An application to Ultra High Resolution SEM of Broad Argon Ion Beam Cross-sectional technique

Shunsuke Asahina[1*], Franck Charles[1], Sam M. Stevens [2,3] and Osamu Terasaki[2]

1. European Application Group, JEOL(Europe) SAS
Espace Claude Monet, 1, allée de Giverny, Croissy sur Seine 78290 FRANCE
2. Department of Physical, Inorganic & Structural Chemistry, Arrhenius Laboratory, Stockholm University, SWEDEN
3. Centre for Nanoporous Materials, School of Chemistry, University of Manchester, UNITED KINGDOM

* e-mail: asahina@jeol.fr

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Progress in the performance of an Ultra High Resolution SEM (UHR-SEM) has been enormous in recent years, the highlight of which is a readily achievable resolution of 1.0-1.5nm at 1kV which facilitates the observation of specimen surface fine structures. Of course, the cross sectional specimen, allowing observation of internal structure, is also attractive for the material scientist. In order to do this, we have to choose a technique for producing a cross section without producing artifacts limiting the observation of internal structures when using UHR-SEM.

Recently, JEOL have developed a new cross sectioning apparatus called The Cross Section Polisher (CP) [1,2]. The apparatus can also produce surfaces suitable for UHR-SEM observation and several other forms of surface analyses such as Auger Electron Microscopy or direct EBSD. The principle of this method is shown schematically in Fig.1. The specimen on the unmasked front side below a shield plate is milled to produce a cross section of the specimen by an Ar-ion beam [3]. Because the ion beam is irradiated parallel to the surface of the cross section, milling rate is almost independent of constituents in the specimen. Radiation damage due to the ion beam is also minimized. The result is a very high quality smooth section with minimal artifacts.

Electron Backscatter Diffraction (EBSD) is a powerful technique capable of characterizing a crystallographic orientation from extremely fine grained microstructures in a SEM. Electron Backscatter diffraction Patterns (EBSP's) are generated near the sample surface, typically from a depth in the range 10-50nm. Consequently, EBSD requires that the surface is adequately free from damage on a crystallographic scale, in order to generate useable EBSP's. Therefore, EBSD may be used as a measure of the quality of cross-sectioned surface. Fig.2 shows a EBSD patterns and high quality Backscattered Electron (BE) image on a cross section of diamond. This is excellent proof that the cross section quality is extremely good after CP processing.

Having shown that the CP process is gentle enough to retain original structures for diamond we are now presented with a harder challenge of making a cross-section of mesoporous silica SBA-15. Generally, SBA-15 is extremely fragile and very sensitive to both electron and ion beams. Thus, that is very difficult preparing cross-section with mechanical polishing and microtome or FIB. Fig 3 shows SEM image with CP cross section (A) and TEM image (B) of SBA-15 with spherical morphology. Both images clearly display mesoporous channels. Whilst TEM provides a higher resolution projection image, UHR-SEM provides a cross section. Also, unlike TEM, crushing of large crystals is not required allowing full use of SEM's ability to observe fine structure over a relatively large area. A

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combined TEM and UHR-SEM study of these materials gives information in both cross-section and projected forms as well as high resolution with a large sense of perspective.

The combination both CP and UHR-SEM is very unique and powerful analytical method to observe original fine cross sectional structures.

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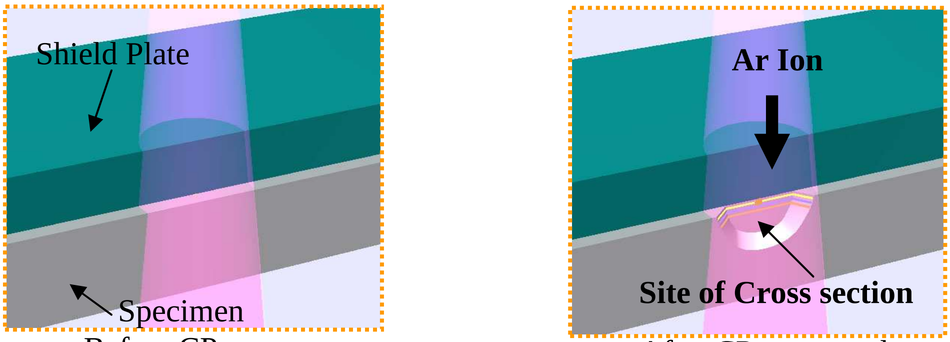

