Cross Section Specimen Preparation Device Using Argon Ion Beam for SEM

-Cross Section Polisher (CP) SM-09010-

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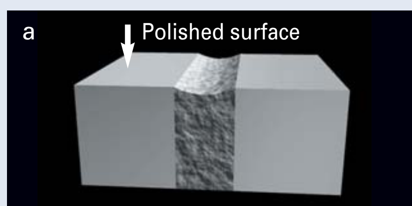
Scanning Electron Microscopes (SEMs) have been used for various applications, such as research and development and failure analysis. There are many cases where not only observation of a specimen surface – but also observation of a cross section – is important. Preparation of a cross section depends on the specimen structure, observation purpose, and materials. Various preparation methods are put into practice: cutting, mechanical polishing, microtome, and FIB (Focused Ion Beam) are the major methods. In this discussion, we evaluate a new cross section specimen preparation method using an argon ion beam (hereinafter called the Cross-section Polishing or CP method). We have found that this method is extremely useful for observation of layer structures, interfaces, and crystalline structures of metals, ceramics, and composites. Here, we introduce examples of applications to various types of specimens.

Introduction

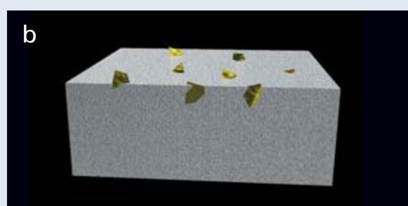
Observation of cross sections of complex materials provides important information about the size of crystal grains, the layer structures, the film thicknesses of reaction layers and the existence of voids. In cross sectional observation, mechanical polishing is generally used to prepare a specimen. However, it is difficult to polish a junction of materials with different hardnesses or a reaction layer formed in the junction into a flat mirror surface by mechanical polishing. Mechanical polishing has several problems as shown in **Fig. 1**: a) if it is applied to materials having different hardnesses, the polished surface becomes uneven as the soft portion is cut faster and more easily, b) if it is applied to soft materials, there are some cases where pieces of hard polish are buried in the material being polished, c) for soft materials, if the material has a void, the material around the void stretches and deforms, d) if the material has a void, pieces of polish get stuck in the void, e) for metals, due to distortion caused by mechanical polishing on the polished surface, the information about the crystal becomes difficult to obtain.

The FIB method is one of the methods known to resolve these problems, and its application in various fields has been reported. The FIB method is effective for cross section specimen preparation of micro area and thin film specimen preparation, however, it is not effective for preparation of a wide surface area of a specimen.

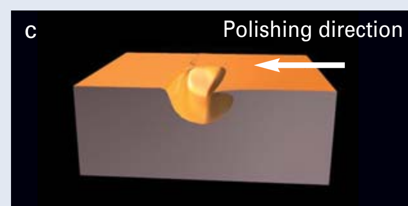
The CP method that we are introducing here does not incur those problems of mechanical polishing mentioned before, and it enables one to prepare a wider area of a specimen than the FIB method. In addition, required skill is minimal compared with other specimen preparation methods, making it simple for anyone to prepare a cross section specimen.



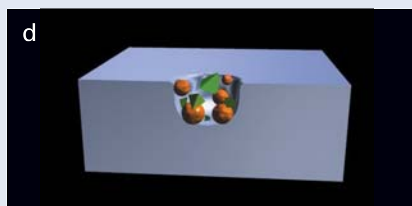
a) On material with different hardnesses, the polished surface becomes uneven as the soft portion is cut faster and easier.



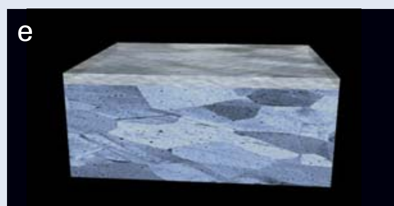
b) On soft material, pieces of hard polisher can be buried in the material being polished.



c) On soft material with a void, the material around the void stretches and deforms.



d) On material with a void, pieces of polisher get stuck in the void.



e) On metals, due to distortion caused by mechanical polishing on the polished surface, information about the crystal becomes difficult to obtain.

