

High Quality Cross Sections of Low Melting Point Samples Prepared with Cryo Ion Slicer – Broad Ar Ion Beam Milling Apparatus with a Newly Developed Specimen Cooling Unit

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Introduction

Ion Slicer (IS) is an instrument used to prepare TEM lamellas and SEM cross-sections by employing an Ar broad ion beam. The IS has been getting quite popular in TEM and SEM laboratories because of its ease of use and high quality results. However, it is difficult to mill low melting point materials by the Ar broad ion beam because of thermal damage during the milling. Therefore, we have developed a specimen cooling unit for the IS. This cooling unit keeps specimen at low temperature during the ion milling to avoid the thermal damage. We named this system Cryo Ion Slicer(CIS).

About the IS

Figure 1 shows the arrangement of the Ar ion gun, the shielding belt and the specimen of the basic IS. The specimen has to be prepared into a small block with the size of 2.8 mm (long) × 1.0 mm (wide) × 0.1 mm (thick) before the Ar ion beam milling. Once the prepared specimen is placed in the IS, the shield belt comes down automatically right on the specimen. By rocking the Ar ion beam up to $\pm 6^\circ$ continuously during the milling, both sides of the specimen will be milled into a thin foil for TEM observation. To make a cross-sectional specimen for SEM observation, the rocking angle is set to 0° , so that the Ar ion beam comes straight down to the top of the shield belt. Then the specimen becomes the same thickness as the shield belt. Both sides of specimen under the shield belt will be cross-sectional planes for SEM observation.

