Thin Film Characterization Utilizing Broad Ion Beam Specimen Preparation and FESEM

Natasha Erdman and Noriyuki Inoue

JEOL USA Inc, Peabody, MA 01960

Examination of materials cross sections often provides essential information about the crystal structure, layer or film thicknesses, existence of voids or cracks and other properties that might impact materials performance and reliability. Cross-sectional observation is especially essential in thin film technology, to examine layer thickness, deposition integrity (voids/adhesion), as well as film growth and crystallographic orientation. Currently various methods are used to prepare specimen cross sections for scanning electron microscope (SEM) observation. Mechanical methods of cutting and polishing are widely used, particularly for metallographic sample preparation. However, mechanical polishing presents several problems: a) in composite materials with different hardness values, the polished surface becomes uneven as the softer components are cut faster and more easily than the harder components; b) in soft materials, particles of hard abrasive can be buried in the material being polished; c) in materials with voids, the edges of the voids can stretch and deform; e) for metals, due to the strain caused by mechanical polishing on the polished surface, the information about the crystal structure by means of electron back-scatter diffraction (EBSD) becomes difficult or impossible to obtain; f) fine features like hairline cracks and small voids can get smeared shut and will not be recognized as such.

This paper presents utilization of broad ion beam instrument (JEOL CP polisher) for cross-sectional preparation of various thin film/substrate combinations. This table-top instrument (Fig 1) utilizes Ar ion beam to produce large area cross-sections of materials, with the ability to employ cryogenic (LN2) temperatures to address beam sensitive and eutectic metal systems. Moreover, the use of FE-SEM equipped with in-lens detectors and high sensitivity backscatter detector allows observation of the resulting samples to investigate nanoscale features, including voids, grain boundaries and layers. Additionally, EDS and EBSD can be utilized to provide additional characterization of the thin film specimens in terms of compositional variations and crystallographic orientation.

We will present examples of Zn thin films - depending on the film composition these may require cryogenic preparation to preserve film integrity (Fig 2). Additionally, we will present examples of other materials - anodized films, evaporated metal thin films, solar films, etc.

References:

1. N. Erdman, R. Campbell, S. Asahina, Microscopy Today, 2006
2. N. Erdman, K. Ogura, R. Campbell, Microscopy and Microanalysis 17 (S2), 388-389, 2011

Figures:

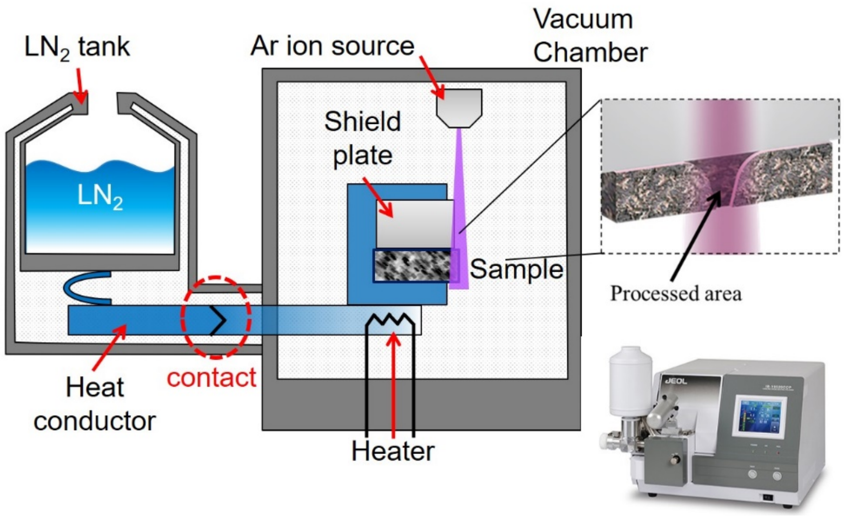

Figure 1 – Schematic of cooling broad ion beam polisher (JEOL IB-19520CCP)

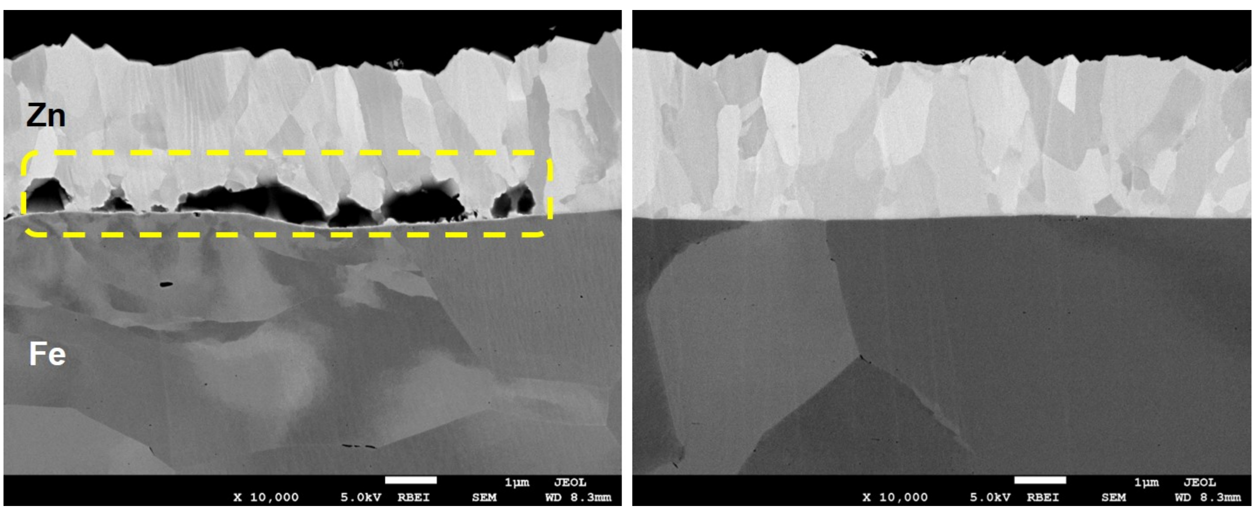

Figure 2 – Example of Zn coating on steel prepared at RT (left) and using cooling (right). The image shows preserved thin-film/substrate interface and crystallographic integrity.