Fig. 1 Problems associated with the mechanical polishing

Instrument Outline

The appearance of the instrument is shown in **Fig. 2**, and the principal specifications are listed in **Table 1**. Because it employs the Penning type argon ion gun which has a long life, it can obtain high current density. It enables one to prepare cross sections consistently. The accelerating voltage is changeable between 2 and 6 kV and the use of low-energy argon ions minimizes damage to the specimen. A turbo-molecular pump and a rotary pump have been employed for the evacuation system and the instrument can be placed wherever the space of an office desk is available.

Features of the Cross Section Polisher

- Provides a good cross section of even a composite of soft and hard materials.
- Minimizes distortion of the polished surface, enabling one to observe grain contrast clearly.
- Polishes an area several hundred micrometers in width, much larger than the FIB method can polish.
- As it is not mechanical polishing, polish never embeds in the polished surface.
- Low running cost
- Ease of operation
- Eco-friendly, free of drainage and waste liquids

Application Examples

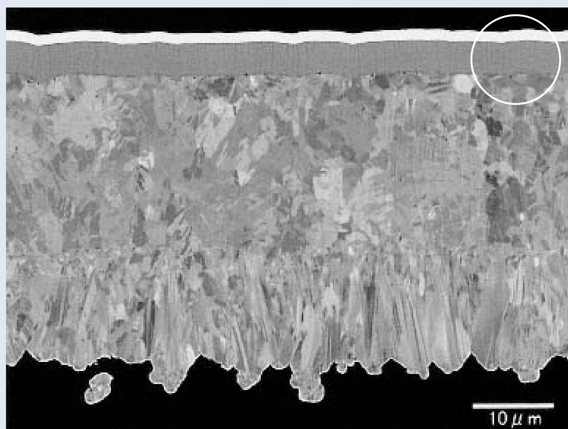
Card Edge Connector

Fig. 3 shows the cross section SEM micrograph of a card edge connector prepared by the CP method. The cross section prepared by mechanical polishing is shown in **Fig. 4** for comparison. In Fig. 3, the specimen consists of resin/Cu (first layer)/Cu (second layer)/NiP/Au from the bottom in order (left), and the surface

Fig. 2 Cross section polisher (CP) SM-09010

Table 1 Principal specifications

Ion gun	Penning type Argon ion gun
Accelerating voltage	2 to 6 kV
Ion beam diameter	500 μm (FWHM)
Milling rate	0.3 μm/min (6kV, silicon, 100μm from edge)
Maximum specimen size	11mm(W)×10mm(D)×2mm(H)
Specimen stage	X : ±3mm, Y : ±3mm
Specimen alignment	±5°
Vacuum system	TMP, RP



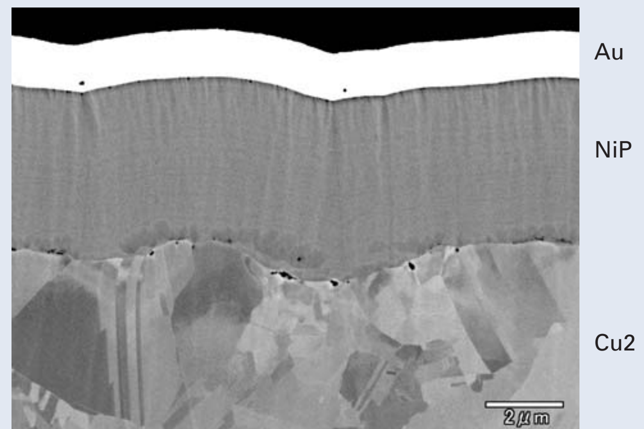
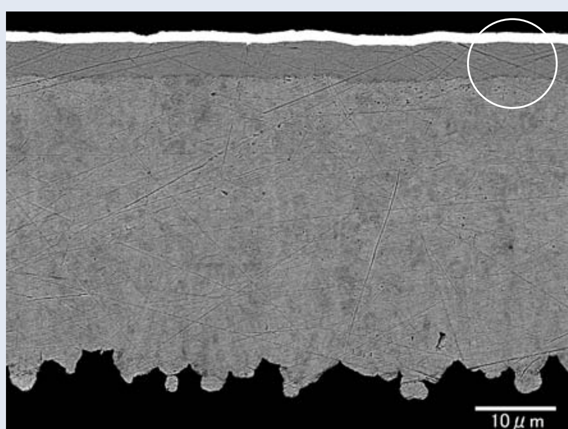


