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Grandite garnet from Nevada: confirmation of origin of iridescence by electron microscopy and interpretation of a moiré-like texture

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Abstract

The presence of regularly stratified layers in an iridescent garnet, predicted by an optical study, is confirmed by electron microscopic observations. Analytical electron microscopy showed that the layer texture is composed of major Fe-rich lamellae and minor Al-rich ones. TEM and SEM observations suggest that the fine layer texture probably resulted from exsolution. A possible mechanism for producing the moiré-like texture is presented.

Introduction

Optical interference measurements on iridescence were carried out by Hirai and Nakazawa (1982) for garnets collected in the Adelaide mining district, Nevada (Ingerson and Barksdale, 1943). Hirai and Nakazawa predicted from the optical study that the iridescence is caused by regularly stratified layers parallel to the {110} crystal surface in each sector of a modified dodecahedral crystal and that the period of the layers is about 1000 Å. Another microscopic lamellar texture was observed with polarizing light microscopy, scanning electron microscopy (SEM), and electron probe microanalysis (EPMA) (Hirai et al., 1982; Akizuki et al., 1984). The directions and spacings of the microscopic lamellae change in different major zones.[1] This lamellar texture resembles an optical moiré pattern, and thus the texture is called in this paper a moiré-like texture. However, this texture is not simply an optical effect, because a chemical difference was detected by back-scattered electron imaging (BEI) (Hirai et al., 1982). In the previous paper, the moiré-like texture was considered to be exsolution lamellae, although such a moiré-like pattern would be unusual as an exsolution texture. From similar observations Akizuki et al. (1984) suggested that iridescence resulted from oscillatory zoning, and the moiré-like texture was a growth texture. The formation mechanisms of this strange pattern and the detailed nature of the layer texture have remained as open questions.

The main purposes of this study are (1) to confirm the presence of the predicted fine layer texture by electron microscopy, (2) to interpret the origin of the fine layer texture, and (3) to clarify the textural relation of the fine layer texture to the moiré-like texture.

Experimental

Transmission electron microscopic observations

After polarizing microscopic observations and optical interference studies (Hirai and Nakazawa, 1982; Hirai et al., 1982), the same thin sections were thinned by ion milling. With an electron microscope (Hitachi H-700), fine layers are observed in the thinned foil (Fig. 1). These layers are arranged parallel to the {110} crystal surface in each sector. At high magnification, the layer texture was found to be composed of alternating major and minor lamellae. Thickness changes along the lamellae were observed but are more noticeable in SEM images described later. In spite of thickness changes in individual lamellae, the period of the alternation of the major and minor lamellae is constant within a major zone and is observed to vary between different major zones from 1800 Å to 7500 Å (e.g., Fig. 2a). Interfaces between the major and minor lamellae are very sharp. The sharpness of the interfaces is better observed in crushed specimens, because the ion-thinned foils were rough and/or bent. Contrast corresponding to the moiré-like texture was not recognized in the TEM.

Chemical analyses by analytical electron microscopy (AEM)

During TEM observations, the major lamellae of crushed specimens were preferentially evaporated by the electron

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[1] The terms major zoning (MZ) and oscillatory zoning (OZ) are used here for two types of chemical zoning that occur during crystal growth; usage is the same as for plagioclase (Smith, 1974, p. 206). The former term is used for relatively large-scale zoning, on the order of microns or more, with discrete compositional gaps (e.g., those observed by Lessing and Standish, 1973; Murad, 1976; Hirai et al., 1982). The latter term is used for fine-scale compositional fluctuations within the major zoning (on the order of a micron or less). This type of fluctuation may be formed by fluctuation of the degree of saturation at solid–liquid interfaces and/or temperature (e.g., Loomis, 1982).

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Fig. 1. A TEM image of an ion-thinned foil. Fine layers with period of about 2000 Å are observed throughout the foil.

