

On the origin of oriented rutile needles in garnet from UHP eclogites

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ABSTRACT Although oriented rutile needles in garnet have been reported from several ultrahigh-pressure (UHP) rocks and considered to be important UHP indicators, their crystallographic features including growth habit and lattice correspondences with garnet host have never been properly characterized. This paper presents a detailed analytical electron microscopic (AEM) study on evenly distributed oriented rutile needles in garnet of two eclogitic rocks from Sulu. Some garnet in one UHP diamondiferous quartzofeldspathic rock from the Saxonian Erzgebirge, and in one high-pressure (HP) felsic granulite from Bohemia also contain a few unevenly distributed oriented rutile needles. They have also been studied for the purpose of comparison. Despite different distribution patterns, AEM revealed that all rutile needles are oriented along the $\langle 111 \rangle$ directions of garnet with their lateral sides surrounded by the $\{110\}$ planes of garnet, and that the growth directions of most needles are close to the normal of the $\{101\}$ planes of rutile. No other specific crystallographic orientation relationships between rutile and garnet host were observed, and there is no pyroxene associated with rutile, as necessitated by the precipitation reaction of rutile in garnet as previously proposed. A simple solid-state precipitation scenario for the formation of the rutile needles in garnet in these two eclogitic rocks is not justified. Three alternative mechanisms are considered for the formation of oriented rutile needles: (i) the rutile needles may be inherited from precursor minerals; (ii) the rutile needles may be formed by a dissolution–reprecipitation mechanism; and (iii) the rutile needles may be formed by cleaving and healing of garnet with rutile deposition. None of these mechanisms can fully explain the observations, although the first one is less likely and the third one is preferred. This study presents an example where the presence of oriented/aligned inclusions in minerals does not necessarily imply a precipitation origin.

Key words: analytical electron microscopy; garnet; oriented precipitates; precipitation; rutile inclusion; ultrahigh-pressure metamorphism.

INTRODUCTION

Oriented rutile needles in garnet within garnet peridotite (Van Roermund *et al.*, 2000; Song *et al.*, 2004), kimberlitic/ultrahigh-pressure (UHP) eclogite (Griffin *et al.*, 1971; Hills & Haggerty, 1989; Zhang & Liou, 1999; Ye *et al.*, 2000; Zhang *et al.*, 2005) and UHP garnet–biotite–kyanite gneiss (Mposkos & Kostopoulos, 2001) were presumed to have a precipitation origin. The more common term precipitation rather than exsolution (Zhang & Liou, 1999) is used here for this type of diffusional phase transformation in the solid state. As these rocks either are of mantle origin or have experienced UHP metamorphism, and Ti solubility in garnet decreases with decreasing pressure and temperature (Ringwood & Lovering, 1970; Zhang *et al.*, 2003), solid-state precipitation upon rock decompression and/or cooling appears to be a reasonable cause

for the formation of these rutile needles (e.g. Haggerty, 1991; Zhang & Liou, 1999; Ye *et al.*, 2000; Zhang *et al.*, 2003, 2005), and the amount of rutile would be a potential pressure/temperature indicator.

Although solid-state precipitation generally gives rise to oriented/aligned inclusion microstructures (Johnson *et al.*, 1975; Putnis, 1992), the presence of oriented/aligned inclusions in minerals does not necessarily imply a precipitation origin, especially when the inclusions bear no specific crystallographic orientations to their host. A solid-state precipitation process always involves epitaxial nucleation for a lower activation energy and minimization of the interfacial energy and strain energy (Johnson *et al.*, 1975; Putnis, 1992; Putnis & Putnis, 2006). Formation of precipitate twin variants and their ordering transformation upon cooling, such as the case of feldspar with its low crystal symmetry and complicated compositions and

microstructures (Putnis, 1992), may cause distortion and hence deviation of the original crystallographic relationships. However, this explanation is not relevant to the present case of rutile needles in garnet.

One notorious example is oriented quartz rods in amphibolite facies garnet, which have been suggested to be the symplectic growth features behind the planar {110} growth fronts of garnet (Burton, 1986). Another example is the aligned multiple-phase inclusion pockets/lamellae in diopside/clinopyroxene from diamondiferous garnet–clinopyroxene rocks (Hwang *et al.*, 2004) and marble (Chu *et al.*, 2004) from the Kokchetav Massif; these well-aligned K-bearing inclusion pockets/lamellae consist of kokchetavite, phengite, cristobalite and an amorphous phase, and were formed from the intruding melts through partings/cleavages of pyroxene. In the light of these two examples, it is clear that any conclusion/inference on precipitation origin of the oriented inclusions in garnet or other minerals based solely on their aligned/oriented inclusion microstructure on the scale of optical microscopy needs additional verification using high-resolution methods.

To investigate further, detailed analytical electron microscopic (AEM) observations were made on evenly distributed oriented rutile needles, which have been considered as the precipitated phase upon decompression, in garnet of two UHP eclogitic rocks from Yangkou in the Sulu terrane (Zhang & Liou, 1999; Ye *et al.*, 2000; Zhang *et al.*, 2003, 2005). For comparison of the growth characteristics, the unevenly distributed oriented rutile needles in garnet of one UHP diamondiferous quartzofeldspathic rock from the Erzgebirge Crystalline Complex and one HP felsic granulite from the southern Bohemian Massif were also analysed.

SAMPLE DESCRIPTIONS AND ANALYTICAL METHODS

The eclogitic samples 96Y5A and 96Y5B were collected from the Yangkou layered mafic–ultramafic complex, Sulu UHP terrane and have been petrologically investigated by Zhang *et al.* (2005). The eclogite is foliated and mainly consists of coarse-grained garnet porphyroblasts (30–40 vol.%; Prp_{29–41}Gr_{19–36}Alm_{33–45}Sps₁), in a matrix of omphacite/clinopyroxene, fine-grained garnet and rutile (Fig. 1a). The porphyroblastic garnet is weakly zoned with decreasing almandine and increasing grossular component from core to rim. The TiO₂ content of garnet ranges from 0.01 to 0.11 wt%, if the rutile needles are not taken into consideration. Most garnet grains show a high Ti core and a low Ti rim, but the reverse was also occasionally observed (Zhang *et al.*, 2005). The matrix garnet is chemically similar to the rim of porphyroblastic garnet (Ye *et al.*, 2000; Zhang *et al.*, 2005). Coesite reportedly occurs not only as inclusions in garnet and omphacite, but also as an intergranular phase in the matrix in nearby eclogite transitional to

metagabbro (Liou & Zhang, 1996). The metamorphic peak *P–T* condition for eclogitic samples 96Y5A and 96Y5B was estimated to be 825–880 °C and 42–45 kbar (Zhang *et al.*, 2005), although Ye *et al.* (2000) reported the presence of majoritic garnet in Yangkou eclogite which implies a recrystallization depth of more than 200 km. Abundant evenly distributed oriented rutile needles and minor apatite inclusions were observed in porphyroblastic garnet cores (Fig. 1b), but not in most porphyroblastic garnet rims and fine-grained matrix garnet.

The diamondiferous quartzofeldspathic rock sample 25966 was collected near the eastern shore of the Saldenbach reservoir about 1.5 km NE of the village of Forchheim in Erzgebirge, Germany. The sample is composed of coarse-grained garnet (Prp_{28–39}Gr_{8–10}Alm_{53–65}) crystals in a matrix of quartz, phengite, biotite, kyanite, albitic plagioclase, K-feldspar, zircon, rutile and graphite (Fig. 1c). Abundant microdiamond was found as inclusions in garnet in this sample (Hwang *et al.*, 2006). The metamorphic peak *P–T* condition was estimated to be ~1200 °C and > 70 kbar (Massonne, 2003). Oriented rutile needles, which have never been observed/reported in other Erzgebirge diamondiferous samples (Massonne, 1999; Hwang *et al.*, 2000, 2001a) are unevenly distributed in some, but not all, garnet crystals in sample 25966 (Fig. 1d). When rutile needles are present in garnet in this sample, they may occur in the core or the rim and microdiamond inclusions are sometimes observed within the area covered by these needles.

