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Reliability Assessment of Indium Micro Bumps for 2.5D/3D Electronic Packaging Through Cryo-Argon Milling Technique

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Abstract—**Indium bumps are widely used in electronic packaging for interconnecting microelectronic devices due to their low melting point of 156.6°C, excellent bonding capabilities at room temperature, high thermal conductivity, ductility at extremely low temperatures, and ability to compensate for the mismatched coefficient of thermal expansion between different materials. However, cross-sectioning indium bumps can be challenging due to the softness and low melting point of indium. The cutting tools and techniques used must be optimized for the specific sample being analyzed, and specialized preparation techniques such as cryogenic freezing or chemical etching may be required, which can be expensive and time-consuming. Cryo-focused ion beam (FIB) is also a technically demanding process that requires careful optimization of milling parameters, multiple iterations of milling and imaging, specialized equipment, and expertise, which can make it too expensive. It should be noted that the maximum achievable cross-sectional area with cryo-FIB is limited, typically in the range of hundreds of micrometers. Cryo-Argon milling is an effective technique for obtaining high-quality cross-sections of indium bump arrays and other materials prone to deformation or melting during traditional cross-sectioning. This process involves freezing the sample using liquid nitrogen and bombarding it with a stream of Argon gas ion, which reduces the deformation and melting of the indium. This paper presents challenges in the sample preparation and mechanical polishing of indium micro bumps. We are showcasing an artifact: polishing particles embedded in the indium micro bumps, hindering the revelation of their microstructure. To address this issue, we are incorporating cryo-Ar milling to remove the embedded particles from the surface of indium micro bumps. This work presents data before and after the application of cryo-Ar milling, demonstrating a significant improvement in the microstructure of indium micro bumps. Millimeter-wide, damage-free cross-sections of arrays of indium micro bumps with a clean and smooth surface were achieved through cryo-Argon milling at −70°C.**

Keywords—**Electronic packaging, failure analysis, indium bump, cross-section, Ar milling**

Introduction

In recent years, interconnect technology has drastically changed, from traditional wire bonding in a chip and wire packaging to newer flip chip packaging. Advances in interconnect technology have greatly reduced the footprint of devices and increased data transfer speeds [1].

Harsh and frigid conditions that surpass the operational limits of military applications, such as temperatures as low as −55°C, necessitate specialized features in electronic systems. Space exploration offers numerous instances of such extreme cold environments, often accompanied by wide temperature cycling ranges. For instance, the equatorial region of the moon experiences temperature variations from −180°C during the night to 120°C during the day. Similarly, the temperature on Mars ranges from −120°C to +20°C. To operate effectively in these environments, space electronics must be capable of withstanding both extremely low temperatures and wide temperature fluctuations [2].

Indium, a malleable metal, is frequently employed for attaching flip-chip dice at lower temperatures. Indium provides numerous benefits compared with conventional solder alloys when used for die attachment. These advantages include its ductility at extremely low temperatures, excellent thermal conductivity, and the ability to compensate for coefficient of thermal expansion (CTE) mismatches between different materials [3, 4]. However, assessing the reliability of indium bump cross-sections present several challenges.

Cross-sectioning of indium bumps can be challenging due to the softness and low melting point of indium, variability in bump size, shape, potential oxidation, and contamination issues.

Conventionally, there are numerous methods for interconnecting various types of packages through nondestructive defect inspection techniques. These methods include x-ray, lock-in thermography, time-domain reflectometry, magnetic current imaging, and scanning acoustic microscopy. However, to comprehend the root causes of manufacturing yield problems, reliability issues, and material interactions in contact interfaces like indium interconnects, nondestructive analysis often proves insufficient. High-resolution material analysis based on scanning transmission electron microscopy, x-ray, or mass spectroscopy is typically necessary [5, 6]. Focused ion beam (FIB) has become a staple in the semiconductor industry for failure analysis, providing a common method to investigate small areas and allowing for high-definition images for examination [7, 8]. However, FIB also has its drawbacks. One of these drawbacks is its limited investigative area, often under 100 μm. Another common liability is specimen damage; a frequent issue observed with FIB cross-sections is sample damage

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or the need for tedious and costly sample preparation to avoid damage [9-11].

