 Combined TEM-AFM studies of “transrotational” spherulites growing in amorphous films

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Crystal growth in amorphous film can be accompanied by formation of well-known spherulites (with primarily azimuth lattice misorientations around axis normal to the film plane - “cylindrites”, in fact) and less-known “transrotational” [1] crystals (with internal lattice bending round axis or axes lying in the film plane). In the present paper we study the “transrotational” spherulites having both 2 kinds of misorientaions, attained as result of nucleation and growth. We use transmission electron microscopy (TEM), primarily bend contour technique and in situ studies (including video-records analysis), combined with atomic force microscopy (AFM) and optical microinterferometry. Two different kinds of spherulites were produced in amorphous films prepared by vacuum condensation (Se, with Te doping) and pyrolysis (Fe₂O₃), separated from the substrate and placed on TEM grids for subsequent electron beam annealing.

Complicated regular change in lattice orientations are indicated by regular bend contour patterns, Figures 1a, 2a-c (main features presented earlier for both Se spherulites [2] and α-Fe₂O₃ [3]). It is the basis for the present combined studies, where the interrelation of crystal lattice orientation and relief of the crystal surface has been of prime interest.

AFM has been used to visualize (and obtain corresponding data by profiling): the spherulite and amorphous surrounding (Figure 1a-b); the macro relief of the crystal as a whole - usually for Se spherulites (about 20-50 µm in diameter) having form of a hat, lying upside down with maximal deflection around 0.5-1 µm (as also revealed by microinterferometry), and being flat for Fe₂O₃; micro relief of concentric zones corresponding to the regularly alternating zones of constant lattice orientations (Figure 1b, 2b); the details of surface variations inside different zones of spherulites and for surrounding amorphous material.

The central region with 2 poles, separated by a distance about 3 µm (P1, P2, Figure 2a) is studied for Se spherulite. Orientation [001] is almost normal to the film plane at the poles and is parallel to this plane in between (TEM data). It corresponds to AFM data: the poles are the lowest points of the crystal; an area including 2 poles has the form of a boat (width ~ 1.5 µm).

Concentric zones of different orientations and imperfection revealed by TEM are also seen in AFM, based on the variations in mean height (for Se, Figure 2b) and on the character of fibrous structure (for Fe₂O₃, Figure 2c) which differs for two types of spherulites. Both spherulites have also radial elongated nodes and hollows of different character revealed by AFM. Further analysis in relation with TEM observations is in progress.

References

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**Figure 1.** TEM (a) and AFM (b) images of Se spherulite grown in amorphous film. The magnified images for selected areas A and B are shown on the Figure 2, a, b correspondingly.

**Figure 2.** TEM (upper row) and AFM (lower row, images developed by standard methods to get aside relief details): central area including “poles” P1, P2 of Se spherulite (a), concentric and radial details of the structure and surface relief revealed for the same Se spherulite (b), structure and surface details for Fe$_2$O$_3$ spherulite (c).