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High Voltage Electron Microscopy of Deformed Sodic Plagioclase from an Alpine Gneiss

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With 6 figures

Abstract

The sodic plagioclase in a biotite-epidote gneiss from the Zillertal Alps (Tyrol, Austria), described by RAITH (1969) as a peristerite containing coarse ($\sim 1 \times 10$ μm) exsolution lamellae has been reinvestigated by 1000 kV and 100 kV transmission electron microscopy. The plagioclase is found to contain only very fine exsolution lamellae 20–80 nm apart and a complex dislocation texture. Planar dislocation networks define disk-shaped elongated subgrains approximately parallel to the exsolution lamellae. These subgrains are approximately 1 μm thick and 10 μm long and could be confused, when viewed by optical microscopy, with a coarse exsolution texture.

INTRODUCTION

This paper describes the application of high voltage (1000 kV) and 100 kV electron microscopy to characterise the exsolution texture and the subgrain fabric due to deformation in the sodic plagioclase component of a strongly deformed metamorphic rock (tectonite).

The rock specimen, number HU 280, was kindly provided by Dr. Michael Raith, University of Kiel, Germany. It is a fine-grained biotite-muscovite-epidote gneiss from Hundskehlgrund, Eastern Zillertal Alps (North Tyrol,

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Austria, adjacent to the Italian border). The gneisses are assumed (RAITH, 1969, 1970) to originate from granites to quartz diorites of pre-alpine age which were deformed and reheated much later during the alpine orogeny under conditions of the staurolite-almandine-amphibolite subfacies («Tauernkristallisation», SANDER 1912).

The composition of the plagioclase of the gneiss was determined by electron microprobe analysis at the University of Manchester as approximately An 12. It was described by RAITH (1969) as peristerite, i. e. exsolved sodic plagioclase, because of the appearance of irregular ellipsoidal to lamellar units in the polarisation, phase contrast and interference microscopes. These planar textural units were found by RAITH to be oriented near $(08\bar{2})$ (indexed for the anorthite cell with $c \approx 14$ Å) which is the plane of exsolution in peristerites. The oligoclase often includes epidote and "amoeboid" quartz. From optical measurements, using the zone method (RITTMANN, 1929) in sections normal to (010), RAITH (1969) concluded that the two kinds of materials differing in their optical contrast represented albite (An 0–5) and oligoclase (An 17–25). The bulk composition was not determined, but specimens with sodic plagioclase regarded as original ("plagioclase I"), which appears homogeneous in the optical microscope, was reported by RAITH to have compositions An 10–17. In the lamellar plagioclase, as investigated in the phase contrast microscope, he reported a higher refractive index for the material supposed to be oligoclase and a lower index for the material regarded as albite. He also assumed "that the exsolved albite domains have elliptoidal to lamellar shape", lengths $\leqq 10-15$ μm and width $\leqq 1$ μm.

ELECTRON MICROSCOPY

Areas in doubly-polished thin-sections suitable for transmission electron microscopy were thinned by ion etching techniques described by BARBER (1970) and CHAMPNESS and LORIMER (1971) and were examined at 100 and 1000 kV. The combination of ion-thinned samples and 1000 kV microscopy proved particularly useful for determining the dislocation distributions over large areas of the sample and in relating the dislocation distributions to the microstructure observed optically.

Fig. 1 is an optical micrograph of the sample showing Albite-law twins with irregular boundaries. These twin lamellae contain smaller, straight, Albite-law twins. A texture can also be resolved which is composed of dark and white ellipsoidic or lamellar units inclined with their elongation direction approximately 13° to the Albite-law twins and approximately 76° to the trace of the (001) cleavages. The section is cut approximately normal to the a-axis, and the micrograph is similar to Fig. 4 of the paper by RAITH (1969). The irregularity

of the twin boundaries is a feature consistent with extensive non-uniform plastic deformation of the matrix.

Figs. 2, 3 and 4 are electron micrographs of typical areas of the sample. Figs. 2 and 3 are high-voltage electron micrographs which show a high density of dislocations, approximately 10^{12} lines/cm^2; many of the dislocations have formed subgrain boundaries (arrowed), and dislocation networks are frequent. The lamellar feature labelled A-A in Fig. 3 is the remnant of a (010) twin which itself contains many smaller twins (compare Fig. 1) as well as numerous dislocations and subgrain boundaries (details in Fig. 4). The displacements normal to the trace of the (010) twin planes are clearly visible and indicate shear of the primary twin during deformation.

