

The broad argon ion beam eliminates problems associated with the conventional methods of specimen cross sectioning for SEM.

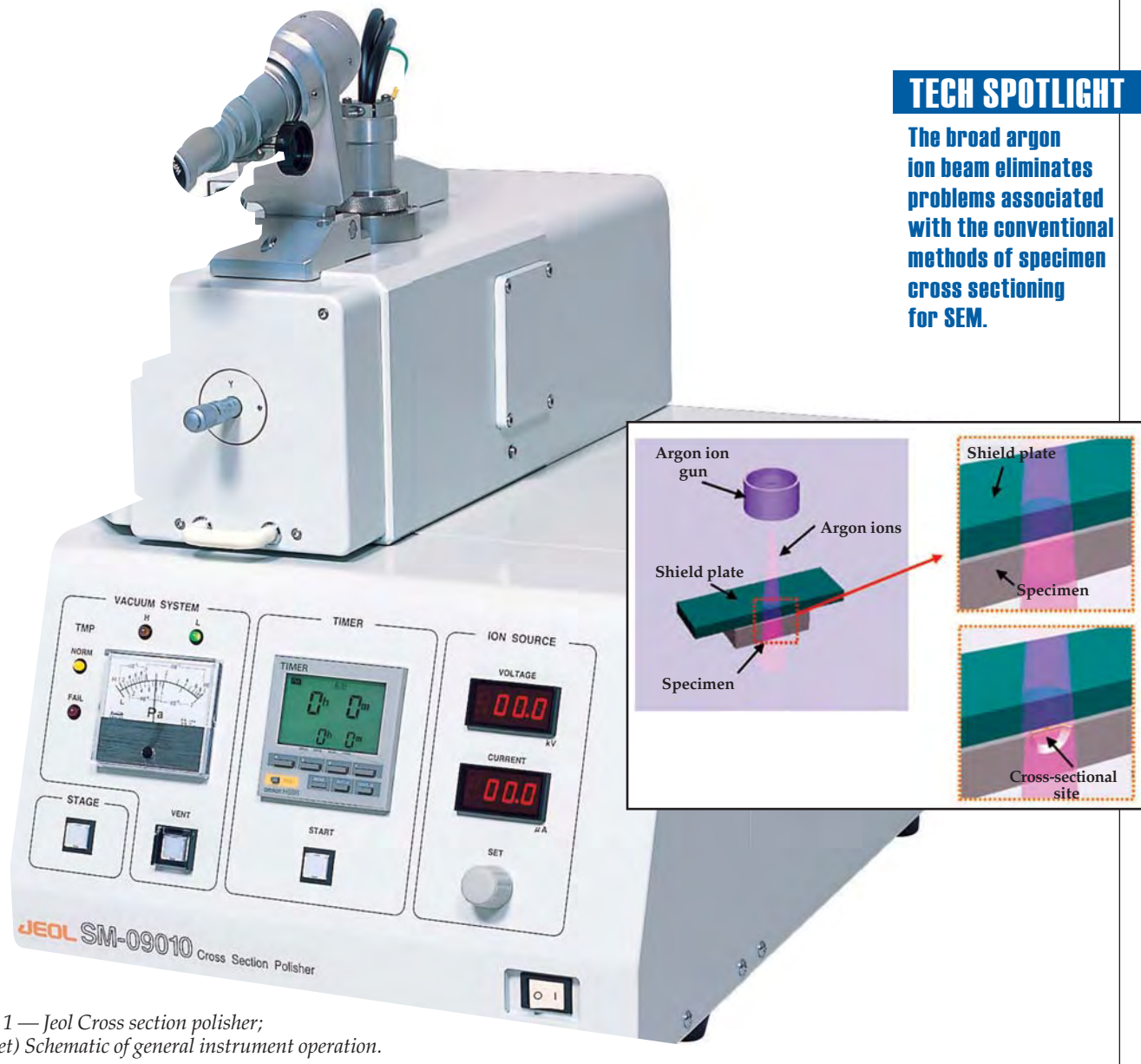


Fig. 1 — Jeol Cross section polisher;
(inset) Schematic of general instrument operation.