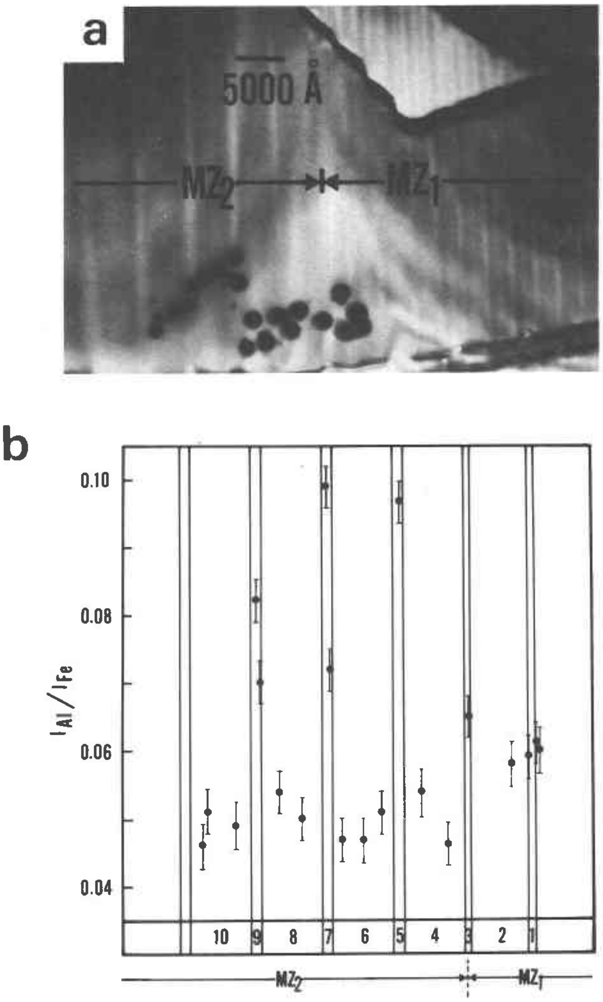

Fig. 2. (a) An electron micrograph showing the area analyzed by AEM. Contamination marks indicate the analytical positions. Note that the periodicity of the layers is different in the major zones indicated by MZ_1 and MZ_2. (b) Plots of intensity ratio of Al to Fe at each lamella. The compositional difference between the minor lamellae (1, 3) and (5, 7, 9) may be due to the fact that they are contained in different major zones, MZ_1 and MZ_2, respectively.

beam, suggesting that there is a difference in composition between the major and minor lamellae. A quantitative analysis was carried out at an accelerating voltage of 100 kV with beam diameter of about 300–400 Å. The major lamellae are relatively Fe-rich, and the minor lamellae are Al-rich (Fig. 2). The compositions are almost constant among the major lamellae and the minor lamellae within a major zone; the mean compositions in the major zone (MZ_2) are And_{87} and And_{78} for the major and minor lamellae, respectively. Considering that the probe size is wider than the minor lamellae, the actual compositions of the minor lamellae may be more enriched in Al than those measured. The change in composition between lamellae appears to be quite abrupt (Fig. 2b).

Electron diffraction patterns and dark field images

The electron diffraction pattern confirms the presence of two phases. Splitting of spots is observed in higher-angle reflections. The direction of the splitting is normal to the lamellae. From the amount of the split, difference in unit cell size was estimated to be about 0.02 Å.

Dark field images show that the major lamellae are all bright or all dark at the same time, depending on orientation. The minor lamellae showed the inverse contrast to that of the major lamellae. The simultaneous change in contrast of each set of lamellae suggests that the individual lamellae within a set possess the same diffraction conditions. The diffraction pattern and dark field images clearly indicate that the layer texture is composed of two discrete phases which have slightly different diffraction conditions.

