Preparation of Cross Sections Utilizing an Argon Beam System

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Figure 1: Ilion+® Cross Sectioning System

Introduction

There are multiple ways to make cross sections of samples. Focused Ga Ion Beam Systems (FIB) offers many advantages over mechanical and chemical preparation techniques for making cross sections. The major advantages of FIB's for cross section preparation is its weak sensitivity to material differences and the ability to precisely position the beam for the location of the cross section . However, FIB's are expensive and not perfect. They may induce some mechanical damage on the nano-scale. Additionally, Ga may chemically interact with the material altering the measured composition of the interface.

An alternative technique utilizes a collimated argon beam. Systems have been commercially available for approximately six years. Gatan, Inc. has recently introduced the Ilion+® which builds upon the expertise within Gatan of using argon beams, as well as addressing limitations of the current commercial systems.

Key Features

The Ilion+® System shown in Figure 1 has the following unique features :

- Two ion guns to Improve uniformity of the cross section.
- The ability to tilt the guns to mill into the cross section and provide depth profiling.
- The sample/blade is an integral unit that is aligned and mounted outside of the vacuum system. This improves the alignment of the blade to the area of interest and permits the sample/blade unit to be removed, viewed in an SEM and reintroduced to the Ilion+® for further milling.
- Liquid nitrogen cold trap to minimize heating during the milling process.
- An optical microscope/CCD camera permits viewing of the sample during the milling process.

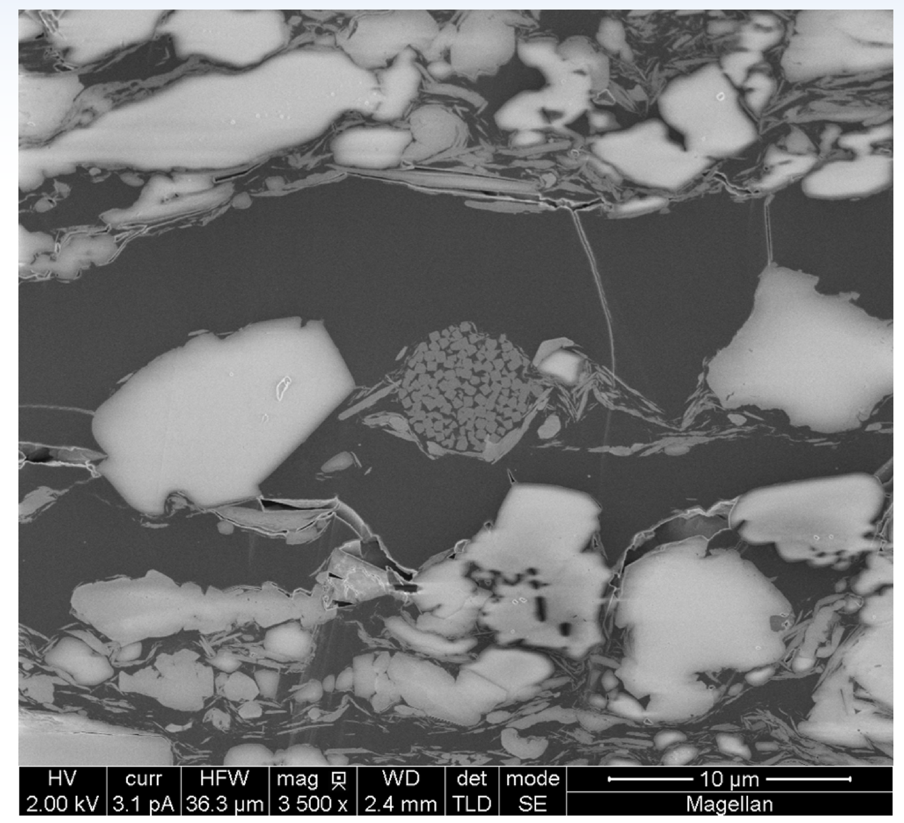
Figure 2 : Low magnification SEM image of oil shale

Milling with LN2 Cold Stage

The milling process with argon may increase the temperature of the sample by 50 to 70 degrees C. This can alter the material present or alter the grain structure of the sample. Figure 2 shows the cross section of a gas shale rock. This key point of preparing and viewing cross sections from this type of sample is to determine the presence of voids and their size to determine the potential of extracting oil from this rock.

