Evaluation of cross-sections fabricated by FIB with a cooling stage for low melting point metals

Hideki MATSUSHIMA¹, Yuichiro OOHORI¹, Noriaki MIZUNO¹, Yusuke KAGAYA¹, Akira TAKISHITA¹, Hideo NISHIOKA¹ and Toshiaki SUZUKI¹

¹ IB Business Unit, JEOL Ltd., 3-1-2, Musashino, Akishima, Tokyo 196-8558 JAPAN.

Electron microscopes, such as scanning and transmission electron microscopes (SEM and TEM) are indispensable to evaluate metals and alloys with low melting points. Therefore to reveal actual topographic and analytical information of them, it is essential to choose and develop a suitable process of sample preparation. A gallium (Ga) focused ion beam (FIB) technique [1] is widely used for sample preparation of cross sectional observation. However, ion beam induced damages, such as ion implantation in the sample and thermal damage from high energy charged particles should be carefully considered [2]. Low melting point metals, such as lead solder are fabricated by FIB [3, 4], but the damage caused by charged particles has not been evaluated and discussed insufficiently so far. Here, a lead solder is milled by a Ga ion beam at -165 °C for cross-section observation and then SEM observation is carried out using a backscattered electron detector. Figure 1 shows the comparison between cross-sections fabricated by low and room temperature milling procedures. In the case of the room temperature milling, many cracks are induced at grain boundaries, while the low temperature milling gives a smooth surface without any cracks which produces a clear channeling contrast. Based on this result, the authors experimentally investigated the beam induced damage caused by the FIB milling and verified an advantage of low temperature milling for low melting point metals. Some interesting results obtained from this work will be reported.

A lead solder and a galvanized sheet iron were used as samples for the present study. The multi-beam system: JIB-4610F (JEOL Ltd.) used in this study was equipped with a cooling stage (PP3010T by Quorum), as shown in Fig. 2. All the sample preparations for cross-sections were made under the same condition, i.e., at the same accelerating voltage of 30kV with an ion beam current of 3nA for fine milling.

Using a lead solder, we estimated a proper temperature below which high quality sections can be prepared without artifacts by the ion milling. The ion milling was carried out under the condition at different temperatures as described previously. Figure 3 shows back-scattered electron (BE) images of cross-sections prepared at three different temperatures taken by the SEM. At 25 °C, cracks along two phase boundaries were clearly observed as well as ingested tin phases in lead. At -50 °C, the cracks were faintly observed as indicated by red arrows, whereas no artifacts such as cracks were observed at -75 °C. The present observation confirmed that a high quality section of the lead solder can be prepared below -75 °C. Figure 4 shows BE images of cross-sections of a galvanized sheet iron prepared at three different temperatures. At 25 °C, etched contrasts were observed in the galvanized sheet. At -50 °C, a flat cross-section of the galvanized sheet was made, and a channeling contrast was not observed. At -100°C, a flat cross-section was made and a channeling contrast was clearly observed. So, the ion milling below -100 °C is necessary to prepare a high quality cross-section of the galvanized sheet iron.

Ion milling at low temperatures was confirmed to be very useful for sample preparation of a high quality cross-section of low melting point metals and alloys. The multi-beam system equipped with a low temperature sample stage is especially useful for observation of samples involving microstructures which change in structure even at low temperatures. In order to suppress such structural change during the ion milling, the observation as well as the sample preparation can be performed at sufficiently low temperatures in the multi beam system.
References:

Figure 1. BE images of cross sections of a lead solder prepared by ion milling at 25°C and -165 °C.

Figure 2. A schematic diagram of the FIB-SEM mounted with the cooling stage.

Figure 3. BE images of cross sections of a lead solder prepared by ion milling at 25, -50 and -75°C.

Figure 4. BE images of cross sections of a galvanized sheet iron prepared by ion milling at 25, -50 and -100°C.