Figs. 2 and 5 show very fine lamellae approximately parallel to $(08\bar{2})$, the plane of peristerite exsolution (for the $c \approx 14$ Å cell). In contrast to other peristerites so far investigated – including those with bluish schiller colour due to interference of light by the regular lamellar texture – the thicknesses and the periodicity of lamellae varies within very small areas from 20 to 80 nm (Fig. 5).

The variation in periodicity is irregular and not related to the local density of dislocations or the distance from the subgrain boundaries. However, the elongation direction of the subgrains and the majority of their boundaries are on the average parallel to the trace of the $(08\bar{2})$ lamellae.

Electron diffraction patterns taken at 100 kV show no evidence of two different cell geometries corresponding to two plagioclase phases, and it is therefore probable that the exsolution structure diffracts coherently (i.e. as one lattice) as described by KOREKAWA et al. (1970). Fig. 6 shows the fine lamellae in greater detail. The (020) spacings are resolved and can be seen to cross the exsolution boundary without any deviation, indicating that at least for the (020) planes there is complete strain-free coherence across the exsolution boundaries.

DISCUSSION

Although the lamellar structure visible optically has to be considered a deformation structure and not an exsolution texture, the specimen is nevertheless exsolved. The very fine scale and the strain-free coherence of the interface between the two phases indicates that this exsolution represents the initial stage of phase separation by spinodal decomposition, whereby the composition modulations are gradually built up without a nucleation stage. Such fine coherent microtextures consistent with the early stages of spinodal decomposition have been found for several compositions between An_2 and An_{25} (KOREKAWA et al., 1970; NISSEN, 1972; MCLAREN, 1974); some of these show a "tweed" structure i.e. modulations in two directions ($(08\bar{2})$ and approxi-

mately (100) for the $c \approx 14$ Å cell) on a smaller scale than the present structure. Studies in pyroxenes (Champness and Lorimer, 1971, 1972) have shown that a "tweed" structure is the primary decomposition product, which is later replaced by a lamellar texture due to the preferential coarsening of one of the two initial modulations. This suggests that the exsolution texture in the plagioclase is extensively coarsened.

The wavelength of the exsolution texture varies from 20 to 80 nm over distances less than 1 μm, as can be seen in Fig. 5. A possible reason for this could be a difference in the chemical composition of the areas containing the fine or the coarse lamellae, although it is difficult to see how such fine scale composition variations could have arisen. We have used the analytical electron microscope, EMMA-4, to test this hypothesis. In a thin foil specimen it is possible to carry out quantitative chemical analyses from areas less than 100 nm using EMMA-4 (for a description of the instrument and its application to the analysis of minerals see Lorimer and Champness (1973) and Nissen et al. (1973)). We did not detect any difference in the Al, Si or Ca concentrations from adjacent areas containing fine and coarse lamellae; we estimate the sensitivity of the technique to $\pm 1\%$ Ca and $\pm 3\%$ Al and Si. No measurement was made of the K concentration in either area and it may thus be possible that variations in K-content influence the periodicity of the lamellae. A second explanation is that plastic deformation (dislocation motion) during the exsolution process provided local "short circuit diffusion paths" and allowed a coarse texture to develop. We have not noted any correlation between the scale of the microstructure and the dislocation density, however a dislocation-enhanced local diffusion cannot be completely ignored. A third explanation for the variation in the scale of the exsolution structure is *random* non-uniform coarsening of an initial decomposition product which, presumably, formed by spinodal decomposition.

The dislocation distributions observed in Fig. 2–4 and the optical features in Fig. 1 are similar to the electron- and light-optical features, respectively, observed by White (1973) in naturally deformed quartzites, by Boland, MacLaren and Hobbs (1971) in naturally deformed olivine, and by Lorimer et al. (1972) and Champness et al. (1973), in naturally deformed omphacite and albite. In each of the above, the dislocation distributions are consistent with solid state creep-deformation by dislocation glide accompanied by the diffusion-controlled climb of dislocations into stable arrays (subgrain boundaries). We propose that the microstructural features observed in the present sample have also arisen during solid state creep. This mode of deformation is consistent with the geological history of the sample.