SEM observations

To elucidate the relationship between the moiré-like texture and the fine layer texture, we used BEI in an SEM. A BEI obtained from a polished thin section cut normal to a {110} crystal surface is reproduced in Figure 3. Similar images were reported by Akizuki et al. (1984). In Figure 3, both the fine layer texture and the moiré-like texture can be seen. The fine layer texture is composed of dark minor lamellae and bright major lamellae. The thickness of each lamella changes almost periodically along its elongation, as drawn schematically in the sketch of Figure 3; there are alternately wider parts and narrower parts in each lamella. This thickness change is more obvious in Figure 7 of Akizuki et al. (1984). In spite of the thickness change, the period of the alternation of the major and minor lamellae is constant within a major zone and it is different in different major zones (Mz_i–Mz_k, Fig. 3) varying from 1700 Å to about one micron.

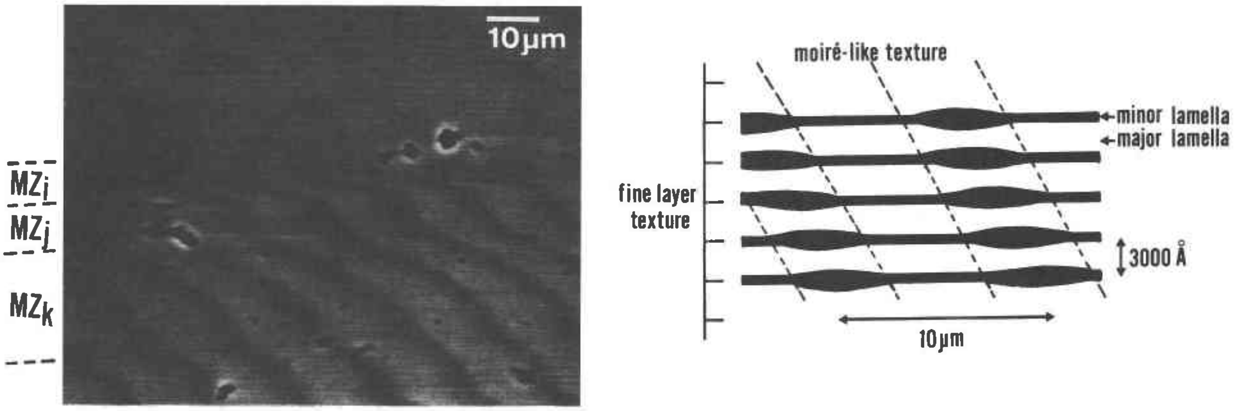

Fig. 3. Back-scattered electron image (BEI) of a polished thin section normal to a {110} face. Brighter parts are relatively Fe-rich and darker parts are Al-rich. Note the thickness change in some of the fine lamellae. The thickness change is more clearly observed in Figure 7 of Akizuki et al. (1984). The correlation of the fine layer texture to the moiré-like texture is schematically shown in the sketch, where the width of each lamellae is emphasized.

Because the thickness change of each fine lamella is almost periodic, the arrangement of such lamellae produces an apparent texture: the wider parts of the bright major lamellae form the bright areas of the moiré-like texture, while the wider parts of the dark minor lamellae alternatively make dark areas (Fig. 3). Under the TEM, the thickness change could, however, not be observed clearly, probably because of complex contrast arising from the roughness and bending of the foil.

Discussion

Interpretation of the origin of the fine layer texture

The detailed observations and analyses with the TEM, SEM and AEM in the present study show that the origin of iridescence is the layer texture mentioned above and that the layer texture is composed of thicker Fe-lamellae and thinner Al-rich lamellae with thickness variations. The origin of the layer texture is probably exsolution or oscillatory zoning, although twinning was previously proposed (Hirai and Nakazawa, 1982).

