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IT-2-P-1677 Preparation of thin film specimen by Cryo Ion Slicer for TEM cross-section (XTEM) observation

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An essential part of research in thin film fabrication is the microstructural analyses like morphology, grain distribution, texture, thickness of the layers and orientation of film structure. For such characterization, cross-sectional transmission electron microscopy (XTEM) is a very essential tool for the study of structure, phase, defects and interfaces. For such analyses, it is necessary to make the film electron transparent in a direction perpendicular to the interfaces. One of the methods is cryo ion slicing (from JEOL) with specific sample preparation procedure different from the PIPS from Gatan. The preparation of cross-sectional specimens with ion slicer are usually done by fabricating a sandwich structure (Thin film/Glue/Cover glass) and subsequently thinning it to transparent for electrons (thickness of the order of <50 nm for TEM and <10nm for HR-TEM). The cross-section specimen preparation is generally time consuming, specimen dependent and consequently a trial and error method. But the features of XTEM observations are in results more informative and necessary in addition with the other methods of the observations, i.e. XRD, optical studies or micrographs of scratched samples from thin films.

The present work describes the preparation of thin film specimen, includes mechanical (pre-preparation) and Ar⁺ ion slicing (milling). It was successfully used for the preparation of a-Si:H/a-SiO₂, nc-Si/a-SiO₂ and ZnO thin film specimens for transmission microscopic analyses. The pre-preparation of sample for ion milling consists of cutting samples by diamond disc using low speed saw cutter (Buehler IsoMet) and mechanically thinning using JEOL Handy Lap to get the specimen dimension 2.5mm×500µm×100µm with plan-parallel to the surfaces. The ion slicing was carried out using JEOL IB-09060CIS Cryo Ion Slicer using Ar gas of purity 99.9999%. Finally thin regions of range from 100nm to 10nm were achieved over the thin film layers. A very thin cross-section of ~10nm could be used to obtain high resolution TEM images.

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