Argon Beam Cross Sectioning

N. Erdman and R. Campbell
Jeol USA Inc.
Peabody, Massachusetts

S. Asahina
Jeol Ltd., Japan

Examination of material cross sections often provides essential information about the crystal structure, layer or film thicknesses, existence of voids or cracks, and other properties that might impact performance and reliability. Many mechanical methods are available to prepare specimen cross sections for scanning electron microscope (SEM) observation, particularly for metallographic sample preparation. However, mechanical polishing presents several problems:

- In composite materials with dif-

ferent hardness values, the polished surface becomes uneven as the softer components are cut faster and more easily than the harder components;

- In soft materials, particles of hard abrasive can be buried in the material being polished;

- In materials with voids, the edges of the voids can stretch and deform;

- For metals, due to the strain caused by mechanical polishing on the polished surface, the information about the crystal structure by means of electron back-scatter diffraction (EBSD) can become difficult or impossible to see;

- Fine features such as hairline cracks and small voids can get smeared shut and will not be recognized as such; and

- Water-soluble phases are challenging to preserve, even with kerosene or oil instead of water.

Microtome methods

An ultra microtome and focused ion beam (FIB) are also often used to prepare cross sections of specimens. The ultra microtome is mainly for preparation of soft materials, such as biological and polymer specimens. Microtome preparation requires embedding of the sample with possible additional staining to reveal certain features or components; this is a lengthy procedure that requires a great deal of skill, and could sometimes lead to unforeseen artifacts in the resulting specimens.

The FIB is a powerful tool when precise positioning of a cross section is required. However, the size of the resulting cross section is very limited, and use of heavy gallium ions can cause some damage to the sample surface.

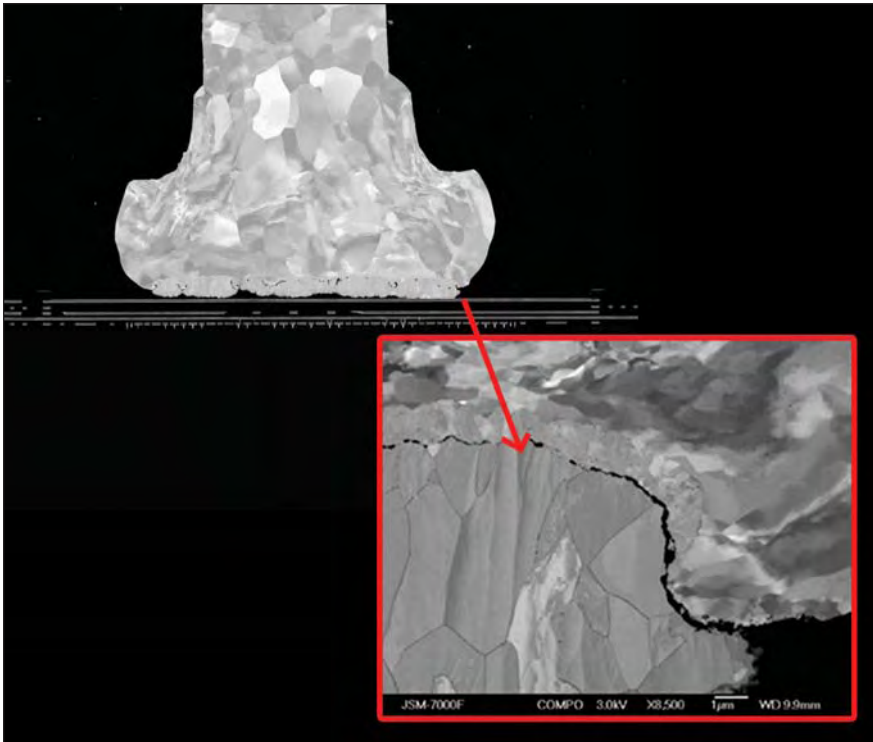


Fig. 2 — Gold wirebond cross section prepared with the Cross Section Polisher. The inset shows magnified view of grain structure and voids.