The parallel orientation of subgrain boundaries and exsolution lamellae may arise through an elastic interaction between the dislocation stress fields and the modulated structure, although this explanation is highly speculative.

Fig. 1. Optical micrograph (crossed polars) showing (010) twins and ellipsoidic or lamellar features approximately parallel to $(08\bar{2})$, slightly inclined to the right. Traces of (001) – cleavage are vertical.

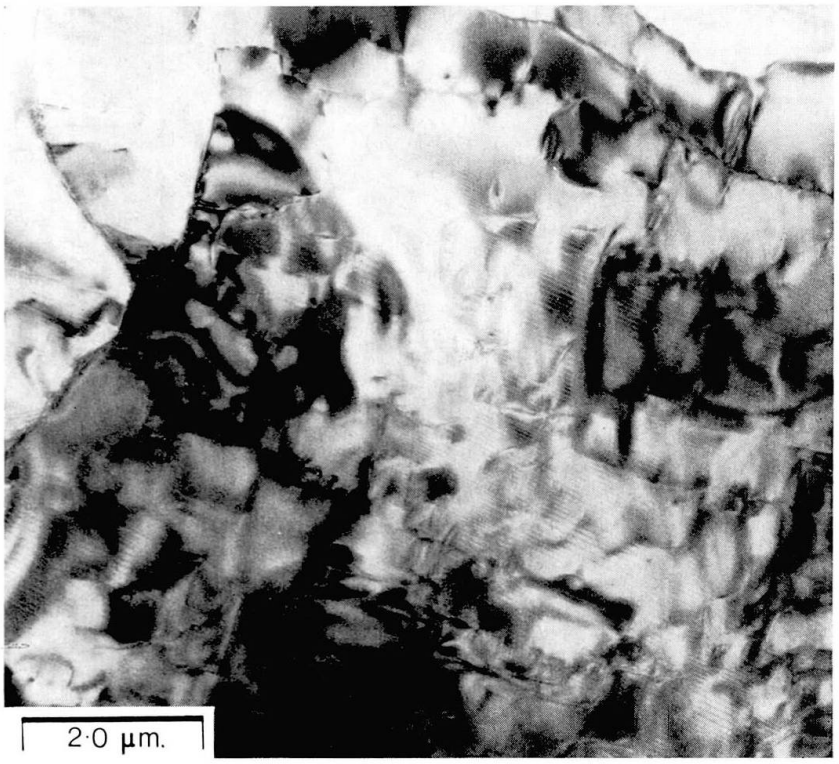
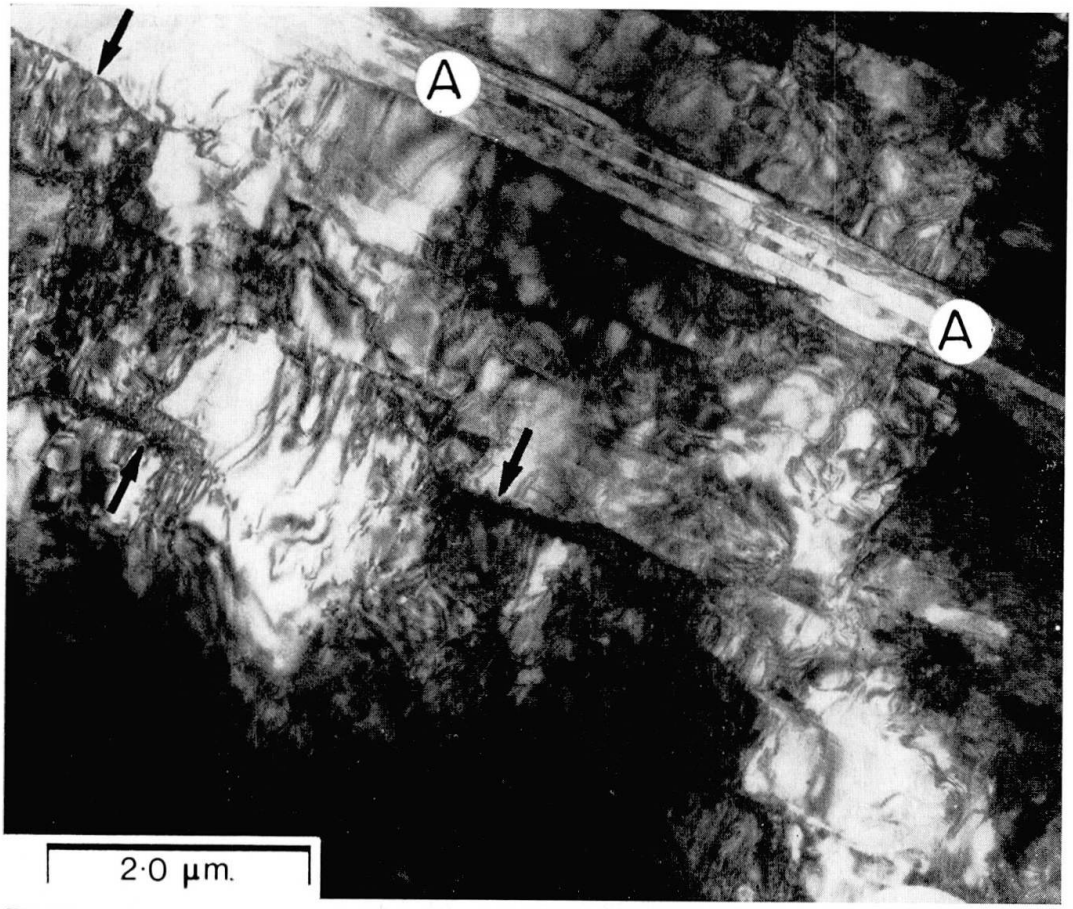