Fig. 3 CP method (Backscattered electron image)



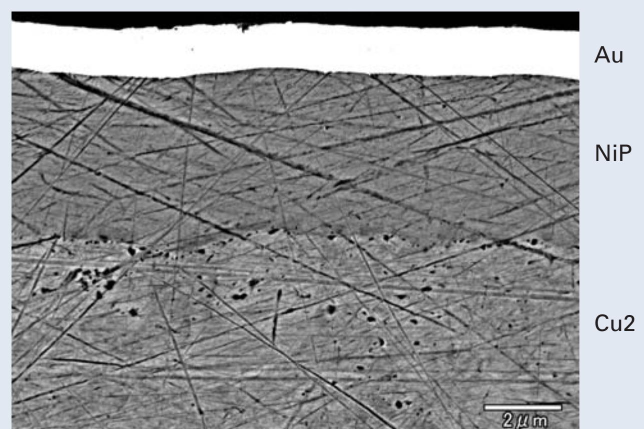


Fig. 4 Mechanical polish (Backscattered electron image)

layers are composed of soft metals of Au and Cu, and hard metal NiP (right). With the CP method, 1) grain contrast in the Cu portion, 2) lateral stripes arising during NiP film formation can be clearly observed. On the other hand, the specimen produced by mechanical polishing in Fig. 4 fails to show good results due to polish damage.

Bonded Gold Wire

A cross section micrograph of a gold wire bonded on a gold plated pad is shown in **Fig. 5.** The condition of the bonded part, film thickness, voids, and grain contrast can be observed. **Fig. 6** shows a cross section and lateral section of the same gold wire. We can observe that the size of the crystal grains varies both in the cross section and lateral section micrographs.

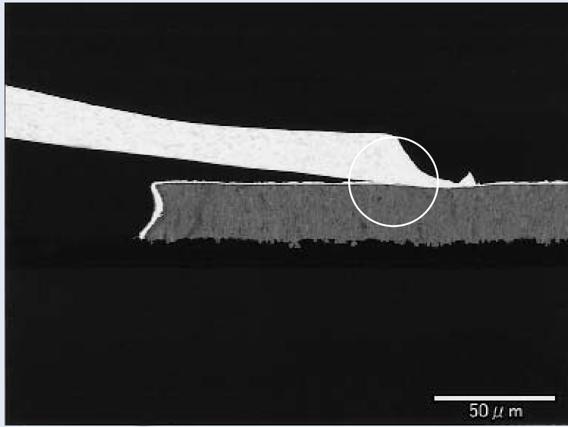
Flexible Printed Circuit

A cross section SEM micrograph of a flexible printed circuit is shown in **Fig. 7**. The specimen consists of resin/Cu/Ni/solder. a) is a good cross section specimen about 350 μm wide, and we can observe a void in the solder in it. b) shows the clear difference between crystal grains of the Cu and Ni portions. The CP method provides a good result, even if the specimen is a composite of resin and metals.