Argon beam specimens

The new specimen preparation apparatus, the Cross Section Polisher (CP), utilizes a broad argon ion beam that eliminates problems associated with the conventional methods of specimen cross sectioning for SEM. The CP consists of a specimen chamber with a TMP vacuum system, an optical microscope for specimen positioning, and controllers for the vacuum system and a stationary ion beam (Fig. 1).

The stage in the specimen chamber features a specimen holder and a masking plate. The region of interest to be cross sectioned is selected under an optical microscope. Then a masking plate is placed across the region. After evacuating the specimen chamber, the region is irradiated with a broad argon ion beam (Fig. 1 inset). Accelerating voltage range of the argon ion beam is 2 to 6 kV.

During ion beam milling, the specimen stage can be rocked 30 degrees. This prevents beam striations and ensures uniform etching of a sample composed of materials with different hardnesses. The instrument is set up on a timer that allows unattended operation.

The main advantages of the Cross Section Polisher over other preparation techniques include:

- High quality cross sections of composites of soft and hard materials

- Minimum strain and distortion of the polished surface, enabling observation of grain contrast (channeling contrast) clearly and easily

- Large cross section areas as compared to FIB (a single cut is typically 1.5 mm wide and several hundred microns deep)

- No particle embedding in the polished surface as compared to mechanical polishing

- Ease of operation

Material applications

- **Gold wire bond:** Figure 2 shows the backscattered electron image of a cross section of gold wire bonding on a silicon integrated circuit prepared with the CP. In spite of the large differences in hardness between all the materials, including silicon, aluminum, and gold, the cross section is very high quality, and clearly reveals narrow cracks and small voids in the bonding layers. Examination of such features typically proves crucial for failure analysis and quality control. Channeling contrast in gold can also be clearly observed.

- **Solder:** Figures 3a and 3b show a backscattered image and corresponding EDS (energy dispersive spectroscopy) map of a solder bump. Cross-section polisher sample preparation allows direct observation of layer crystal structure (backscattered imaging and channeling contrast), as

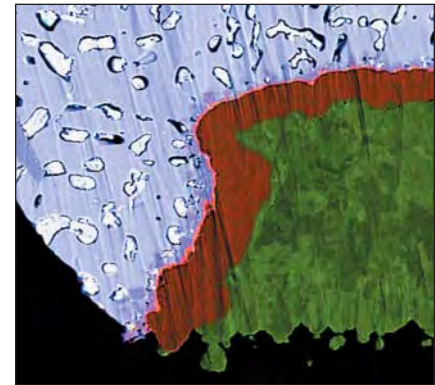
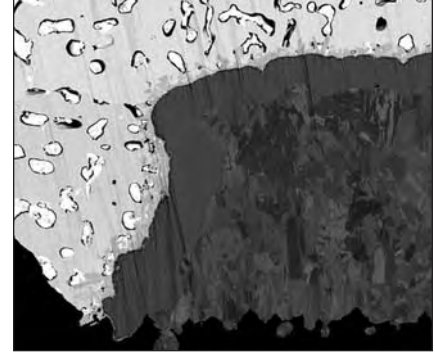


Fig. 3 — (a) Backscattered image of solder bump; (b) EDS map of the same location.

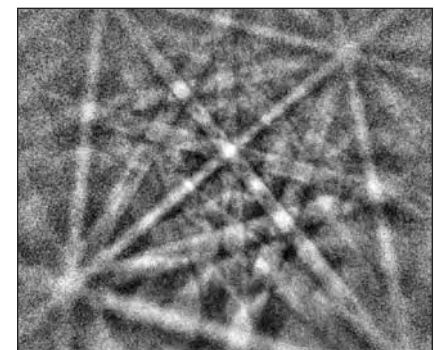
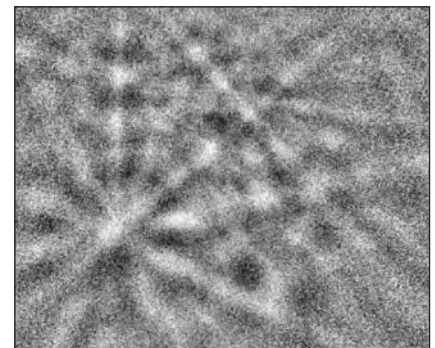


Fig. 4 — (top) Comparison of EBSD patterns collected from mechanically and Cross Section Polisher (center) prepared samples of steel cord; (bottom) EBSD map of the CP sample.