The felsic granulite sample U3 was collected from the Blansky les massif of the Gföhl Unit, the tectonically uppermost of a series of high-grade nappe units of the southern Bohemian Massif. The sample is strongly foliated and light-coloured. Garnet (Prp₂₀Gr₁₀Alm₇₀), quartz, plagioclase, K-feldspar, biotite, spinel, apatite, rutile, ilmenite and zircon are the major constituents (Fig. 1e). The metamorphic peak *P–T* condition was estimated to be > 900 °C and > 15 kbar (e.g. Kröner *et al.*, 2000; O'Brien & Rotzler, 2003). Besides the common feldspar, kyanite and quartz inclusions, a few unevenly distributed oriented rutile needles were also observed in almost every garnet crystal (Fig. 1f).

Thin sections showing garnet grains with oriented needle-like inclusions under the optical microscope (OM) were first clamped between two copper rings to ensure sample integrity, followed by argon-ion-beam milling (Gatan, PIS) to perforation (operation condition: 4.0 kV, 9° incident angle). A total of 18 AEM discs containing acicular rutile at the thinned edge of garnet grains were analysed in the present study. These discs were prepared from the following samples: AG1–AG5 from sample 96Y5A, BG1–BG5 from sample 96Y5B, GR1–GR4 from sample 25966 and UG1–UG4 from sample U3. The specimens were coated with carbon after ion thinning. Microstructure and composition of minerals were studied using an AEM

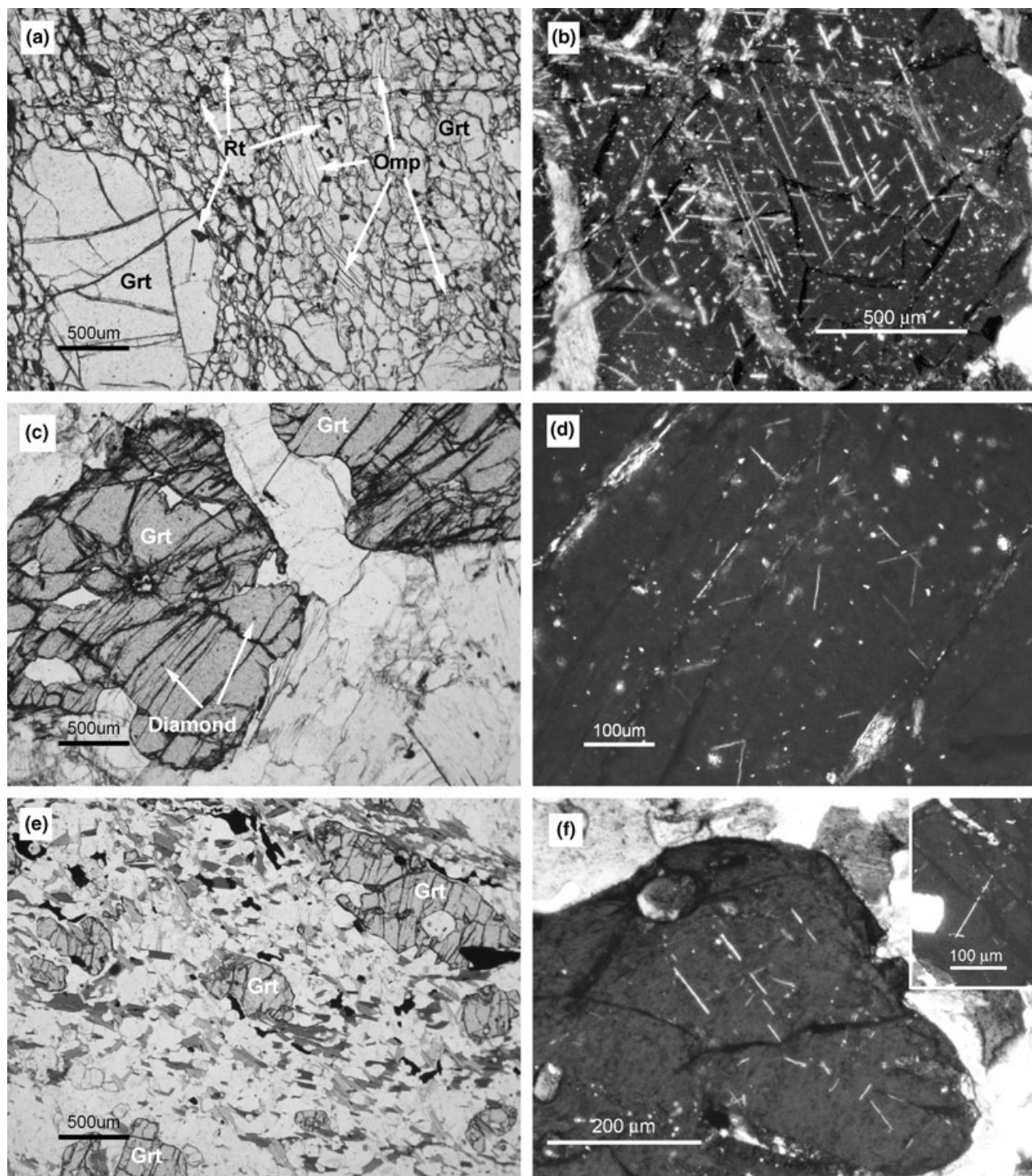


Fig. 1. OM micrographs showing the microstructures of the rocks studied and the oriented rutile needles in garnet from samples (a, b) 96Y5A/5B, (c, d) 25966 and (e, f) U3. The insert in (f) shows a garnet grain with only one long rutile needle across the grain. (a, c, e) Plane polarized light; (b, d, f) crossed polarized light.

(JEOL JEM-3010 operated at 300 kV) equipped with an energy dispersive X-ray (EDX) spectrometer (Oxford EDS-6636) with an ultra-thin window and a Si(Li)

detector, capable of detecting elements from boron to uranium. EDX spectra of inclusions were collected for 100 s.

RESULTS

UHP eclogites from Sulu area (samples 96Y5A and 96Y5B)

OM micrograph shows that there are abundant rutile needles up to 500 μm in length in the core, but not the rim, of porphyroblastic garnet of samples 96Y5A and 96Y5B (Fig. 1b). Within each porphyroblastic garnet grain, there are usually four non-intersecting arrays of parallel needles of rutile, three of them forming equilateral triangle patterns when viewed down the long-axis of the fourth array (Fig. 1b). Although the needles which belong to the same array are parallel to each other, they have oblique extinction with different extinction angles under crossed polars, implying that the lattices of the rutile needles may not have a consistent orientation relative to the garnet host. AEM indicated the traces of rutile needles are parallel to $\langle 111 \rangle / \langle 100 \rangle$, $\langle 112 \rangle$ and $\langle 110 \rangle$ directions of garnet when viewed along $\langle 110 \rangle$ (Fig. 2a,b), $\langle 111 \rangle$ (Fig. 2c) and $\langle 100 \rangle$ (Fig. 2d) zone axes of garnet, respectively. These observations unambiguously require that the long axes of needles are all parallel to the $\langle 111 \rangle$ directions of the garnet host, forming four arrays of needles as observed by OM. The rutile needles have prismatic cross-sections (Fig. 2e) with the sides bounded by the lowest energy $\{110\}$ planes of garnet (Fig. 2b–e).

The pronounced needle-like morphology of rutile also suggests that they could have grown along a specific lattice direction of rutile, i.e. the long axis of rutile needle could be parallel to a specific lattice direction of rutile. For this reason, seven needles from samples 96Y5A/96Y5B were analysed in detail in order to define their growth directions. Five needles are within $\sim 7^\circ$ off from the normal of $\{101\}_{\text{rt}}$ (cf. Fig. 2f for example), one needle is $\sim 23^\circ$ off from the normal of $\{101\}_{\text{rt}}$ and is nearly parallel to the $\langle 101 \rangle_{\text{rt}}$, the other is $\sim 61^\circ$ off from the normal of $\{101\}_{\text{rt}}$ and is nearly parallel to the normal of $(210)_{\text{rt}}$. The preferred $\{101\}$ -normal growth explains the oblique extinction of the rutile needles in OM observations. Note that the oblique extinction of oriented rutile inclusions in garnet was previously taken as a probable indication of the presence of high-pressure polymorphs of titania in other UHP rocks (Song *et al.*, 2004). By plotting the long axes of all needles along the $[\bar{1}11]$ direction of garnet, stereographic projections of the rutile a- and c-axes show their random orientation relative to the garnet host (Fig. 3). In other words, except for the gross alignment between the $\langle 111 \rangle_{\text{grt}}$ and $\{101\}$ -normal of rutile, there are no specific crystallographic orientation relationships between garnet and rutile.