IC Analysis

Fig. 8 shows a cross section SEM micrograph of a molded IC. **Fig. 9** shows a magnified image and EDS maps of the Cu terminal connecting portion. On the Si substrate of the molded IC, various materials, such as SiO_2, SiN, Au, Sn, Al (wiring), and Cu (terminal), are laminated. It is seen that a good cross section is obtained even from a composite with different hardnesses.

Fig. 10 shows an X-ray spectrum from the area where EDS mapping was performed. As seen from the spectrum, the peak of the ion source, Ar, cannot be found. Of course,



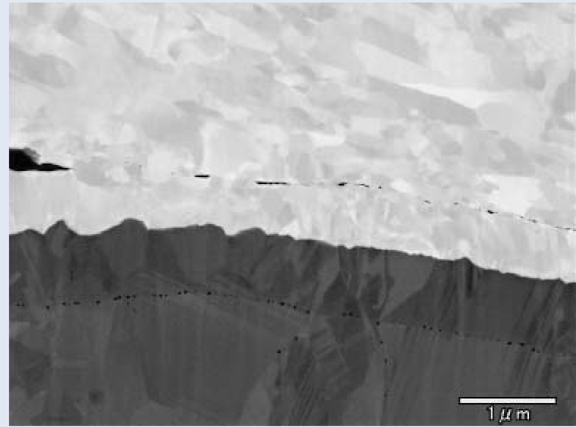
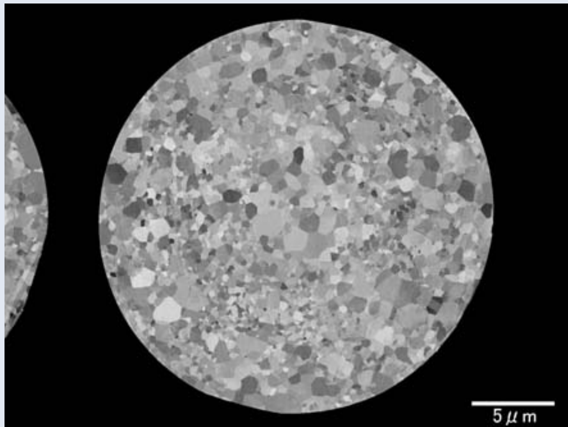


Fig. 5 Bonded gold wire (Backscattered electron image)



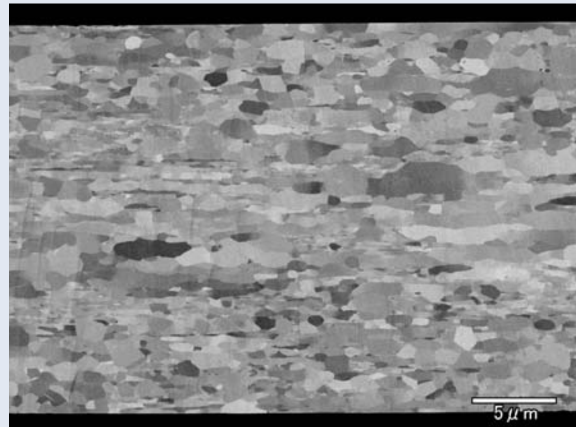
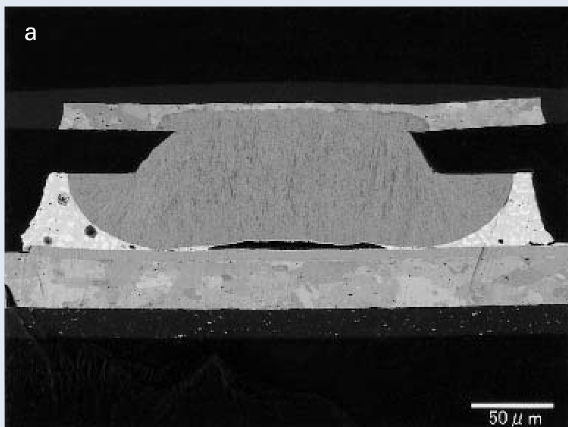