In exsolution, or phase separation from a homogeneous phase into two discrete phases, the interface of two phases is commonly sharp except in the case of spinodal decomposition. On the other hand, oscillatory zoning generally consists of fine-scale compositional fluctuations in a major zone and is due to the change in the degree of saturation at solid–liquid interfaces (e.g., Loomis, 1982). In such a fine-scale fluctuation, compositions may be randomly varied, and the compositional changes from one to the next

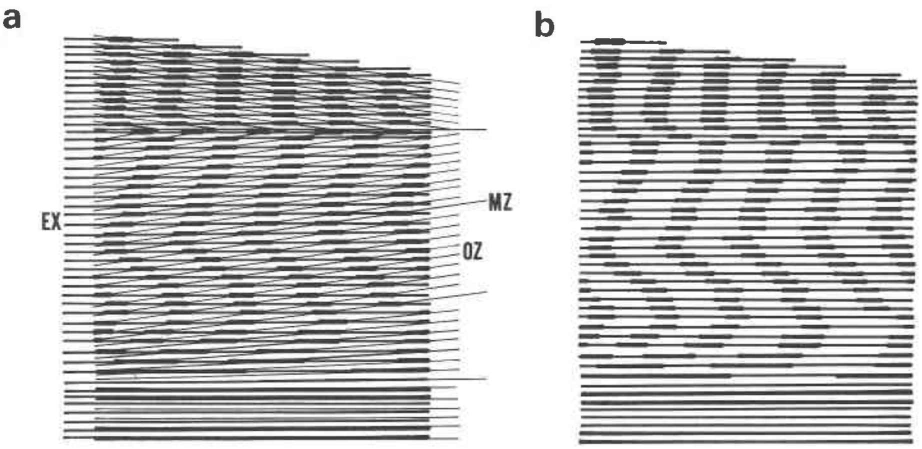

Fig. 4. A possible model to explain the formation of the moiré-like texture. Compositional distribution in the section normal to {110} is schematically illustrated. (a) Longer light lines indicate the boundaries of major zones (MZ). The crests of Al-content (or Fe-content) in the oscillatory zoning are indicated by shorter light lines (OZ). The inclinations of oscillatory zoning to {110}, which are different in different major zones, are exaggarated. Al-rich (or Fe-rich) exsolution lamellae which are parallel to {110} in all major zones are indicated by heavy lines which have thickness changes (EX). (b) The resulting compositional distribution at the present time. Thickness changes of the exsolution lamellae produce the apparent moiré-like pattern.

layer are expected to be more gradual. The two discrete phases observed in the dark field images and the diffraction pattern and also the sharp boundaries between the two cannot readily be explained by oscillatory zoning, and we therefore suggest that they were formed by subsolidus decomposition. Exsolution is thus the most likely explanation for the origin of the layer texture, although oscillatory zoning, as suggested by Akizuki et al. (1984), is not completely ruled out.

A model to explain the moiré-like texture

In order to explain how the compositional distributions result in an apparent moiré pattern, a possible model is presented below and in Figure 4.

Relatively large scale oscillatory zoning (indicated by OZ in Fig. 4) is present first in the garnet. The oscillatory zoning is thought to have been slightly inclined to {110} in the range of a few degrees (Fig. 4a), because the major zones (MZ) are often observed to be slightly inclined by a few degrees (e.g., Fig. 3). With decreasing temperature, exsolution occurred, forming lamellae exactly parallel to {110}. Because diffusion is probably limited at low temperature, the distribution of elements after subsolidus decomposition may reflect that of the oscillatory zoning; therefore, an Al-rich (or Fe-rich) exsolution lamella may become thicker where it intersects an Al-rich (or Fe-rich) oscillatory zone (Fig. 4a). At the final stage of exsolution, the relic oscillatory zoning remains and is reflected as thickness changes of the exsolution lamellae (Fig. 4b). This is a probable explanation of the formation mechanism of the moiré-like texture.

Since the inclination and the period of the oscillatory zoning and also the period of the exsolution lamellae are different in different major zones, intersecting positions of both layerings change with different major zones, resulting in wavy and straight features that look like a moiré pattern (Fig. 4b). Compositional gaps are observed among the major zones in the garnet, suggesting that long-range diffusion among major zones did not occur, while short-range diffusion within a major zone did occur during exsolution.

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