Most interestingly, at the garnet/rutile phase boundaries, ilmenite islands of less than 100 nm thickness were commonly observed by AEM (Figs 2e & 4a,b,d). These ilmenite islands bear no crystallographic orientation relationships to the adjacent rutile and garnet. Besides ilmenite islands, a continuous

amorphous film of 1–2 nm thickness along the garnet/rutile phase boundaries was also revealed by AEM (Fig. 4b–c). EDX analysis of these films was difficult because of strong interference from the adjacent rutile and garnet. However, by comparing the EDX spectra collected (using 5 nm spots), respectively, over the garnet matrix and the film (Fig. 5a,b, respectively), it was found that the film is compositionally characterized by higher Ca and lower Mg content than that found in the garnet matrix. Interfacial films with high Al/Cl content (Fig. 5c) and occasional (Fe, Co, Ni)-arsenide crystals (Figs 4d & 5d) were also detected in sample 96Y5B. Nanometer-size pockets also were commonly observed on surfaces of rutile needles (Fig. 4e–f). These pockets are bounded by low index facets such as $\{100\}_{\text{rt}}$ and $\{110\}_{\text{rt}}$ and were always brighter than rutile in bright-field images, indicating that they are either empty or contain elements lighter than titanium. Because of strong interference from rutile, it was not possible to obtain composition signatures of these pockets by EDX.

In addition to the needle-like rutile crystals, globular rutile and oriented apatite (1–2 μm in size) were also observed as inclusions in porphyroblastic garnet of samples 96Y5A/96Y5B. Chlorite was commonly found to be associated with globular rutile crystals. AEM–EDX revealed that oriented apatite crystals are enriched in Cl with Cl/P = 0.27–0.29 (i.e. chlorapatite) and contain traces of Sr (Fig. 5e). Some apatite crystals are bounded by $\{110\}$ planes of the garnet host following the specific crystallographic relationships of $[111]_{\text{grt}}/[0001]_{\text{ap}}$; $(\bar{1}10)_{\text{grt}}/(1010)_{\text{ap}}$. However, oriented apatite crystals in random orientations with respect to the garnet host were also observed. Oriented pyroxene (+ minor rutile and apatite) inclusions in garnet as reported from another Yangkou eclogite and interpreted as majoritic garnet (Ye *et al.*, 2000) were not observed in the present samples.

Diamondiferous quartzofeldspathic rock from Erzgebirge (sample 25966) and felsic granulite from Bohemia (sample U3)

Although lower in abundance and finer in grain size than those in samples 96Y5A/96Y5B, oriented rutile needles were also found within garnet crystals in a diamondiferous gneissic rock from Erzgebirge (Fig. 1d) and a felsic granulite from Bohemia (Fig. 1f). The distributions of rutile needles in these two samples are highly uneven. Some of the garnet does not contain any needles (in 25966), or only one or two long needles extending halfway across the garnet grains (in U3). These observations suggest that the oriented needles in these occurrences are less likely to have a precipitation origin. On the other hand, similar to samples 96Y5A/96Y5B from Sulu, the rutile needles in samples 25966 and U3 are parallel to the $\langle 111 \rangle$ directions and bounded by the $\{110\}$ planes of the garnet host, i.e. the lateral sides of the needles are surrounded by $\{110\}_{\text{grt}}$

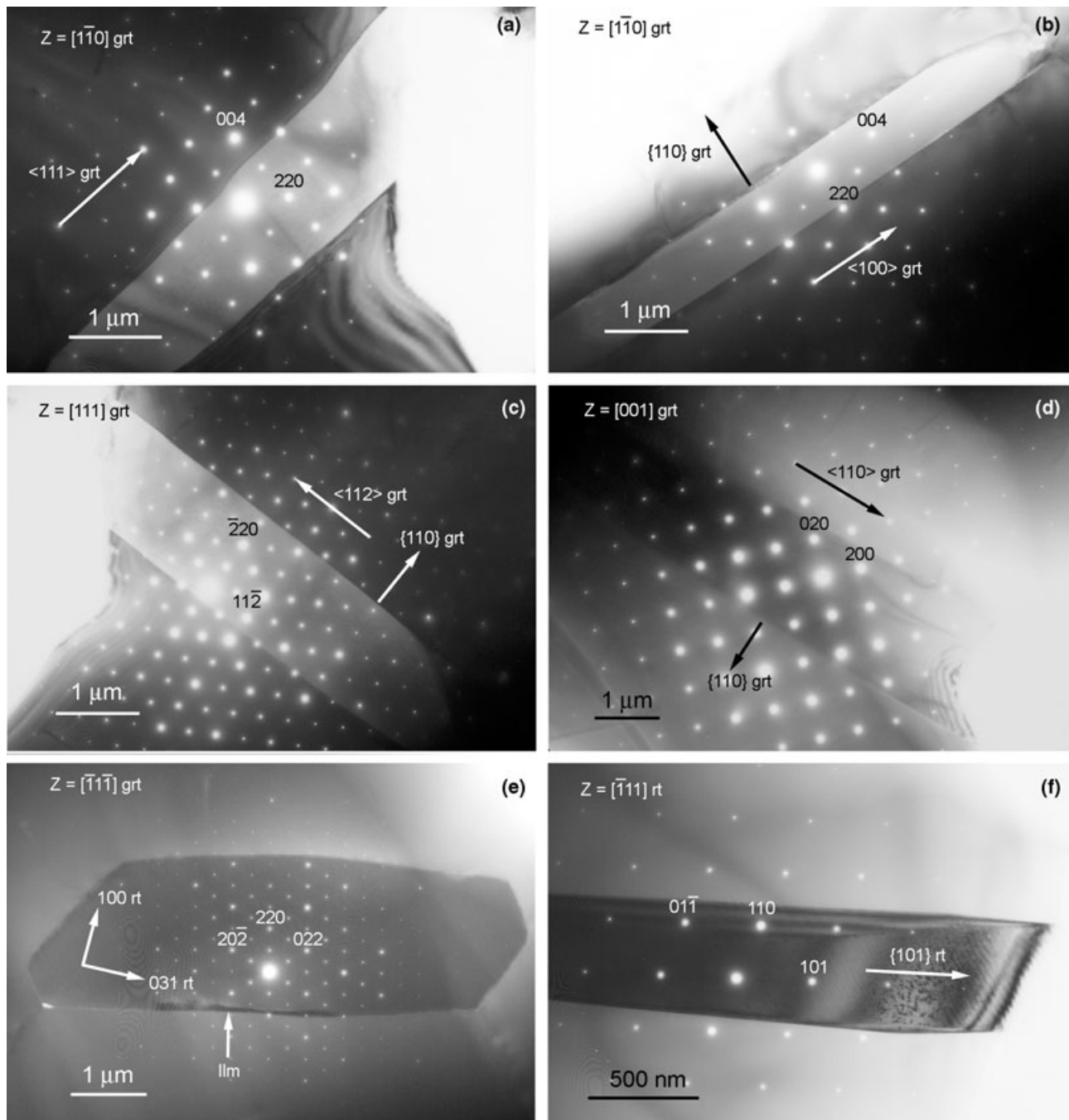


Fig. 2. AEM micrographs with superimposed selected area electron diffraction (SAED) patterns from samples 96Y5A/5B showing (a–d) the trace of rutile needle parallel to $\langle 111 \rangle_{\text{grt}}$ or $\langle 100 \rangle_{\text{grt}}$ when viewed along $\langle 110 \rangle_{\text{grt}}$, $\langle 112 \rangle_{\text{grt}}$ when viewed along $\langle 111 \rangle_{\text{grt}}$, and $\langle 110 \rangle_{\text{grt}}$ when viewed along $\langle 100 \rangle_{\text{grt}}$, (e) the prismatic cross-section of rutile needle viewed along $\langle 111 \rangle_{\text{grt}}/\{101\}\text{-normal}_{\text{rt}}$, and (f) the $\{101\}\text{-normal}_{\text{rt}}$ growth direction of rutile needle. Also shown in (e) are the directions of 100_{rt} and 031_{rt} reciprocal vectors in $[013]_{\text{rt}}$ zone axis ($\sim 6^\circ$ from $\{101\}\text{-normal}_{\text{rt}}$). Ilmenite in (e) is marked by arrow. The zone-axis of the superimposed SAED pattern is given at the top left corner of each micrograph.