Fig. 6 Cross section and lateral section of the same gold wire (Backscattered electron image)



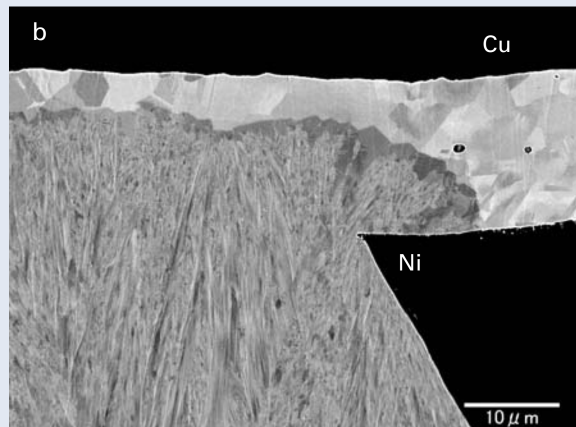


Fig. 7 Flexible printed circuit (Backscattered electron image)

depending on the constituent elements, the amount of Ar ion implantation varies, but this IC specimen has so little Ar ion implantation that we can disregard it. With the FIB method, there are cases where the ion source, Ga, is detected. It is necessary to take the overlap of peaks into consideration, depending on the analysis elements.

Cross Section Preparation of Ceramic Powder Specimen

Fig. 11 shows a SEM micrograph of the surface of a ceramic powder specimen, and **Fig. 12** shows a cross section SEM micrograph. The inner cavity that was not seen by the surface observation can be observed clearly in Fig. 12. For cross section observation of powder specimens, it is often important to observe the inner structure, the differences between specimens, and the core of the powder. In these cases, a cross section specimen that allows observation of a large number of powder grains is needed and so the CP method is considered to be extremely suitable.

Conclusion

The cross section specimen preparation method using an argon ion beam is one of the excellent methods among various cross section specimen preparation methods. It enables one to easily create cross sections of polymers, metals, ceramics, and composites with few artifacts. It is suitable for not only image observation but also EDS analysis, and it can prepare a cross section from a specimen area much wider than the FIB method can.

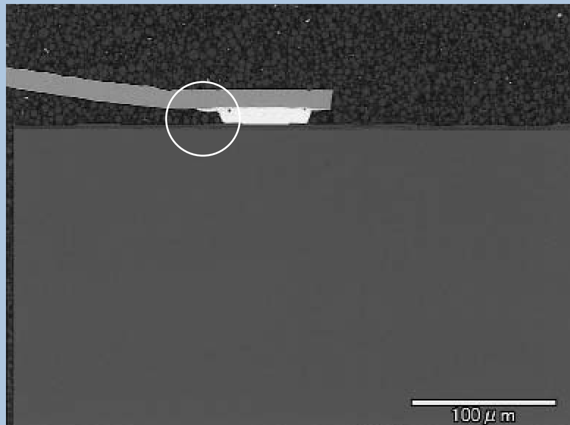
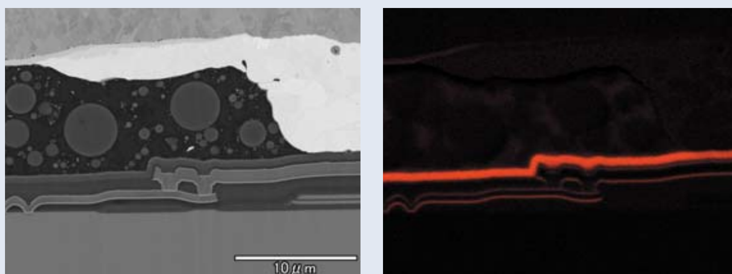
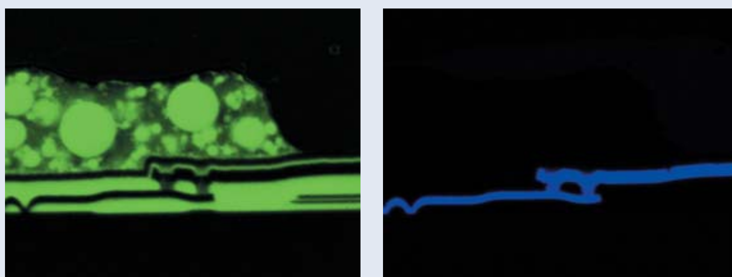


