Although the sub-grain boundary network in Type 2 garnets has similar effects on the diffusional re-equilibration of garnet interiors, the large textural difference between the internal sub-structures in Type 1 and 2 garnets suggest a different development of the sub-grain patterns in both types of garnet in our samples. The preservation of garnet 2.1 fragments as well as of compositional domains in the Fe and Ca mappings (Fig. 4) allows an interpretation of the development of the complex sub-grain texture in Type 2 garnets (Fig. 5): (1) In an initial stage fracturing and resorption of garnet 2.1 leads to differential preservation of larger and smaller garnet 2.1 fragments with similar crystallographic orientation inherited from the host grain (Fig. 5A). Examples of relatively well preserved but intensely fractured garnet 2.1 fragments are abundant in our samples (*cf.* BSE and OC images Fig. 5A). (2) Prograde metamorphism leads to the formation of a grossular-rich garnet 2.2 that fills the cracks between large garnet 2.1 fragments as well as overgrows smaller garnet 2.1 fragments, from which it inherits the crystallographic orientation (Fig. 5B, black arrows). Additionally, depending on fragment-size, sub-grain boundary density as well as element diffusivity, smaller garnet 2.1 fragments or areas within larger fragments are diffusionally homogenised (grey arrows in Fig. 5B). As a result of the amalgamation of the garnet 2.2 grains, a sub-grain boundary network develops within the large amalgamated grains. This stage is preserved in the compositional pattern of Ca and Fe as shown in Fig. 3 and 5C. (3) During the further prograde evolution garnet growth proceeds with grossular-poorer garnet 2.2, which also inherits the crystallographic orientation of pre-existing grains, but exhibits palisade-like radial growth as a result of the only possible growth direction (arrows in Fig. 5C). (4) During the last stage, but possibly also during all previous stages, selective compositional re-equilibration occurs along the sub-grain boundaries and progressively modifies garnet interiors with respect to Mg content and X_{Mg} value (Fig. 5D). Interest-

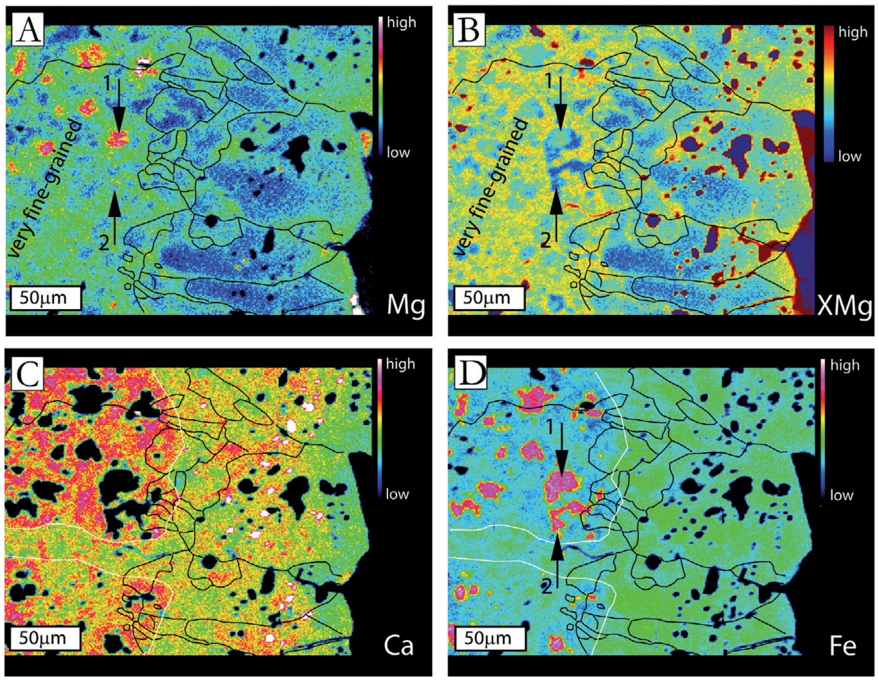

Fig. 4. Compositional maps of the same area as shown in Fig. 3B show that Mg (A) and X_{Mg} (B) have highest values along sub-grain boundaries as well as in the fine-grained core and correspond well with the sub-grain pattern. In contrast, Ca (C) and Fe (D) allow recognition of older compositional pattern in the core (white framed regions). Differential re-equilibration between major elements (arrows) leads to a complex X_{Mg} pattern that cannot be interpreted in terms of a P-T path (see text for further explanation).