This is where specialized techniques, such as cryogenic freezing or chemical etching, may be necessary to improve the quality of the cross-sections. Traditional manual grinding and polishing methods can cause pressure cracking, making it challenging to determine if cracks in structures are a result of sectioning. Cryo-FIB is a time-consuming and technically demanding process, requiring careful optimization of milling parameters, multiple iterations of milling, imaging, specialized equipment, expertise, and can be expensive. It should be noted that the maximum achievable cross-sectional area with cryo-FIB is limited and is typically in the range of hundreds of micrometers rather than millimeters.

The broad ion beam (Ar BIB) method is a widely used technique for creating cross-sections. It shares similarities with the (FIB) method, where the focused ion beam sputters a specific position on the specimen without scanning (Fig. 1). Adjusting the irradiation angle can be done by tilting, rotating, and counter-rotating the specimen, which aids in achieving surface planarity and minimizing the impact of milling rate variations caused by crystal orientation or material composition.

An argon ion milling system can produce cross-sections of FIB quality up to 500 times faster and 1,000 times wider than traditional FIB instruments. It offers two milling modes: (1) flat milling and (2) cross-section milling.

During flat milling mode (Fig. 2), the ion beam exhibits a Gaussian-shaped current density profile. Material removal occurs at a higher rate than the surrounding area. By varying the specimen rotation and swing center relative to the ion beam center, a broader area can be sputtered with enhanced uniformity. In general, the flat milling technique is used for removing stains or polishing flaws, clarifying interfaces and crystal contrast, determining crystalline grain boundaries, and elucidating metal composites without the need for chemical etching.

For cross-section milling mode (Fig. 3), a mask is placed directly on top of the specimen, serving to protect the surface while creating a sharp edge. This edge ensures a damage-free flat cross-section face by sputtering away the exposed material beyond the masked edge.

Ar milling techniques enable the production of bulk sample cross-sections and ultra-fast mill rates, allowing for cross-sections

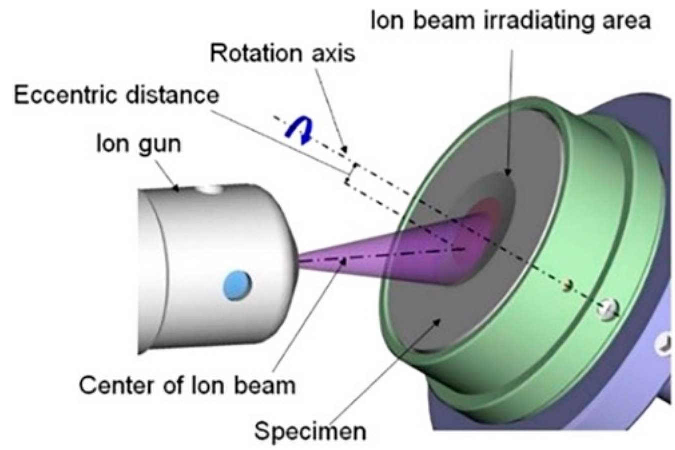

Fig. 2. Ar Blade flat milling mode [12].

of 500 μm through a localized sample area in under 1 h (estimated for silicon). In contrast, FIB typically achieves cross-section depths of 1-20 μm (with 20 μm requiring several hours depending on the materials).

Cryo-Ar Blade mode facilitates stable cross-sectioning of thermally sensitive materials by utilizing cryogenic stage cooling, which is especially advantageous for low-temperature metals, photoresists, benzo cyclobutene, and similar substances. Cryo-Argon milling is one such technique that involves freezing the sample and bombarding it with Argon gas ions, providing high-resolution imaging and overcoming the challenges associated with traditional cross-sectioning. It is effective in obtaining high-quality cross-sections of indium bumps and other materials prone to deformation or melting [5].

This research paper examines the preparation of cross-sections to evaluate the joint formation of thermal compression indium bumps. These indium interconnect joints are created through a thermocompression process that involves the application of heat and pressure. The cross-section analysis plays a crucial role in assessing joint formation and bonding quality for the interconnect. The data collected from this analysis is of paramount importance in evaluating the success of the interconnect formation and ensuring the reliability and integrity of the joints.