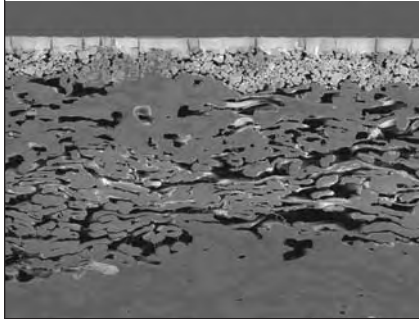


Fig. 5 — Cross section of a paper sample.



Fig. 6 — Backscattered image of fiber optic cross section.

well as compositional analysis of intermetallic layers. The structure of the solder joint is important in evaluation of flip chips; in particular, formation of voids and cracks in intermetallic bonding layers will likely influence the reliability of the device.

• **Crystal structure of steel cord:** Figure 4a shows a comparison of the EBSD patterns between mechanically

polished and CP-prepared samples of high strength steel cord. The EBSD pattern of the mechanically polished cross section was not detailed enough to provide high spatial resolution in EBSD mapping. However, a high spatial quality EBSD pattern was possible with the CP method. Figure 4b shows an EBSD map of a sample prepared by the CP method. The small grains of pearlite structure were observed from the EBSD map, including a grain size smaller than 50 nm.

• **Paper:** In general, sample preparation of a paper cross section involves either razor blade cutting or an embedding/staining/mechanical polishing procedure, both of which usually introduce a substantial number of artifacts into the resulting sample. The CP method provides a unique inside view of coated papers (Fig. 5), allowing analysis of the absorbency and ink penetration critical to the paper manufacturing process. The information helps in quality control and optimization of the printing characteristics of the paper.

• **Fiber optics:** Fiber optic cable specimens are difficult to prepare via

traditional methods due to differences in hardness between the layers of thermoplastic polymer cladding and the thin polymer/glass multi-layers comprising the overall structure. The Cross Section Polisher allows straightforward preparation and observation of the layers and measurement of layer uniformity and thickness. The resulting SEM image is presented in Fig. 6.

As these examples show, use of an argon ion beam makes it easy to prepare cross-sections of a wide variety of materials, including polymers, metals, ceramics, and composites, with minimal artifacts. It is suitable not only for image observation, but also for microanalysis and determination of crystal structure. ●

For more information: Natasha Erdman, Jeol USA Inc., 11 Dearborn Road, Peabody, MA 01960; tel: 978/536-2273; nerdman@jeol.com; www.jeol.com.

References:

M. Shibata, S. Asahina, T. Negishi, *Proc. 8 APEM*, (Kanazawa, 2004) p. 258
W. Hauffe, *Electron Microscopy*, Vol. 2, EUREM 92, Granada, Spain, 1992




HAYNES

International

A Tradition of Innovation Spanning a Century

Global service centers stocking
HASTELLOY® and HAYNES® alloys

- Sheet
- Plate
- Bar
- Welding Products
- Fittings
- Flanges
- Pipe & Tubing

Offering plasma, water-jet, and laser precision cutting
and shearing options for near-net shapes

World-class leader in technical support and alloy innovation

ISO9002 CERTIFIED

Corporate Office

1020 West Park Avenue, Kokomo, In. 46904

Phone: (800) 354-0806, Fax: (765) 456-6905

Sales Offices & Service Centers

Windsor, Ct. • La Mirada, Ca. • Houston, Tx • Arcadia, La.
England • France • Canada • Singapore • China • Italy • India • Switzerland

www.haynesintl.com

©HAYNES and HASTELLOY are registered trademarks of Haynes International, Inc.