Fig. 8 Cross section of IC (Backscattered electron image)



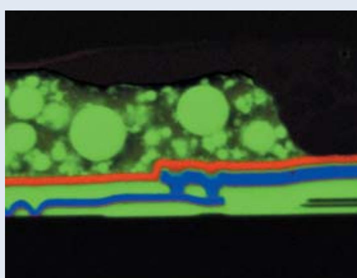
Backscattered electron image

Elemental map of nitrogen



Elemental map of oxygen

Elemental map of aluminum



Superimposed image of N, O, and Al

Fig.9 EDS elemental maps on Cu bonding region

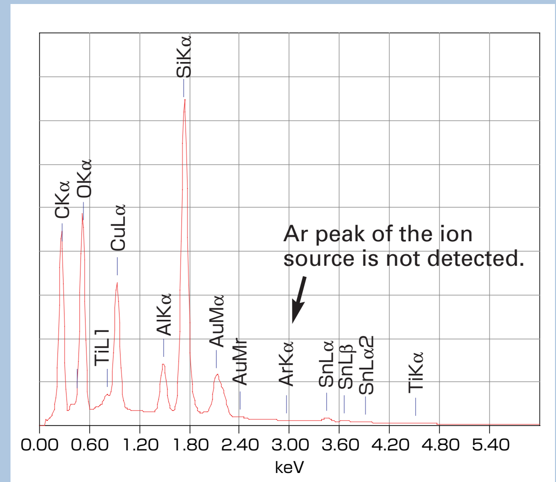


Fig. 10 Spectrum from the elemental mapping area.

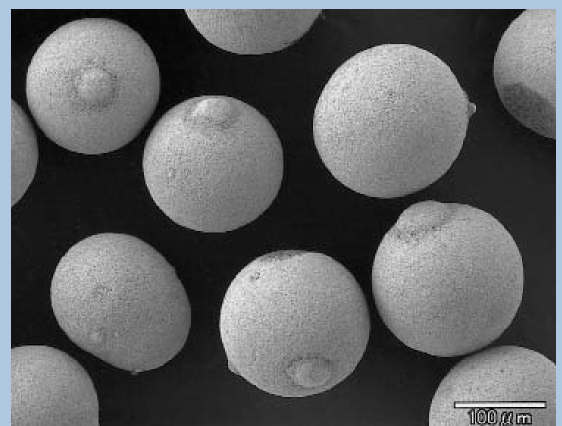


Fig. 11 Surface of ceramic powder (Secondary electron image)

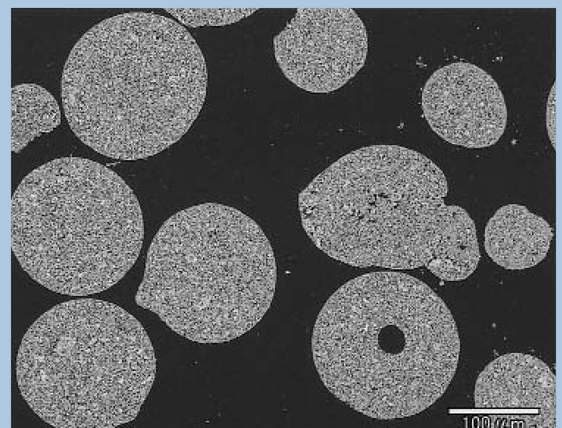


Fig. 12 Cross section of ceramic powder (Backscattered electron image)