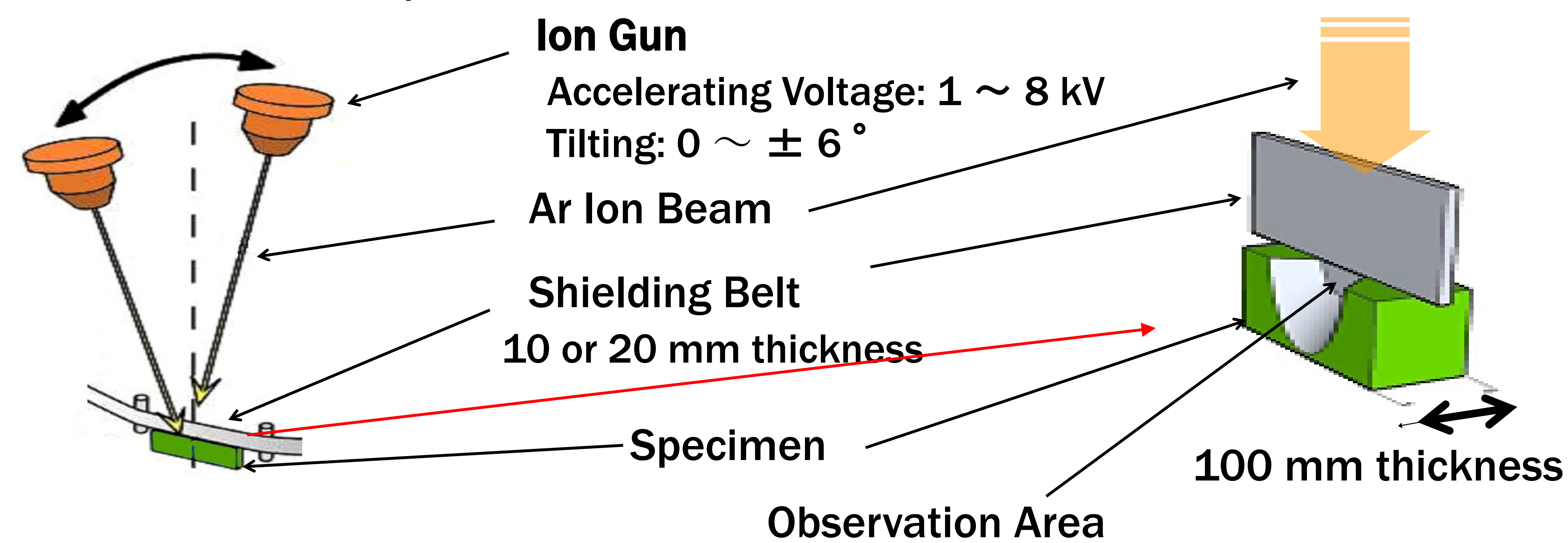


Fig. 1 A schematic illustration of the principle of the Ion Slicer

About the CIS

Figure 2 shows an external view of the CIS. Figure 3(a) and (b) show the cooling mechanism of the CIS. The specimen holder is cooled down to -120°C by liquid nitrogen when the cooling conductor is inserted. It takes 60 minutes to reach -120°C . The cooling conductor can be pulled out, and the specimen holder can be heated up when the specimen exchange is required. It takes 30 minutes to reach the room temperature. Therefore, we can exchange the specimen with liquid nitrogen in the tank.