faces, as indicated by AEM (Fig. 6a–c). Likewise, the preferred growth directions of the needles are also close to the normal of the $\{101\}$ planes of rutile (Fig. 6d). Besides the gross parallelism between the $\langle 111 \rangle_{\text{grt}}$ and $\{101\}\text{-normal}$ of rutile, no further specific

crystallographic orientation relationships were identified between needles and their garnet host. Epitaxial crystalline phases with R_3O_4 spinel ((Mg, Fe, Zn) Al_2O_4) and RO rock-salt (FeO) structures were observed at the tip of a rutile needle in sample U3

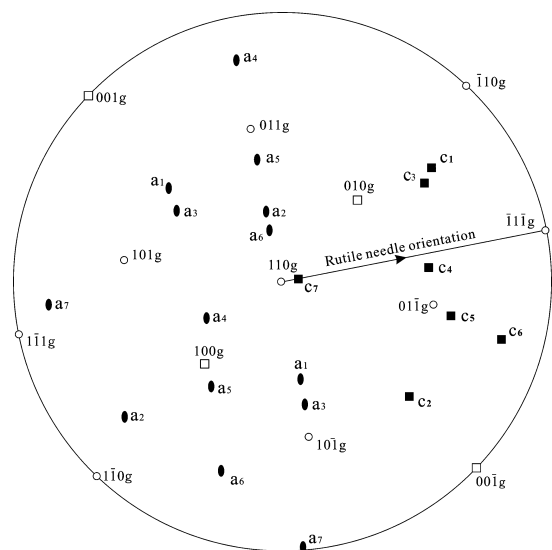


Fig. 3. Stereographic projections of the a- (solid ellipse) and c-axes (solid square) of seven rutile needles oriented along $[111]_{\text{grt}}$ showing random orientations of rutile needles relative to garnet host in samples 96Y5A/5B. Some garnet poles (open circle and square) are also included. Needle orientation: a_2/c_2 , $\langle 101 \rangle_{\text{rt}}$; a_7/c_7 , $\{210\}$ -normal $_{\text{rt}}$; a_1/c_1 , a_3/c_3 – a_6/c_6 , $\{101\}$ -normal $_{\text{rt}}$.

(Fig. 7a). Along the garnet-rutile phase boundaries in sample U3, high-resolution imaging and AEM–EDX analysis further revealed the presence of a continuous Al-rich amorphous film of thickness less than 1 nm (Fig. 7b). A similar amorphous film with slightly higher Al and Ca and lower Mg than garnet was also identified at the garnet/rutile phase boundaries in sample 25966 (Fig. 7c).

DISCUSSION

Are the oriented rutile needles in garnet of Yangkou eclogitic rocks products of solid-state precipitation?

The detailed OM and AEM observations on the oriented rutile needles in garnet of Yangkou UHP eclogitic rocks in the present study show the following general features:

1. Oriented rutile needles are evenly distributed in the cores of garnet porphyroblasts; garnet rims usually contain less or no rutile needles.
2. Rutile needles are oriented along the $\langle 111 \rangle$ directions of garnet and bounded by the lowest energy $\{110\}$ planes of garnet at the sides.
3. Most rutile needles possess growth directions close to the $\{101\}$ -normal of rutile (0 – 7° off). Some deviate by several degrees from the above direction. Besides the gross alignment between the $\{101\}$ -normal of rutile and the $\langle 111 \rangle$ directions of garnet, there are no specific crystallographic orientation relationships between rutile needles and the garnet host.
4. An amorphous film differing in composition from the garnet host and many ilmenite islands are present

between rutile needles and the garnet host. Facetted pockets are present on surfaces of rutile needles.

5. No pyroxene phase(s)/lamellae were observed in garnet.

Except for the first observation, the above features, especially the lack of specific crystallographic orientation relationships between rutile and garnet host, as well as the absence of pyroxene lamellae in garnet strongly argue against a simple solid-state (or sub-solidus) precipitation model for the oriented rutile needles within garnet of Yangkou UHP eclogitic rocks.

The lack of specific orientation relationships between rutile and garnet host

Oriented rutile needles in star sapphire and sagenitic biotite are well-known analogues to the rutile needles in garnet in the present study and were studied in detail by TEM. The rutile needles in sapphire lie on the close-packed basal plane of sapphire and are elongated along $\langle 101 \rangle_{\text{rt}}$, following the orientation relationship $\langle 011 \rangle_{\text{rt}} // \langle 10\bar{1}0 \rangle_{\text{s}}$, $(100)_{\text{rt}} // (0001)_{\text{s}}$ (Phillips *et al.*, 1980). Such a preferred orientation and growth morphology of rutile represent good matching in close-packed oxygen-sublattices across the rutile/sapphire interfaces as well as the least misfit strain along the needle axis. Analogously, the rutile needles in sagenitic biotite are parallel to the close-packed (001) plane of biotite, and are elongated along $[001]_{\text{rt}}$. The orientation relationship between rutile and biotite was found to be $(100)_{\text{rt}} // (001)_{\text{bt}}$, $[001]_{\text{rt}} // [010]_{\text{bt}}$ (Shau *et al.*, 1991), which is in accordance with their mutually parallel close-packed oxygen planes and chains of edge-sharing octahedra.

In contrast to sapphire and biotite, garnet has more complicated oxygen-sublattice, which does not resemble either the hexagonal or the cubic close-packed structures. This renders the prediction on the preferred growth morphology and orientation of rutile precipitates in garnet difficult. However, such growth morphology/orientation representing low interfacial energies between rutile inclusions and garnet host could be obtained from the study on the submicroscopic rutile inclusions in garnet of high-grade metamorphic rocks, which presumably adjusted their orientations with respect to garnet host easily under high temperatures. In our AEM study of submicroscopic inclusions in almandine-rich garnet from the white-schist of Kulet area, Kokchetav massif, two interesting topotactic relationships between equiaxed rutile inclusions and garnet host were identified either $[001]_{\text{rt}} // [001]_{\text{grt}}$ and $\{100\}_{\text{rt}} // \{110\}_{\text{grt}}$, or $\langle 101 \rangle_{\text{rt}} // \langle 111 \rangle_{\text{grt}}$ and $\{101\}_{\text{rt}} // \{1\bar{1}0\}_{\text{grt}}$ (see Appendix; refer to Bonev, 1972 for the terminology for the phenomena of mutual crystal orientation). For these submicroscopic inclusions, the inclusion pockets are always enclosed by the lowest energy $\{110\}$ facets of garnet and good matching between the $\{110\}$ facets of garnet and the low index and hence low energy $\{100\}$ or $\{011\}$ planes of rutile were found.