The low magnification and high magnification SEM images (Figures 2 and 3) show the quality and smoothness of the cross section from the Ilion+® as well as in the high magnification image showing voids of approximately 3-4 nm in diameter. These nano-voids are preserved by the low KeV milling of the argon beam, as well as the cooling of the sample in the Ilion+®.

Milling of Large Cross Sections

A Unique aspect of the Ilion+® compared to FIB's is the ability to mill large cross sections as great as 1.5 mm. The SEM image in Figure 4 shows a completed LED device prepared on the Ilion+®. The cross section is of a complete device purchased commercially and is approximately 1 mm in length.

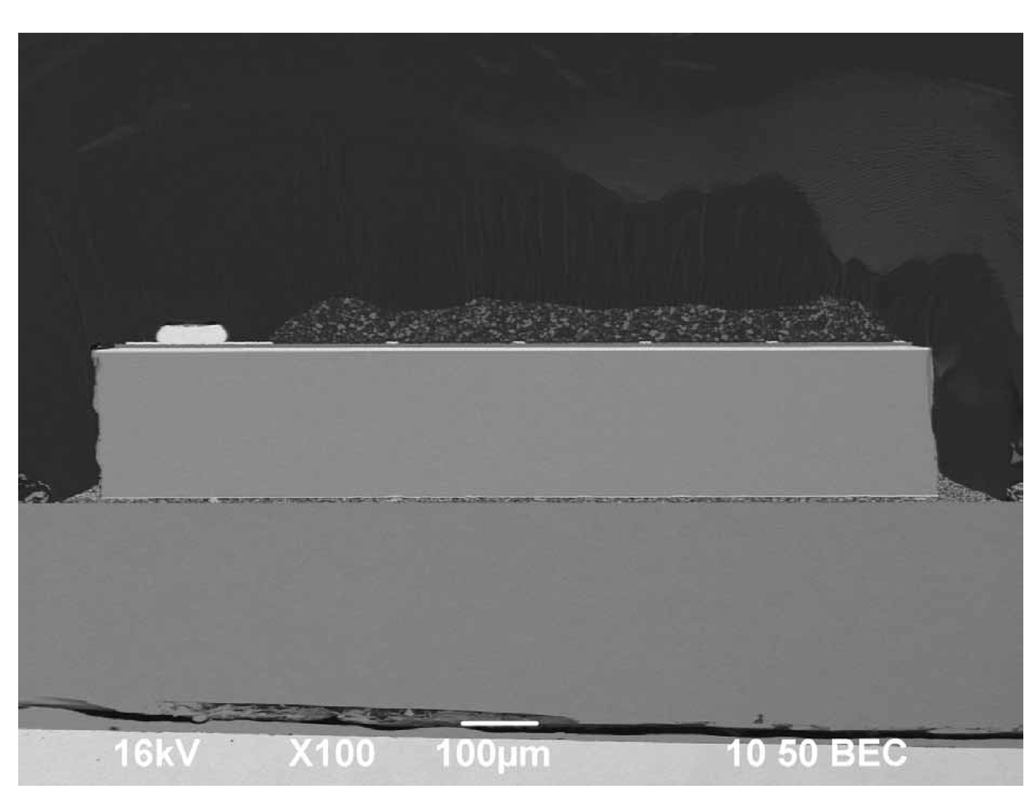

Figure 4 : Complete HB LED

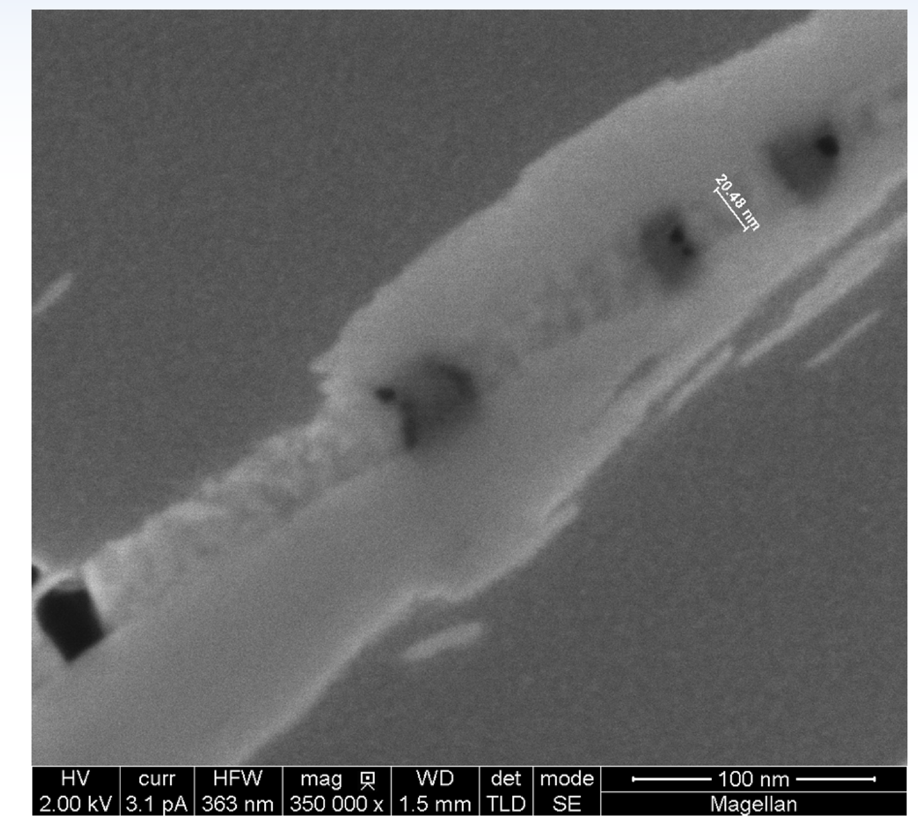
Figure 3 : High magnification SEM image of oil shale showing <5 nm pores

The cross section was prepared in less than 3 hours and contains a large number of interfaces from materials of extremely differing material characteristics. The materials present include, silicon, sapphire, plastic, gold bond, silicates, plated copper, phosphor and silicate.

The quality of the interface permits not only high resolution SEM imaging but CL Imaging, which requires low damage to the cross section in order for the light to be generated and escape as shown in Figure 5.

Milling of Metals to Observe Grain Structure

Argon milling is an effective method for polishing surfaces to bring out grain structure for either direct observation in the SEM or with EBSD. Ga milling will frequently chemically interact with the material changing the structure or create a damage layer due to the high energy and the mass of the Ga ion. Frequently a cold stage is required to prevent formation of intermetallics at the interface. Figure 6 shows the interface prepared on the Ilion+® with no voids or intermetallics at the interface of tin on copper. Figure 7 shows a copper grain structure prepared in the Ilion+®. Mechanical polishing would be very difficult due to the softness of the material resulting in smearing of the surface. In this image the grain structure is clearly visible in the SEM.