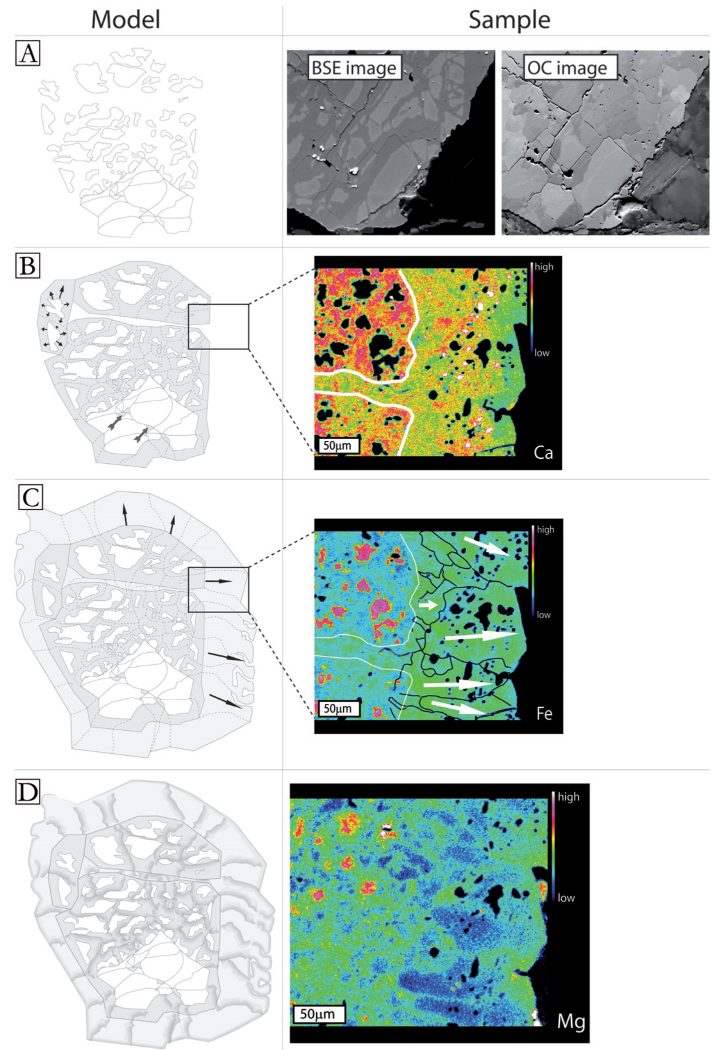

Fig. 5. Sub-texture development of Type 2 garnets. A) Fragmentation and resorption of garnet 2.1 grains without significant rotation of fragments. The comparison of BSE and OC images indicates that each subgrain contains a garnet 2.1 fragment in the core (see text). B) Overgrowth of Ca-rich garnet 2.2 inheriting the crystallographic orientation of garnet 2.1 fragments. The overgrowth stage is preserved in the Ca-rich amalgamated cores (X-ray mapping). C) Radial overgrowth of Ca-poorer, Fe-richer garnet 2.2 that forms palisade-like sub-grains. D) Differential compositional re-equilibration of all garnet generations along sub-grain boundaries. This stage is best preserved in the Mg and X_{Mg} mappings (see also Fig. 4).

ingly, the irregular overgrowth of Grt 2.3, as observed along parts of the rim in the sample shown in Fig. 1D, is not observed in the highly re-equilibrated grain in Fig. 1F. A possible reason for this lack might be fractionation effects during prograde garnet growth, such that growth of Grt 2.3 is thermodynamically hindered in those samples with an intense element exchange between core and matrix due to small scale variations in the effective bulk rock composition around the garnet crystal (*cf.* Konrad-Schmolke *et al.*, 2005). Thus, clarifying the influence of element recycling using thermodynamic forward modelling with the consideration of element fractionation (*e.g.* Spear & Selverstone 1983; Menard & Spear 1993; Connolly 2005; Konrad-Schmolke *et al.*, 2006) should yield further important insight into garnet growth and composition in such partly re-equilibrated phase assemblages.

Conclusions

The examples in this work show that in contrast to the assumption of a spherical geometry for garnet, the presence of a complex, sub-grain texture may allow significant core-rim differences in diffusional re-equilibration. The result is a much faster homogenisation of cores and thus, if incorrectly modelled, a much longer timescale for peak metamorphism than actually experienced. Our samples indicate that the extent of diffusional resetting is clearly dependent on the size of sub-grains, which might have severe consequences for the interpretation of diffusional relaxation timescales for garnets with a critical sub-grain size. In our samples the size of the sub-grains in the garnet interior is small enough (< 20 μm) to enable almost complete resetting with respect to X_{Mg} whereas larger sub-grains still preserve lower X_{Mg} values.

Furthermore, chemical re-equilibration of existing garnets may influence the growth of new garnet in two ways. On the one hand, garnet growth might be enhanced by recycling of manganese from existing grains into the matrix. On the other hand, metastable cores of older garnets might be completely replaced by re-equilibration, thus leading to a situation where the garnet core is younger than the rim. An example of this phenomenon is the growth of newly formed garnet in the lagoons of atoll-like garnets (*e.g.* Godard *et al.*, 1981). In addition, differential resetting could cause major problems in the interpretation of isotopic ages of garnet as trace elements yielding age information will become decoupled from major element patterns used for geothermobarometry (*e.g.* Hermann & Rubatto 2003). The procedure for the determination of orientation contrast in natural garnet-bearing samples is now well developed and could become a standard technique in all studies of relaxation of compositional zoning where different diffusion mechanisms may be involved in different parts of the grains.

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