Fig. 2 Cryo Ion Slicer

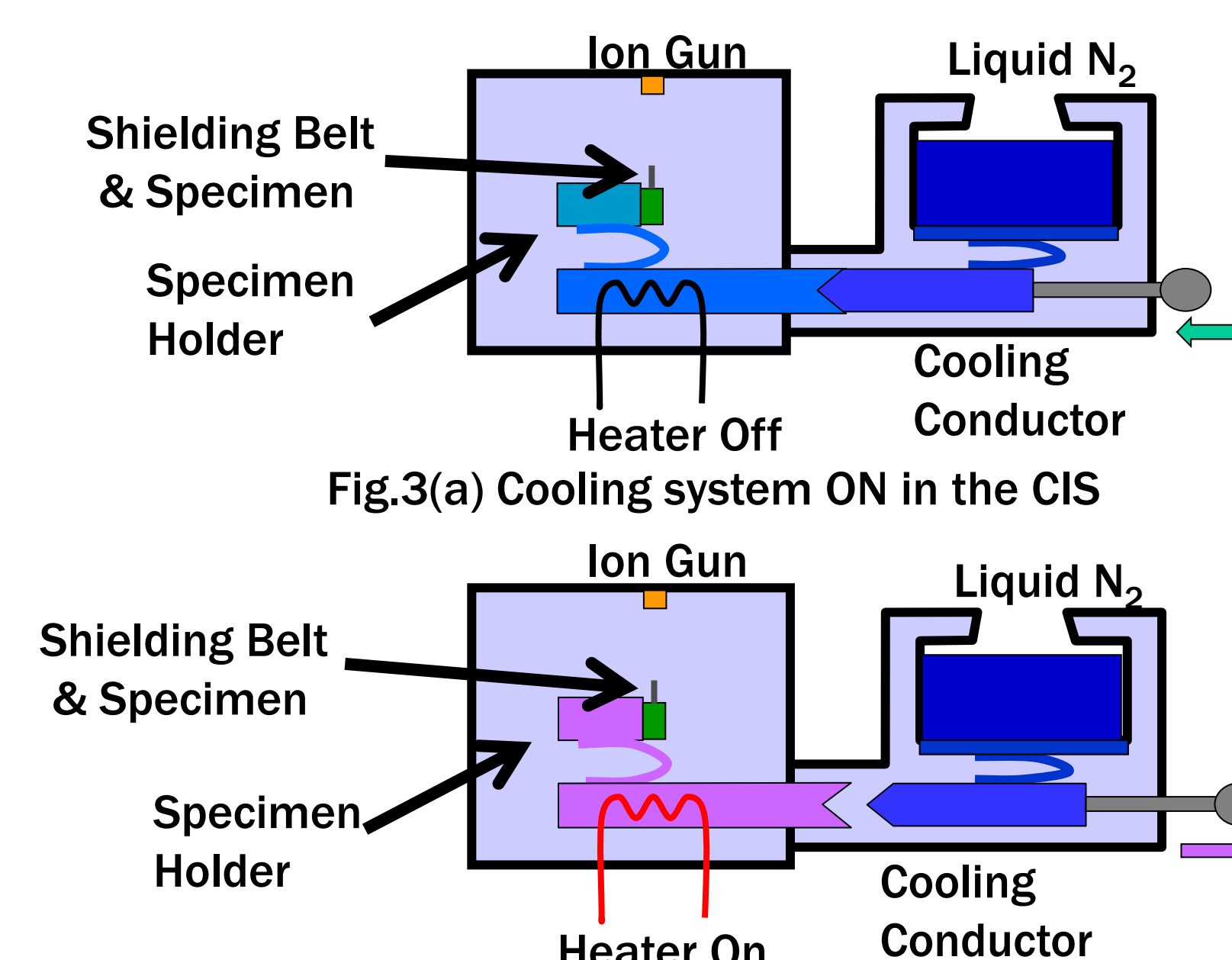


Fig.3(b)
Cooling system OFF in the CIS for specimen exchange

Result

Wood's metal (Melting point : about 70°C)