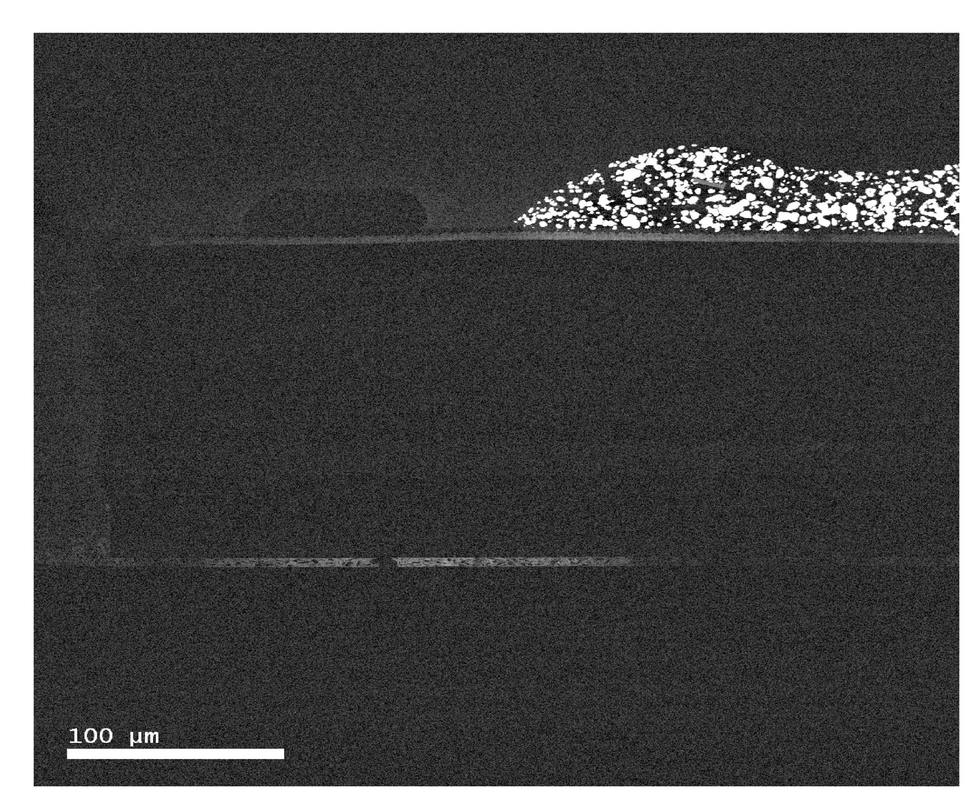

Figure 5: CL image of cross section of a HB LED

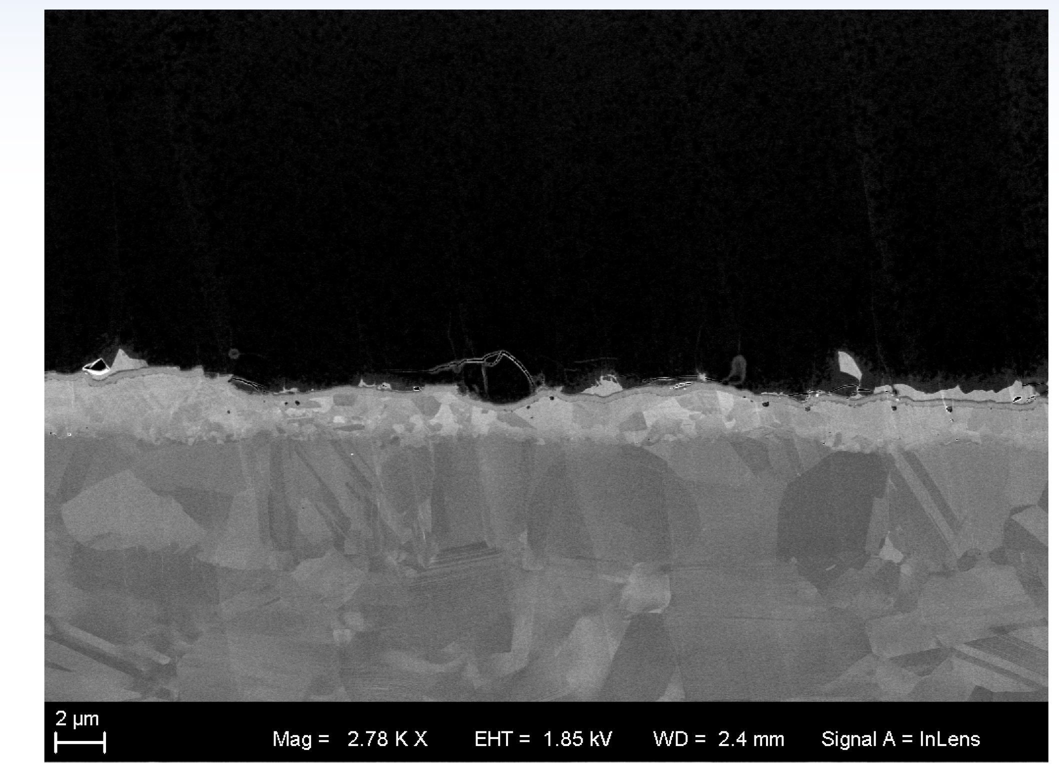
Figure: Cross section of tin coat on copper wire