Before CP process

After CP processed

Fig.1 Principle of Cross Section Polisher

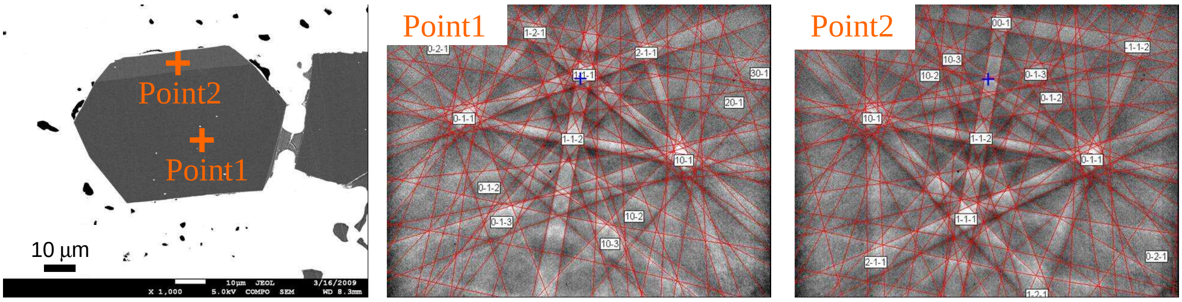

Fig.2 backscattered electron (BE) image and EBSD patterns from a Diamond cross section

The difference of BE contrast is showing different orientation. Actually, We obtained different EBSD pattern between point 1 and point 2.

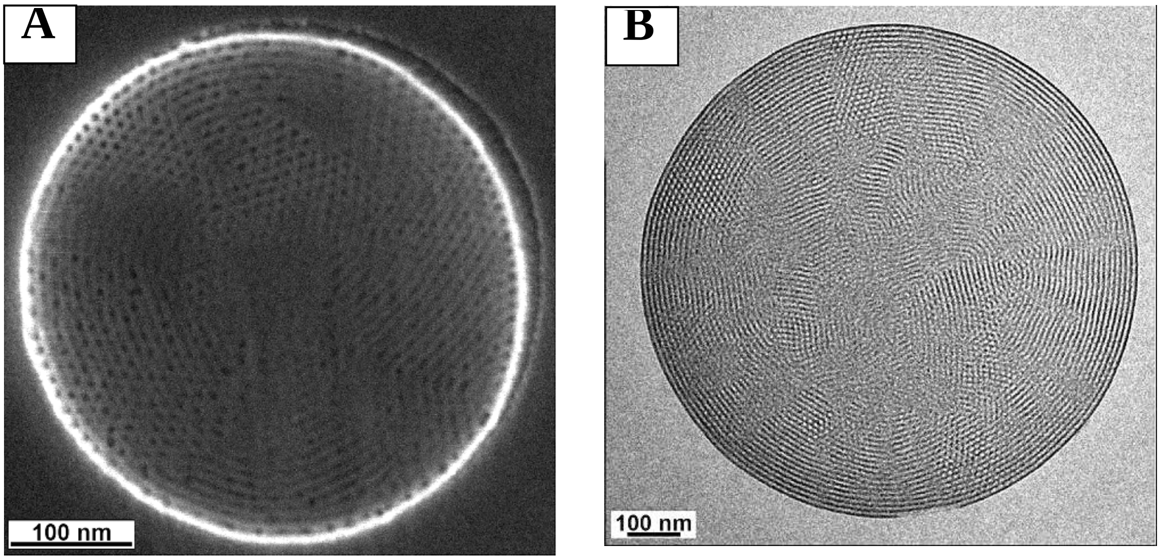

Fig .3 SEM image from the specimen prepared CP cross sectioning (A) and TEM image (B) of SBA-15