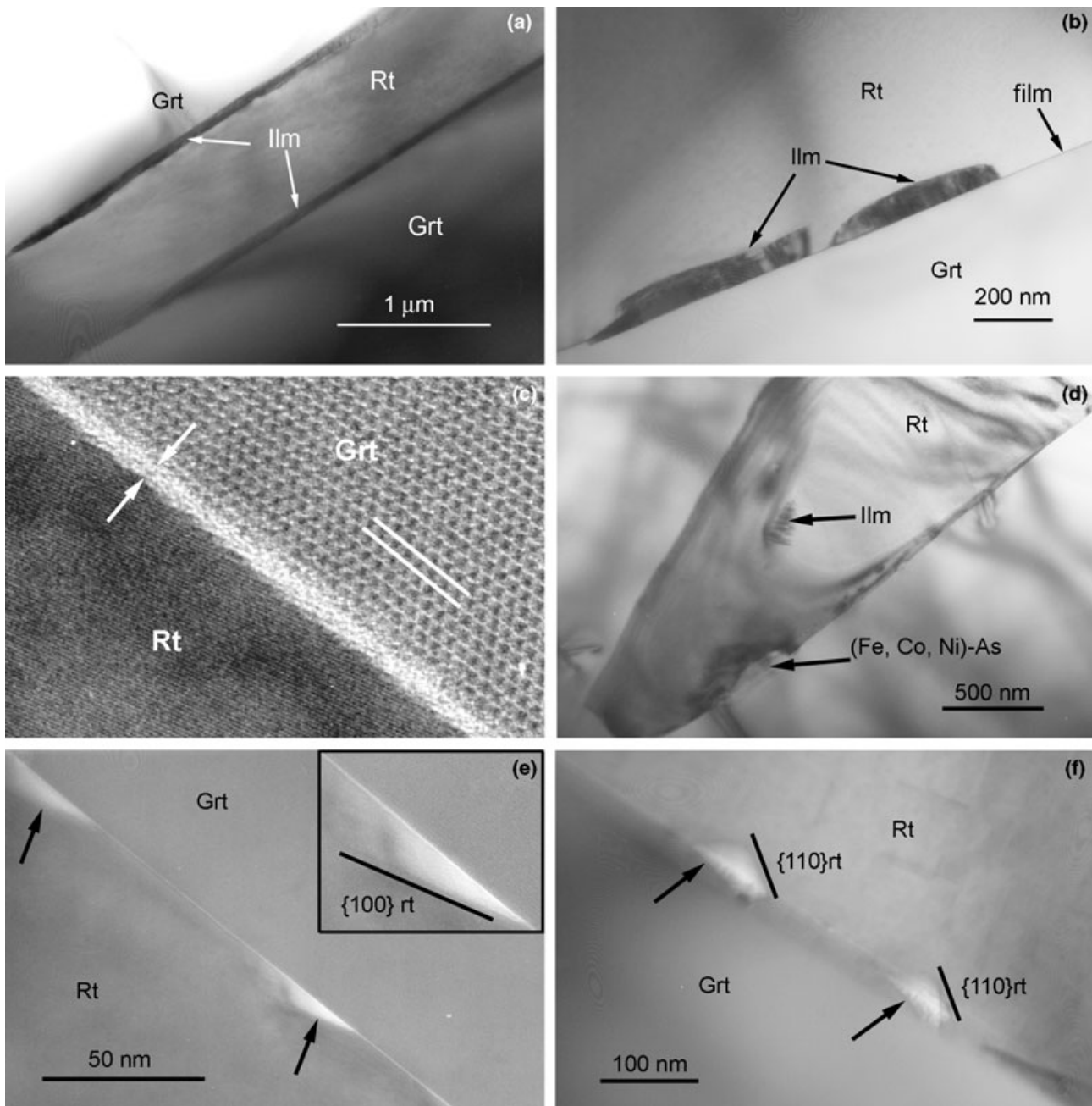


Fig. 4. AEM micrographs showing (a, b, d) the ilmenite islands, (b, c) the amorphous film (arrowed) of 1–2 nm thickness, (d) (Fe, Co, Ni)-arsenide (As) crystal at garnet–rutile interfaces, and (e, f) nanometer-size faceted pockets at surfaces of rutile needles in samples 96Y5A/5B. The spacing of the $\{110\}$ fringes of garnet in (c) is 8.1 Å.

Although the orientation relationships described above for rutile in garnet were most likely formed through epitaxial deposition and/or the post-entrapment crystal rotation of rutile inside the garnet crystal, they nonetheless represent the low energy interfacial configurations between rutile and garnet, and would therefore be preferentially followed if the rutile inclusions were formed by solid-state precipitation. Among all data plotted in Fig. 3, only one needle (a_2/c_2 , grew

along the $\langle 101 \rangle$ direction of rutile) follows the second set of these orientation relationships, providing matching between $\{110\}_{\text{grt}}$ and $\{011\}_{\text{rt}}$ along two opposite interfaces. For the rest of needles oriented along the $\langle 101 \rangle$ -normal of rutile, no specific orientation relationships were noted, and the $\{100\}$ side-faces of rutile needles are disposed randomly relative to the $\{110\}$ faces of garnet host (see the poles of the rutile a-axes plotted at the big circle perpendicular to the

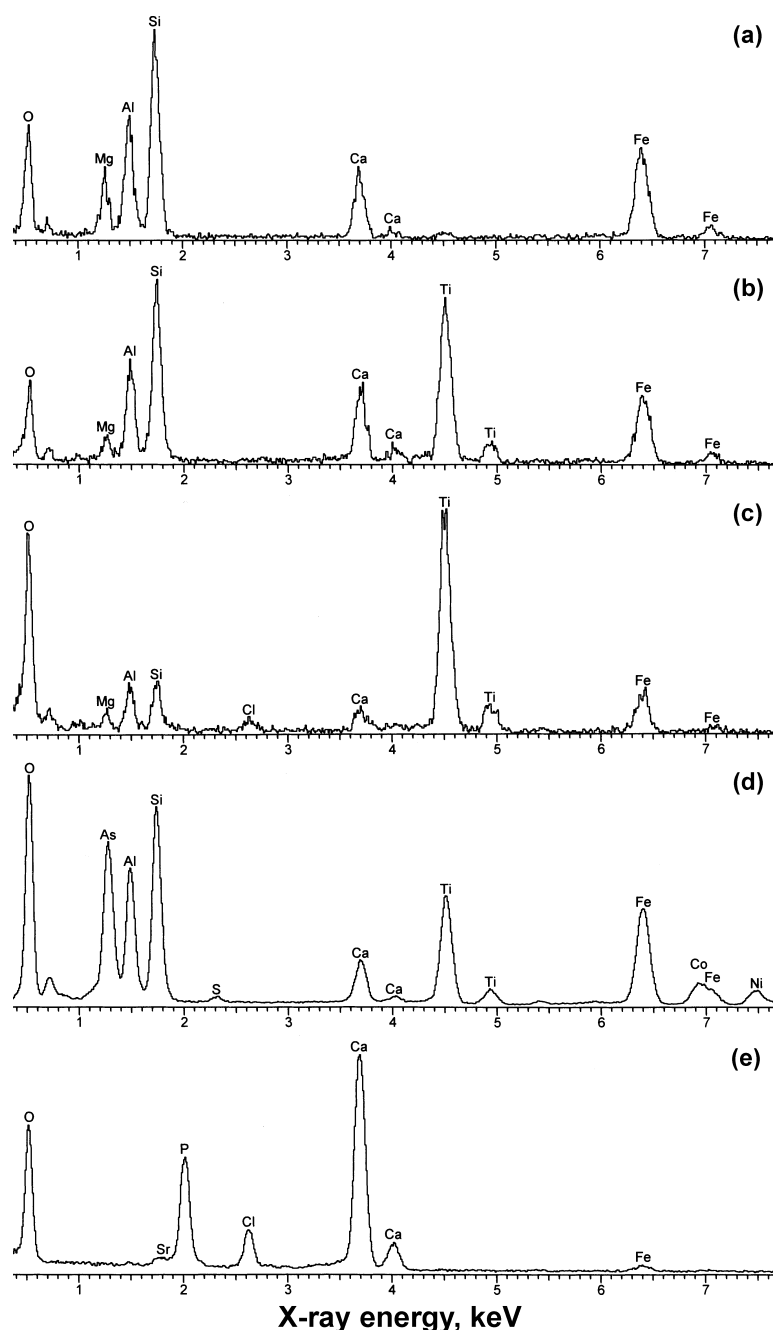


Fig. 5. Representative EDX spectra of (a) garnet matrix, (b, c) amorphous film along garnet-rutile interfaces, (d) (Fe, Co, Ni)-arsenide, and (e) apatite in samples 96Y5A/5B. The spectra of the films were interfered by neighbouring garnet and rutile. The abscissa is X-ray energy in the unit of keV, and the ordinate is X-ray relative intensity.

needle long-axis in Fig. 3). If the rutile was formed by solid-state precipitation and constrained to grow along the $\langle 111 \rangle_{\text{grt}}/\{101\}_{\text{normal}_{\text{rt}}}$, it is hard to explain why the $\{110\}_{\text{grt}}$ faces do not match the $\{100\}_{\text{rt}}$ faces to form the low energy $\{110\}_{\text{grt}}/\{100\}_{\text{rt}}$ interfaces as observed in the submicroscopic rutile inclusions in the garnet described above.

It is noted that ductile deformation at depth may modify the coherency of the topotactic relations between the oriented rutile needles and host garnet. However, this mechanism cannot explain the observed random lattice orientations in the present study (Fig. 3).