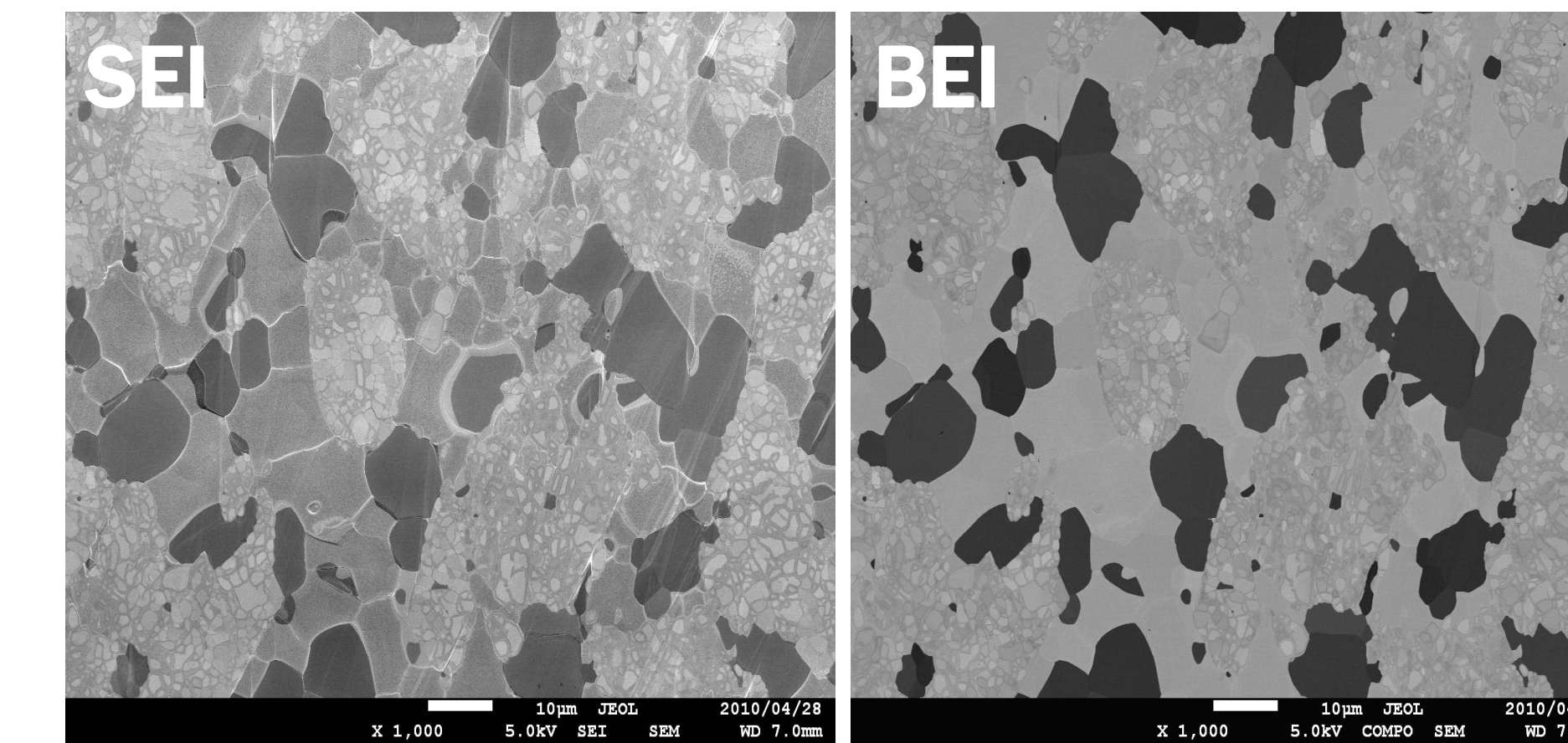


Fig.4(a) A cross section of wood's metal with CIS
left: secondary electron image(SEI) right: backscattered electron image(BEI)