Fig. 2. 1000 kV electron micrograph showing elongated subgrains approximately parallel to exsolution lamellae.



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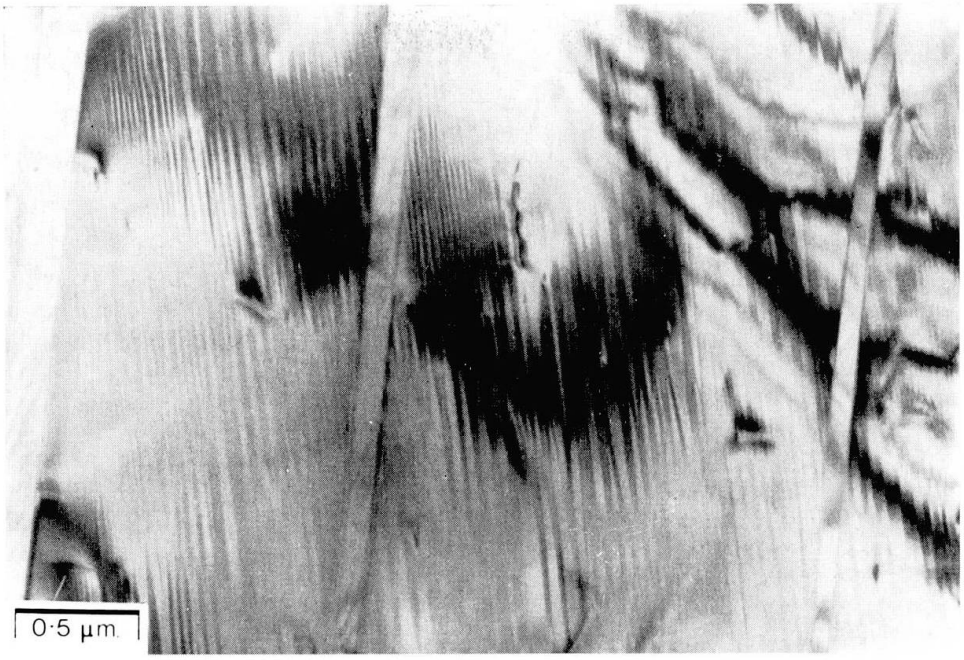

Fig. 5. 100 kV electron micrograph showing the variation in periodicity of the exsolution lamellae and three Albite twins (approximately vertical). The wavelength of the exsolution varies from 20 to 80 nm.