If the random lattice orientations of rutile were caused by deformation, the dominant $\langle 111 \rangle\{110\}$ slip systems of garnet would force rutile needles to follow the active $\{110\}$ slip plane instead of being evenly aligned along four $\langle 111 \rangle$ directions of the garnet host. Moreover, dislocation densities and subgrain boundaries revealed by AEM observations are rather limited (not shown).

The absence of pyroxene lamellae in garnet

In addition to the above microstructural evidence, consideration of the stoichiometry of precipitation

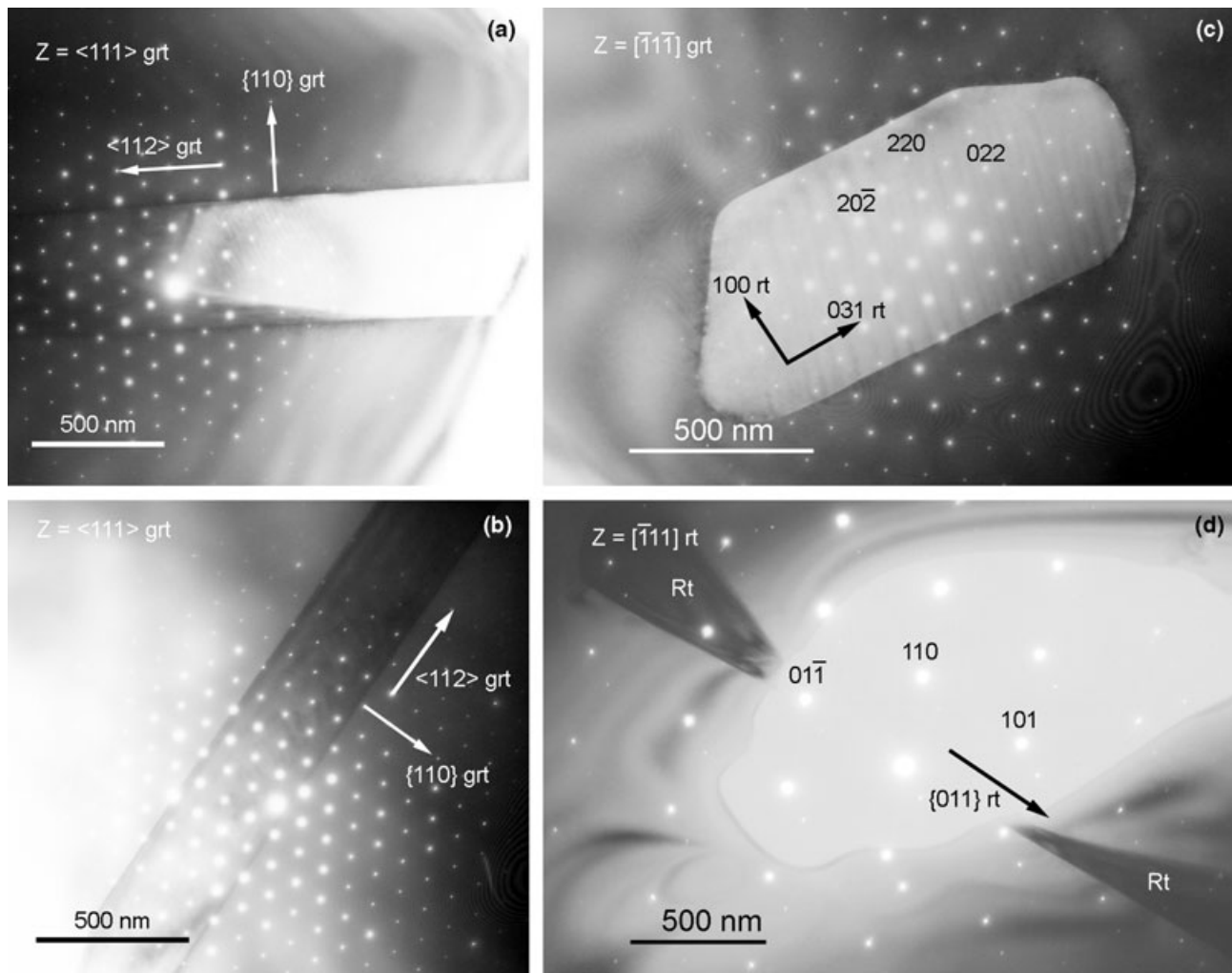


Fig. 6. Representative AEM micrographs with superimposed SAED patterns showing (a, b) the trace of rutile needle parallel to $\langle 112 \rangle_{\text{grt}}$ in samples (a) 25966 and (b) U3 when viewed along $\langle 111 \rangle_{\text{grt}}$, (c) the nearly prismatic cross-section of rutile needle in sample U3 viewed along $\langle 111 \rangle_{\text{grt}}/\{101\}$ -normal_{rt}, and (d) the $\{101\}$ -normal_{rt} growth direction of rutile needle in sample U3. Also shown in (c) are the directions of 100_{rt} and 031_{rt} reciprocal vectors in $[013]_{\text{rt}}$ zone axis ($\sim 6^\circ$ from $\{101\}$ -normal_{rt}). The zone-axis of the superimposed SAED pattern is given at the top left corner of each micrograph.

reaction(s) of rutile needles in garnet also does not support a simple solid-state precipitation model. The principal substitution for Ti solubility in garnet was suggested to be $\text{M}^{2+}\text{Ti}^{4+} = 2\text{Al}^{3+}$, with only subordinate $\text{Na}^{1+}\text{Ti}^{4+} = \text{M}^{2+}\text{Al}^{3+}$ ($\text{M} = \text{Ca}, \text{Mg} \text{ \& \; Fe}$) (Ringwood & Lovering, 1970; Armbruster *et al.*, 1998; Zhang *et al.*, 2003). Considering the mass balance, release of Ti from garnet would be accompanied by the loss of pyroxene and oxide components following a simple stoichiometric reaction where $\text{M}_3(\text{MTi})\text{Si}_3\text{O}_{12} = \text{TiO}_2 + 3/2 (\text{M}_2\text{Si}_2\text{O}_6)$ (pyroxene) + MO, or in a more sophisticated reaction involving Ca-Tschermak molecule and silica $\text{M}_3(\text{MTi})\text{Si}_3\text{O}_{12} + \text{CaAl}_2\text{SiO}_6 + \text{SiO}_2 = \text{M}_3\text{Al}_2\text{Si}_3\text{O}_{12} + \text{TiO}_2 + (\text{M}_2\text{Si}_2\text{O}_6)$ (pyroxene), as proposed by Zhang *et al.* (2005). Except for replacing $\text{TiO}_2 + \text{MO}$ with MTiO_3 , a reaction similar to the first one has also been sug-

gested by Ringwood & Lovering (1970) to explain the pyroxene-ilmenite intergrowths in mantle xenoliths. Although minor amounts of oxide were observed to be associated with rutile needles in the present study, pyroxene was absent, which contradicts the expectation that pyroxene with a much higher volume abundance (four to six times) than rutile should be present as a result of these reactions. The absence of pyroxene lamellae in association with rutile needles in garnet in the Yangkou eclogitic rocks is considered to be an argument against the precipitation scenario or a more complicated solid-state reaction.

It is worth mentioning here that the pyroxene precipitates in garnet have been suggested to indicate a parental high-pressure majoritic garnet (e.g. Haggerty & Sautter, 1990). However, following the above stoichiometric reactions, there should be caution in applying the

