JEOL Application Data Sheet Relevant Product: Sample Prep. Products (Cross-section Polisher)

Cross-sectioning of urethane rubber by an Ar broad ion beam for SEM imaging

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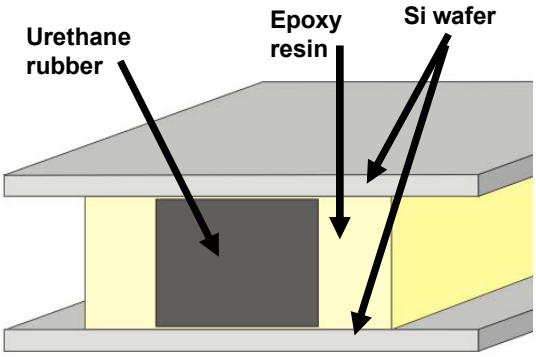
Introduction

Mechanical cross sectioning with a microtome is widely used to prepare high polymer materials for SEM imaging. However, fine skills are required to manipulate the microtome for high molecular weight polymer materials having a relatively low level of hardness, A new cross sectioning technique has recently been proposed using a broad argon ion beam, proving its effectiveness for a variety of materials.[1] The device, featuring an automated cross sectioning process, is easy to use and produces desired samples consistently. This technique, however, generates heat from irradiation during the milling process, and is therefore unfit for high molecular weight polymer materials with low heat resistance.[2] We have designed a technique to eliminate the effect of heat, in which a sample is sandwiched between silicone plates for etching, and will discuss its effectiveness below.

Experiment

Urethane rubber (heat resistance: approximately 100℃)
To enhance the thermal transmission, urethane rubber was cut into a segment about 0.5 mm thick, embedded in resin, sandwiched by Si plates at the top and bottom, and etched with Ar ions. For comparison, two more cross section samples were prepared by the CP without the sandwich technique and by the cryo microtome.

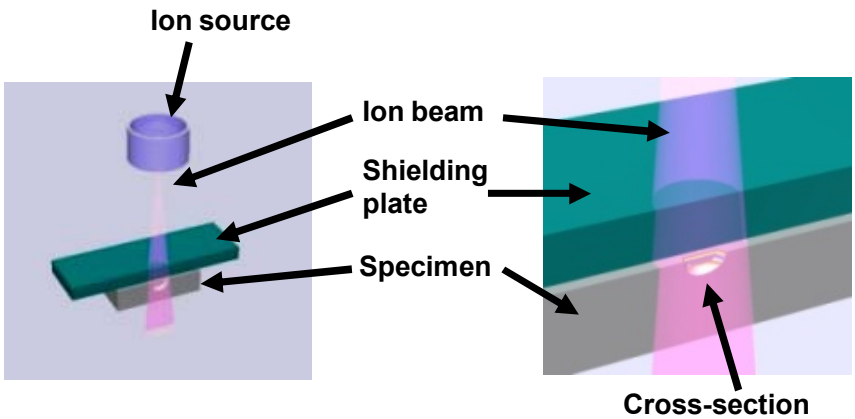


Schematic diagram

Instrument: What is a CP?

Sample preparation using a broad argon ion beam
- Milling by an argon beam shaped with a shielding plate -

A shielding plate is placed onto a sample to block argon ions to have a portion for milling protrude from the shielding plate. When the ion beam irradiates the protruding portion of the sample, it is etched, resulting in a smooth cross section.

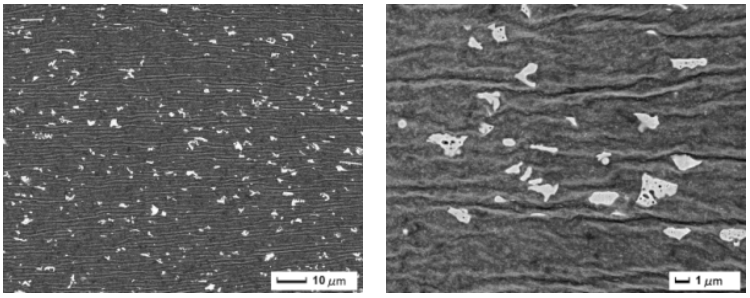


Schematic diagram of CP

Cross-section polisher

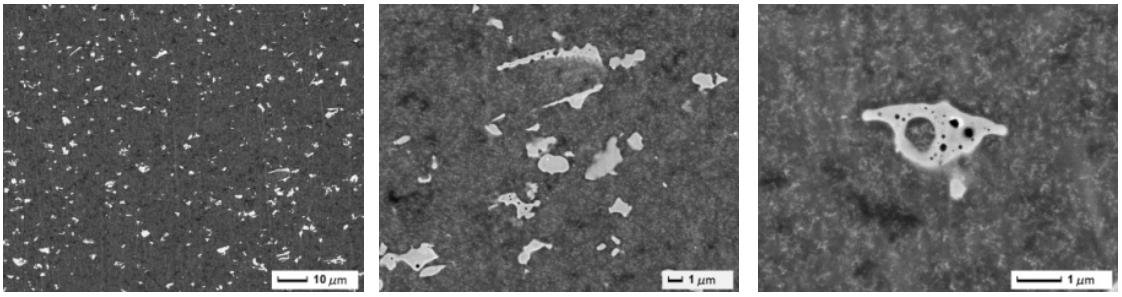
Results

CP normal method

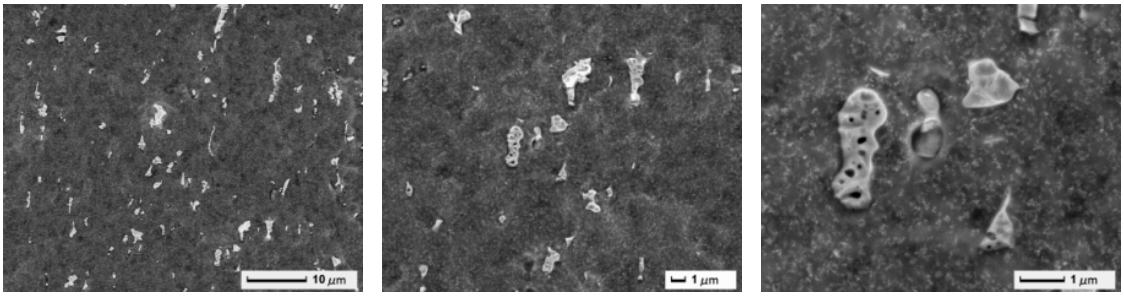


Imaging conditions
Instrument: JSM-6701F
Acc. voltage: 5 kV
Backscattered electron image

CP sandwich method



Cryo-microtome method



Images of urethane rubber cross-sections prepared by different methods

The photos above compare the results obtained by the different methods. The sample prepared by the CP normal method contracted due to the effect of heat. The sample prepared by the sandwich method suffered no contraction. The image quality of this sample is equal to that of the sample prepared by the cryo microtome, demonstrating that the sandwich method is effective for samples with low heat resistance.

Conclusion

The new technique to sandwich a sample between silicone plates was proven effective for samples with low thermal resistance, creating cross sections having fewer artifacts due to heat. The technique is expected to facilitate cross sectioning of high polymer molecular materials.

Reference

[1] M.Shibata: JEOL News, [39] (2004) p. 28.
[2] Satoru Mamiya, Toshiyasu Okui, and Ikumi Kimura: Proceedings of High Polymer Analysis Meeeting, [11] (2006) p. 47