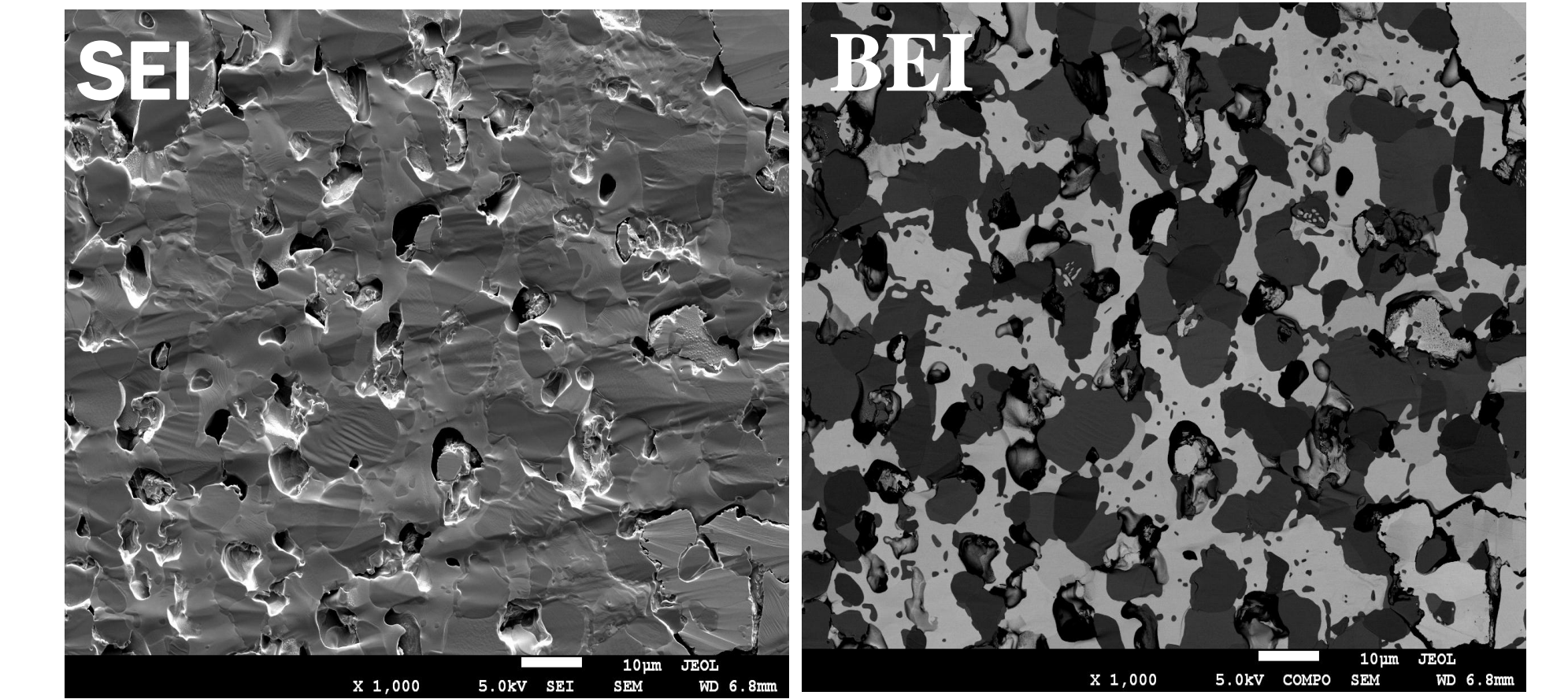


Fig.4(b) A cross section of wood's metal with IS

The melting point of wood's metal is about 70°C , and it is a eutectic alloy of Bi (50%wt), Pb (26.7%wt), Sn (13.3%wt) and Cd (10%wt). It is well known that this specimen consists of four phases. Two phases that are Bi and Pb rich are brighter than the other two phases that are Sn and Cd rich in BE image in SEM. After the CIS milling, four phases are clearly observed in Figure 4(a). However, after the milling without specimen cooling, there are a lot of cracks on the surface, and the four phases are not easily observed (Figure 4(b)).

Pb-Sn solder on printed circle board (Melting point : 183°C)