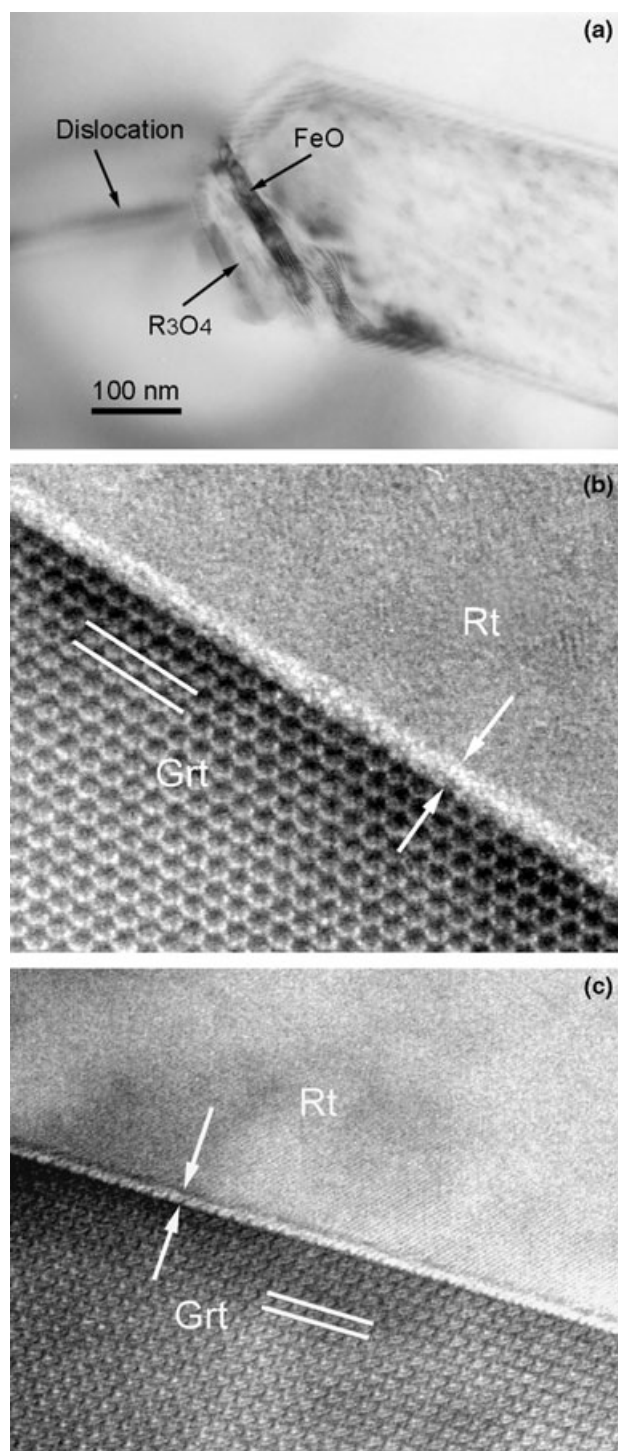


Fig. 7. AEM micrographs showing (a) the Al-, Fe-rich oxide phases (RO, R_3O_4) at the tip of a needle in sample U3, and the amorphous film (arrowed) of 1–2 nm thickness at garnet–rutile interfaces in (b) sample U3 and (c) sample 25966. The spacing of the {110} fringes of garnet is 8.1 Å.

scenario of majoritic component when both oriented pyroxene and rutile (or ilmenite) are present in garnet, because such a pyroxene phase may result from sub-

stitutions that are unrelated to the majoritic one, i.e. $M^{2+}Ti^{4+}$ rather than $M^{2+}Si^{4+} = 2Al^{3+}$.

Possible mechanisms for the formation of rutile needles in garnet in the present study

The samples in the present study have different characters. The two samples from Yangkou are eclogitic in nature with the oriented rutile needles evenly distributed within porphyroblastic garnet cores, whereas samples from Erzgebirge and Bohemia are quartzofeldspathic rock and felsic granulite, respectively, with oriented rutile needles unevenly distributed in (some) garnet grains. It is surprising to see that, despite the differences, all oriented rutile needles in these samples exhibit almost identical shape- and crystallographic-preferred orientation (Sidman *et al.*, 2003). This implies that there might be a common mechanism, other than solid-state precipitation, responsible for the formation of these oriented rutile needles in garnet.

Rutile needles inherited from precursor minerals

In studying microstructures of metapelites from the Eclogite Zone, Tauern Window, Stöckhert *et al.* (1997) suggested that some tiny rutile needles in garnet might have been formed from the TiO_2 content of the replaced mica. There are two possibilities following this postulate. First, the rutile needles now in garnet were originally rutile needles in sagenitic biotite. Such rutile needles were embraced within garnet crystals when garnet replaced biotite in the prograde stage. Second, rutile needles were newly formed when Ti-rich biotite were replaced by garnet during prograde reactions. The first possibility may be less likely because the rutile needles in sagenitic biotite lie within planes parallel to (001) of biotite and have a growth direction parallel to the c-axis of rutile (Shau *et al.*, 1991). These characteristics, obviously, are not compatible with those of the rutile needles in garnet in the present study. The second possibility, however, needs explanation as to why newly formed rutile needles lie specifically along the $\langle 111 \rangle$ direction of garnet formed contemporaneously, and why the rutile inclusions did not arrange into growth-sector patterns as observed in the oriented quartz rods in porphyroblastic garnet (Burton, 1986). Other similar postulates, such as rutile inclusions formed from Ti release as garnet grows by consuming other Ti-bearing minerals like titanite (Manning & Bohlen, 1991), ilmenite (Bohlen *et al.*, 1983) and hornblende (Blattner, 1976), would also face similar difficulties. Under all these hypotheses, Ti was not originally present in garnet.

Formation of rutile needles by dissolution–reprecipitation

It is possible that rutile needles, presumably accompanied by pyroxene lamellae, were indeed precipitation products of Ti-rich garnet as discussed above.

Melt infiltration processes might cause dissolution–reprecipitation of titania to demolish the lattice coherency between precipitated rutile and garnet, as well as leaching of pyroxene lamellae and formation of an amorphous layer between rutile and garnet. Most of the Ti may have an internal source, but some Ti import or leaching by the melt is also possible. The source as well as the chemical characteristics of the infiltrated melt need to be explored. The results of this process are actually difficult to distinguish from those of the cleaving–healing mechanism discussed below. Under this scenario, however, it is difficult to envisage that many mantle/UHP samples with the alleged rutile–pyroxene precipitation within garnet had all been subjected to subsequent, similar melt infiltration processes that left no trace or much less an amount of pyroxene and/or oxide relics within garnet (Griffin *et al.*, 1971; Zhang & Liou, 1999; Van Roermund *et al.*, 2000; Ye *et al.*, 2000; Mposkos & Kostopoulos, 2001; Song *et al.*, 2004; Zhang *et al.*, 2005).

Cleaving and healing of garnet with rutile deposition

One of the prominent features of the rutile needles in garnet in the present study is their oriented alignment within garnet but without specific crystallographic relationship. Similar characteristics were also reported for the oriented titanite needles in biotite, in which three sets of titanite needles form equilateral triangles parallel to the {001} of biotite and one set of such needles is parallel to the *a*-axis of biotite. In addition, titanite has no specific crystallographic relationship with biotite (Shau *et al.*, 1991). These oriented titanite needles in biotite have been suggested not to result from solid-state precipitation but as a corollary of fluid infiltrating into and reacting with biotite having percussion figures (fractures) induced by repeated high strain rate events (Xu & Ji, 1991; Yui *et al.*, 2001).

An analogue mechanism can be employed to account for the oriented rutile needles as well as the Al/Ca-rich thin amorphous layer and the impurity phase(s) in garnet in the present study, i.e. rutile needles were formed as a result of melt infiltration and reaction with garnet exhibiting {110} cleaving/parting planes. It could be that it is the garnet <111> directions defined by the intersections of garnet {110} partings that determined the apparent respective orientations of the rutile needles in garnet. Similar to the triple junctions in polycrystalline minerals, such <111> channels in garnet might be the energetically/kinetically favoured sites for the nucleation and growth of rutile crystals.

Under this cleaving–healing mechanism, garnet partings/cleavings and melt infiltration are the two major components in forming rutile needles in garnet. In the absence of either one component, there would be no such oriented rutile needles in garnet. This may explain that some garnet grains and/or garnet rims are free of rutile needles, whereas other grains and/or

garnet cores contain rutile needles in the samples studied. The contrast between the evenly distributed rutile needles in the garnet core of the Yangkou eclogite and the unevenly distributed rutile needles in the garnet of the Erzgebirge quartzofeldspathic rock and the Bohemia granulite can also be accounted for by different extent of garnet partings.

Extensive garnet {110} micropartings have been reported to be induced by high strain rate events for the UHP eclogitic rocks (Hwang *et al.*, 2001b), shock metamorphic Precambrian rocks (Dressler, 1990), and granulites next to pseudotachylytes (Austrheim *et al.*, 1996). Thermal and/or compressibility mismatch between mineral inclusions and garnet host could also introduce minor cracks (Whitney *et al.*, 2000). The infiltrated melt might be related to the high strain rate event, such as the formation of pseudotachylite in the Norwegian eclogites (Austrheim & Boundy, 1994). Partial melting during decompression might be another alternative (e.g. Schmidt *et al.*, 2004). Increasing evidence has shown that partial melting, at least on a limited scale, might have prevailed during the peak and immediate postpeak history of UHP metamorphism (Hwang *et al.*, 2001a, 2004; Stöckhert *et al.*, 2001; Massonne, 2003; Wallis *et al.*, 2005).