CONCLUSIONS

The plagioclase contains a lamellar exsolution texture parallel to $(08\bar{2})$ (for $c \approx 14$ Å) with a periodicity of 20–80 nm.

The sample has been extensively plastically deformed; the microstructure consists of individual dislocations and dislocation networks along sub-grain boundaries approximately parallel to the exsolution lamellae. The structure is consistent with a solid-state creep process.

This study shows the value of transmission electron microscopy for investigating microstructural features at or near the resolution limit of the optical microscope. The combination of high voltage transmission electron microscopy and ion thinning enables the *distribution* of microstructural features to be ascertained and allows its correlation with the optically observable microstructure.

Fig. 3. 1000 kV electron micrograph showing deformation substructure. The feature labelled A–A is an (010) Albite twin remnant which has been sheared during the deformation. Numerous sub-grain boundaries can be resolved (some of which are arrowed) as well as individual dislocations.

Fig. 4. 1000 kV electron micrograph showing details of substructure in Fig. 3. The twin contains a high density of "second generation" twins.

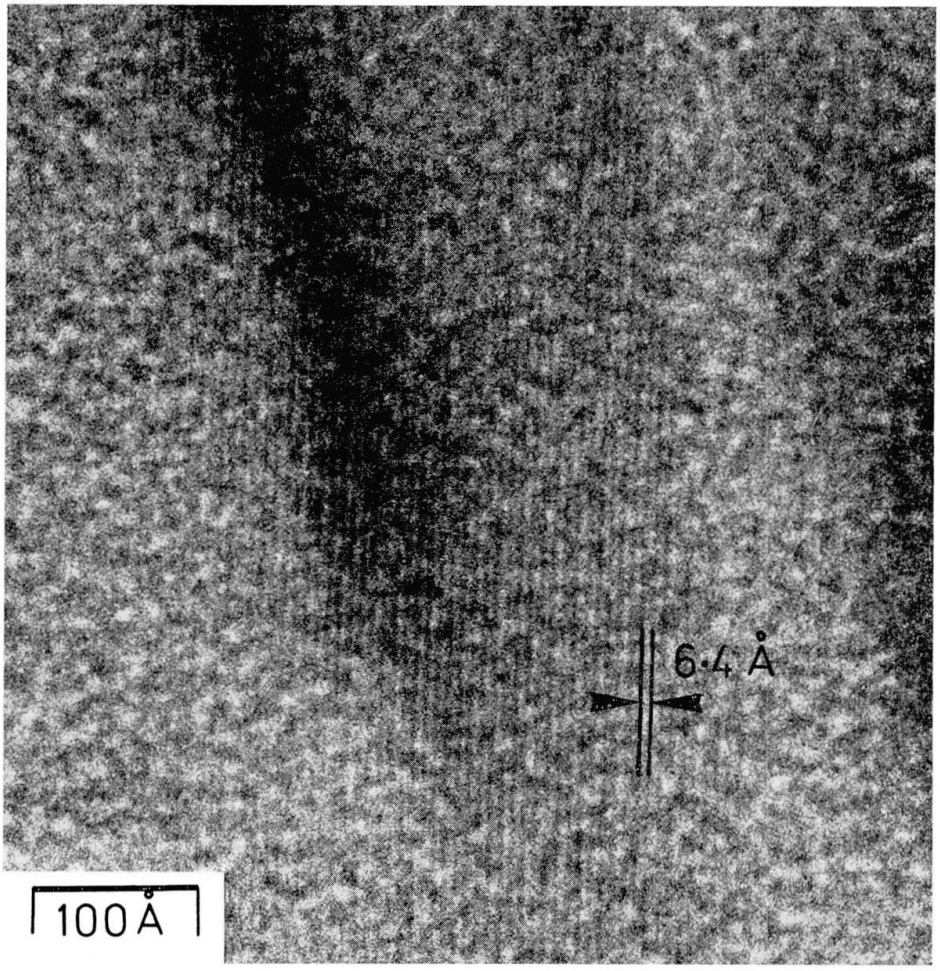

Fig. 6. Direct lattice image (100 kV) of (020) planes showing coherency of (010) lattice planes across the interface between two lamellae.

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