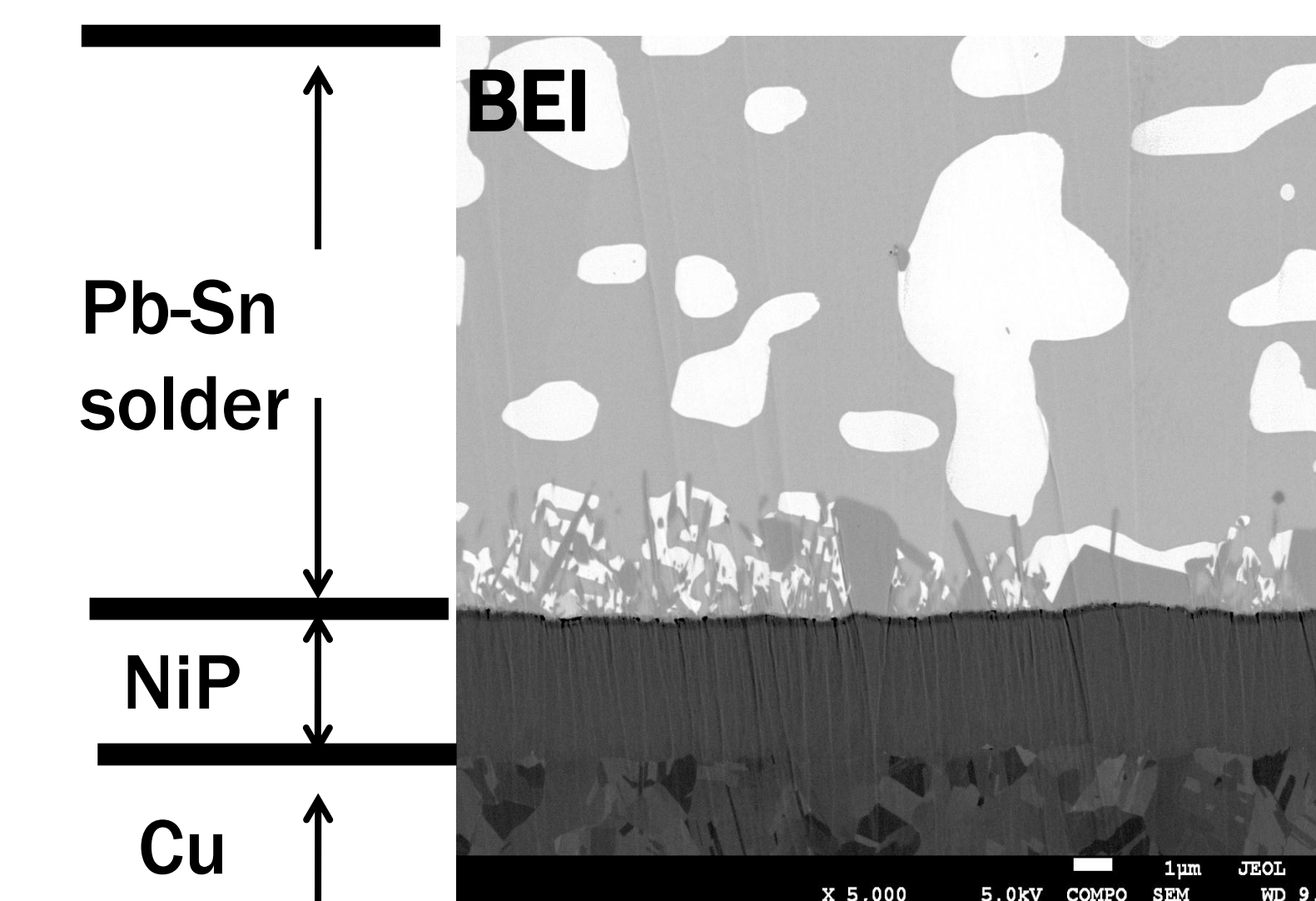


Fig.5(a) A cross section of Pb-Sn solder with CIS

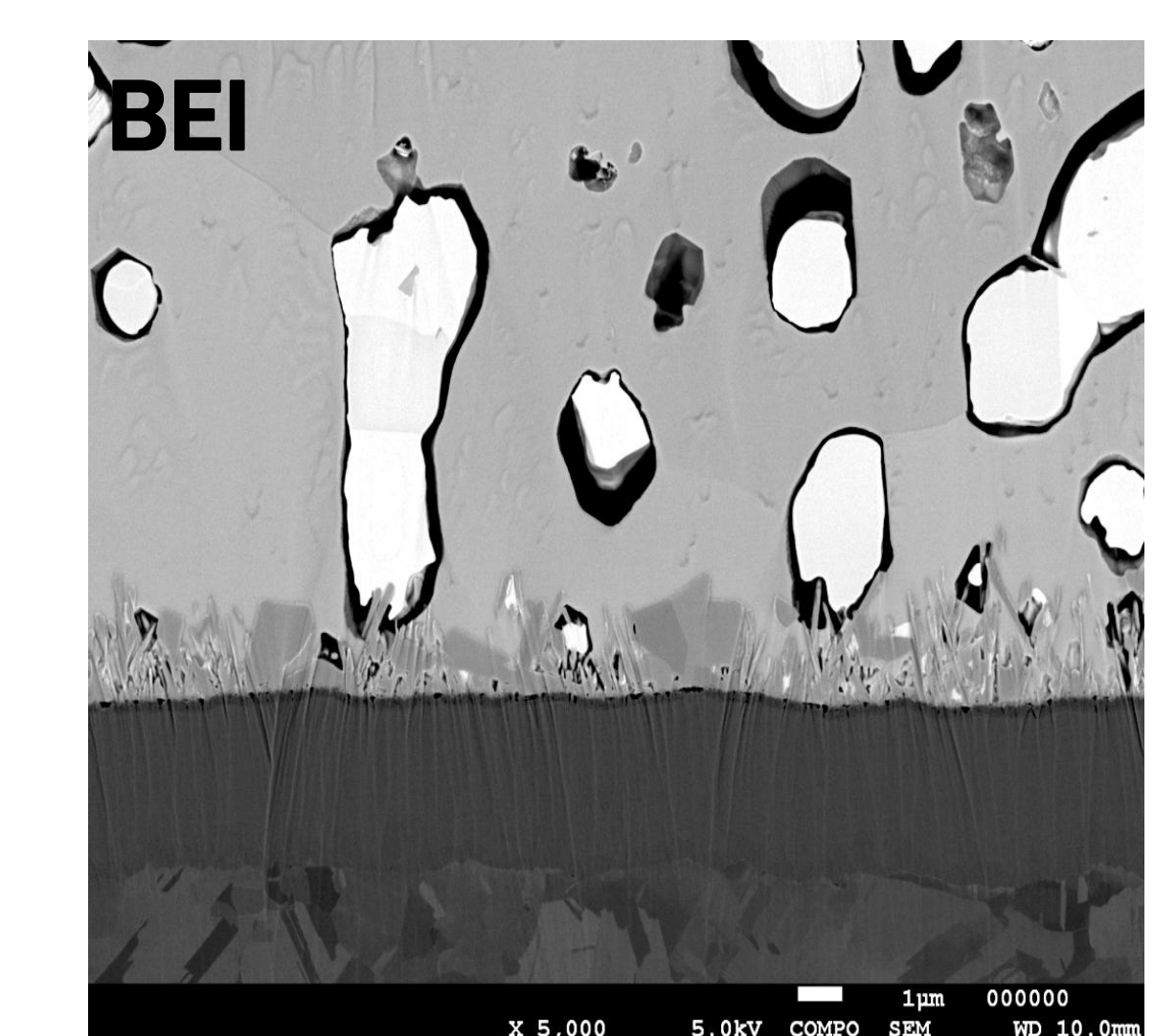


Fig.5(b) A cross section of Pb-Sn solder with IS

The melting point of Pb-Sn solder is 183°C . There are two phases. Pb rich phase is brighter than Sn rich phase in backscattered electron image. The CIS milling result shows a very smooth surface and good contrast of two phases. However, the result without specimen cooling shows cracks around Pb phase.

Galvanized sheet iron (Melting point : 419.5°C)

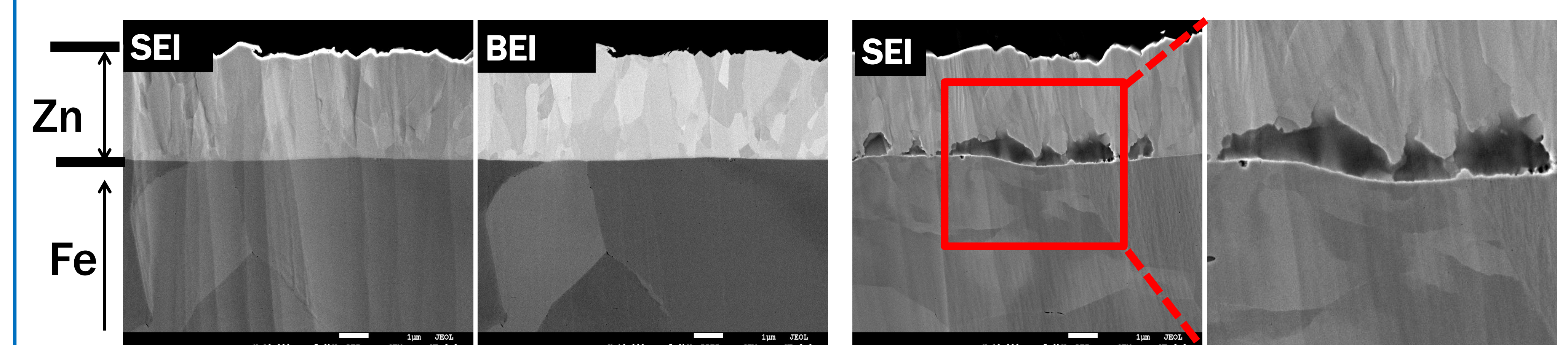


Fig.6(a)

Fig.6(b)

A cross section of galvanized sheet iron with CIS A cross section of galvanized sheet iron with IS

The melting point of Zn is 419.5°C , and is higher than other two materials described before. However, there was some difference between with and without specimen cooling during the milling. In the case of without the specimen cooling, we found out crack in Zn region right on the interface with Fe.

Conclusion

We have tried to make cross-sectional planes of low melting point materials by employing the CIS, which can keep the specimen at low temperature during the Ar ion milling. We found out that the CIS allows to make cross-sectional planes of low melting point materials without thermal damage during the Ar ion milling.