To fit the postulated parting and melt infiltration process into the respective *P–T–t* history of the samples studied, as well as to determine the origin/chemical characteristics of the infiltrated melt, would be important issues to substantiate the proposed cleaving–healing scenario. It is unfortunate that no geothermobarometer is available for *P–T* estimations of the cleaving–healing processes. It is also difficult, if not impossible, to determine the exact temporal relation or time gap between garnet {110} parting, the formation of thin amorphous layer, and the rutile deposition. However, considering that similar oriented rutile needles were mostly reported from samples of high pressure and temperature conditions (Griffin *et al.*, 1971; Zhang & Liou, 1999; Van Roermund *et al.*, 2000; Ye *et al.*, 2000; Mposkos & Kostopoulos, 2001; Song *et al.*, 2004; Zhang *et al.*, 2005), such cleaving–healing processes may have favourably taken place under very high *P–T* environments. The source of Ti in this case could be internal and/or external. In the former case, Ti was released from garnet through interaction with infiltrated melt; whereas in the latter case, Ti-rich fluid/melt has been shown to be present under high *P–T* conditions as inferred, for example, by the Ti-rich oxide mineral inclusions in mantle pyrope (Wang *et al.*, 1999) and rutile veins in eclogite (Klemd *et al.*, 2006). Although this crack-filling mechanism accounts for most observations and is preferred by the authors, further study is required to clarify whether rutile having a needle-like, but not a plate-like habit, is due to diffusion channels defined by the intersections of the garnet {110} partings as mentioned or partial healing of the partings before TiO₂ filling.

The peculiar growth habits of rutile inclusions in pyroxene and garnet of kimberlitic/UHP rocks

Despite the lack of specific orientation relationships between rutile and garnet host, it has been demonstrated that the needles preferred to grow along predominant $\{101\}$ -normal and subordinate $\langle 101 \rangle$ directions of rutile. These growth directions are markedly different from the $\langle 001 \rangle$ growth direction, which is typical for freely growing natural rutile crystals. Interestingly, these peculiar $\{101\}$ -normal/ $\langle 101 \rangle$ growth directions were also observed from rutile inclusions in pyroxene of some kimberlitic eclogites (Griffin *et al.*, 1971). In one of the pioneer papers on rutile inclusions of kimberlitic eclogites, Griffin *et al.* (1971) reported elongated rutile inclusions in both pyroxene and garnet grains. In comparison with the tiny and well-oriented rutile inclusions within garnet grains, the rod-like rutile inclusions are larger and less well-oriented along three directions in pyroxene grains of kimberlitic eclogites. By extracting one large rod-like rutile grain from pyroxene, Griffin *et al.* (1971) observed an apparent extinction angle of $32 \pm 1^\circ$, which is close to the inter-axial angle of either 32.8° between the $\{101\}$ -normal and the c-axis of rutile, or 33° between the $\langle 101 \rangle$ and the a-axis of rutile. In other words, the rod-like rutile inclusions in pyroxene might well be elongated along either the $\{101\}$ -normal or $\langle 101 \rangle$ directions of rutile, the same as the case in garnet despite the prominent structural difference of the two hosts. Thus the non-epitaxial rutile inclusions in pyroxene and garnet of kimberlitic/UHP rocks must have been subjected to rather limited matrix constraint during nucleation and growth, i.e. little to do with a precipitation origin. The structural and/or kinetic factors dictating these peculiar growth habits of rutile inclusions within kimberlitic/UHP rocks are yet to be explored.

CONCLUDING REMARKS

The oriented rutile needles evenly distributed in the garnet core of the Yangkou UHP eclogitic rocks and previously inferred to be precipitation features are shown to have no specific crystallographic relation with the garnet host in the present study. Together with the absence of pyroxene lamellae in garnet, the presence of a thin amorphous layer and ilmenite between rutile and garnet, as well as the presence of faceted pockets on rutile surfaces, the present AEM observations strongly argue against the simple solid-state precipitation scenario. It is also surprising that similar microstructural and crystallographic characteristics were observed for some oriented rutile needles unevenly distributed within garnet in other UHP quartzofeldspathic rock and HP felsic granulite samples besides eclogitic rocks.

Three mechanisms are considered to account for the observations: such that the rutile needles may be

(i) inherited from precursor minerals; (ii) formed by a dissolution–reprecipitation mechanism; and (iii) formed by cleaving and healing of garnet with rutile deposition. Each mechanism has its own difficulties to fully explain the available data. Whereas the first mechanism is considered less likely, the third mechanism is preferred because of the natural analogue of oriented titanite needles in biotite, although it is still difficult to explain the rutile habit. It should be noted that the source of Ti for these rutile needles would be largely external for the first mechanism, and external and/or internal for the second and the third mechanism. In both the second and the third mechanisms, a melt phase is suggested to be involved.

The presence of oriented apatite in garnet of UHP rocks is usually taken as evidence for the phosphorous-solubility in garnet according to the coupled substitution $\text{Na}^+ \text{P}^{+5} = \text{Ca}^{+2} \text{Si}^{+4}$ (Bishop *et al.*, 1976; Brunet *et al.*, 2006). However, the high Cl-content in apatite, as well as the presence of both epitaxial and non-epitaxial apatite in the Yangkou eclogitic garnet do not support the solid-state precipitation model. The formation mechanism and the growth behaviour of apatite inclusions appear to be similar to, although may not necessarily be syngenetic with, rutile needles.

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APPENDIX

Crystallographic orientation relationships between nanometer-size rutile inclusions and the garnet host

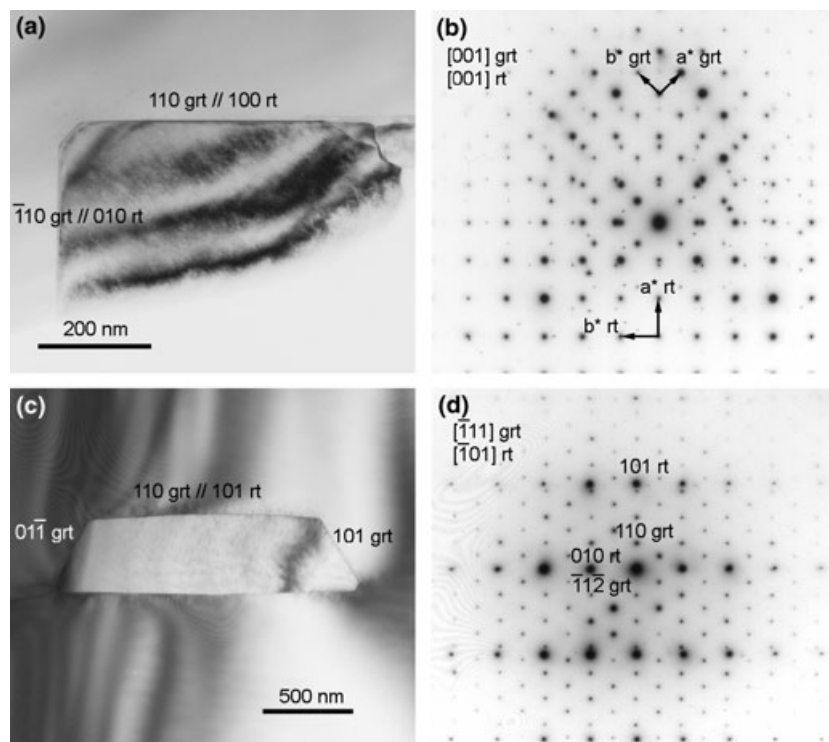


Fig. A1. Bright-field image and associated SAED patterns showing the crystallographic orientation relationships: (a, b) $\{100\}_{\text{rt}}//\{110\}_{\text{grt}}$ and $[001]_{\text{rt}}//[001]_{\text{grt}}$; or (c, d) $(101)_{\text{rt}}// (110)_{\text{grt}}$ and $[101]_{\text{rt}}/[111]_{\text{grt}}$ between submicroscopic rutile inclusions and garnet host from whiteschist of Kulev area, Kokchetav massif.