Preparation of Bismuth Telluride Specimens for TEM

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Bismuth telluride (Bi₂Te₃) and its alloys are an important class of thermoelectric material. How well a thermoelectric material works is dependent on a variety of factors such as electrical and thermal conductivity and the Seebeck coefficient. Because the electrical and thermal conductivity can be affected by defects in the material, there is much interest in the basic understanding the microstructures of these materials [1]. There are challenges in preparation of TEM specimens from telluride-based materials due to their sensitivity to ion-milling artifacts. For instance, nanoscale defect arrangements have been shown to form in lead telluride (PbTe) specimens prepared under aggressive ion milling conditions if cooling and power density is not suitably controlled [2].

In this presentation we discuss methods and conditions for preparing TEM specimens of Bi₂Te₃ specimens considering both ion-milling and electropolishing techniques. In all cases TEM specimens were mechanically pre-thinned using conventional mechanical dimpling and polishing techniques prior to final thinning to electron transparency. The ion-milled specimens were prepared with Ar⁺ ion sputtering using a Fischione Model 1010 ion mill with LN cooling. The electropolished specimens were prepared using a Fischione Model 120 electropolisher and an electrolyte consisting of 53% water, 38% glycerol, 5% sodium hydroxide, and 4% tartaric acid. The electrolyte was set in an ice bath and cooled to 2° C, and electropolished at 25V and 35mA.

Figures 1 and 2 show dark-field TEM micrographs comparing ion milled and electropolished Bi₂Te₃ specimens. The ion milled sample shows fine-scale contrast modulations, or striations, and are often observed in TEM specimens of Bi₂Te₃ and its alloys prepared using energetic methods such as ion milling and FIB. Modulations typically have periodicities on the order of 10 to 20 nm and exhibit strong diffraction contrast that can be imaged in dark-field TEM using, for instance, {1,0,-1,5} type reflections. Similar contrast in ion-milled Bi₂Te₃ has been reported in the literature and interpreted as resulting from intrinsic periodic strain in the Bi₂Te₃ structure [3,4]. In contrast the electropolished specimen shows no sign of these features, suggesting that striations are an artifact of the ion milling process.

References:
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Figure 1. Dark Field TEM images from an Ion milled specimen prepared under aggressive conditions show fine-scale contrast modulations, or striations, with periodicities on the order of 10 to 20 nm. Specimen imaged near a [5, -5, 1] zone under strong-beam dark field conditions.

Figure 2. The fine scale striations are not observed in electropolished specimen. Specimen imaged near a [5, -5, 1] zone under strong-beam dark field conditions.