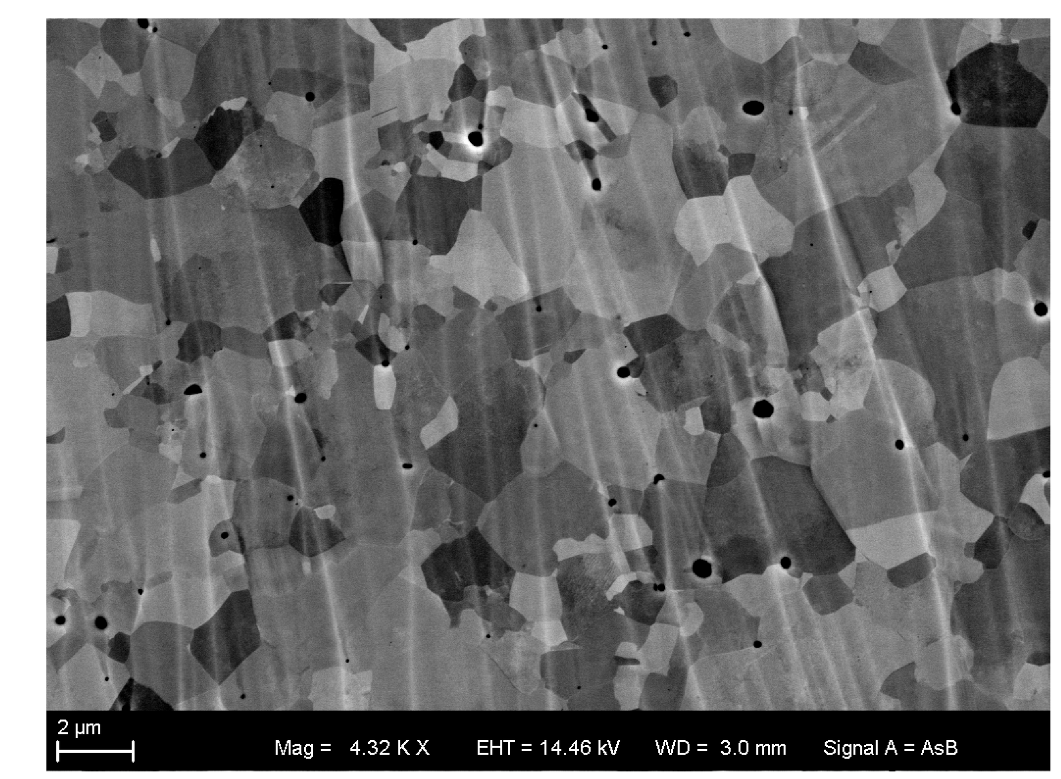
Figure 7: Copper grain structure after argon milling

Summary

Argon milling with the Ilion+® is an excellent technique for preparing a near infinite variety of materials for examination in the SEM for either imaging modes or analytical modes.

The Ilion+® from Gatan offers many advantages over other commercial offerings including the ability to re-introduce the sample back into the Ilion+® after observation in the SEM. The gun angles can be changed which then permits milling into the interface for observation of features in three dimensions. The Liquid Nitrogen Cold Stage permits milling of materials, such as plastics or rock samples containing vegetation to be milled preserving the true structure of the interface.

Finally, although FIB is a very useful technique frequently the need for large cross sectional areas (>1 mm) or without creating beam induced damage would suggest an argon milled cross section is a better choice.