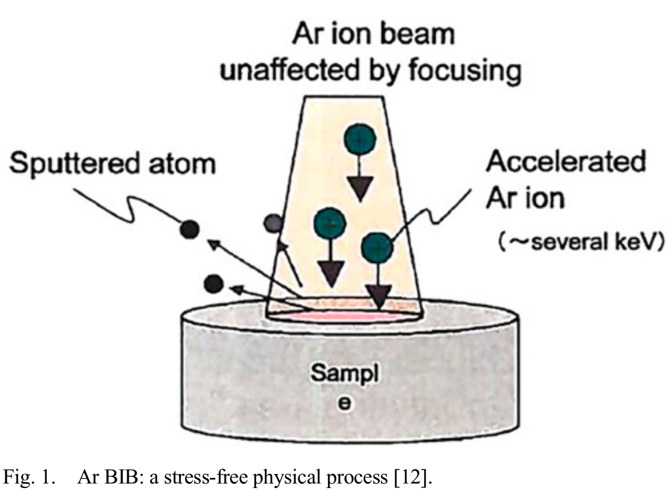

Fig. 1. Ar BIB: a stress-free physical process [12].

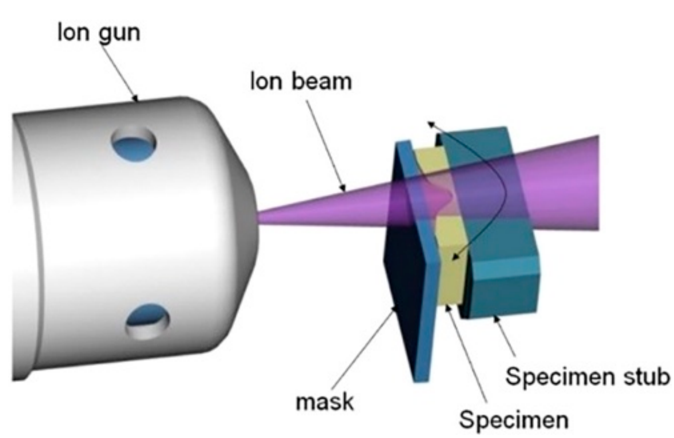

Fig. 3. Ar Blade cross-section mode [12].

Table I
Guideline for Mechanical Polishing of Indium Bumps

Grinding sequence	DLF (μm)	Wheel RPM	Grinding distance from area of interest (μm)	Grinding length (μm)
1	30	175	300	100
2	15	150	200	3 × 30 = 90
3	9	125	110	3 × 15 = 45
4	6	100	65	3 × 9 = 27
5	3	75	38	3 × 6 = 18
6	1	50	20	3 × 3 = 9
7	0.5	25	11	3 × 1 = 3

The primary focus of this study is to investigate the cross-section of indium bumps using the cryo-Ar mill mode. The optimization of various factors, including sample preparation, mechanical polishing, and the Ar mill process, is essential to achieve successful and reliable cross-sectioning of indium micro bumps using the cryo-milling method.

Experimental Procedure

Indium-plated bumped tiles, featuring indium bumps bonded to die pads through a thermocompression mechanism, underwent dicing with a diamond saw to obtain smaller pieces for the purpose of mechanical cross-sectioning. Subsequently, the samples were mounted using epoxy resin, defoamed in a vacuum chamber, and cured on a hot plate at 100°C for a duration of 30 min. To achieve the desired interface between the pad and die, mechanical polishing was performed using successive grades of diamond lapping films (Table I).

Fig. 4a illustrates the direction of movement of the mounted sample on the polishing wheel, which rotates in a clockwise direction. Fig. 4b demonstrates the sequential process of changing the diamond lapping film grade as the material is progressively removed, approaching the location of the bumps. Fig. 4c exemplifies the removal of scratches as the polishing steps transition from a course to a finer film.

For cross-section preparation, an ion milling polishing machine was employed (Ar Blade). During the Ar milling operation, an acceleration voltage of 8 kV was set, and the milling process was carried out for a duration of 5 h across a 5 mm wide interface. The surface of the micro bumps was analyzed utilizing the scanning electron microscopy (SEM) technique. For investigating the elemental composition at the interface, energy dispersive x-ray spectroscopy (EDS) was performed. Additionally, the FIB technique was employed to cross-section the unbounded indium micro bumps and analyze the SEM micrograph.

A series of experiments were conducted, and the results were analyzed to examine the quality of indium micro bumps. The following procedures were carried out: (1) The mounted samples underwent traditional mechanical polishing, and

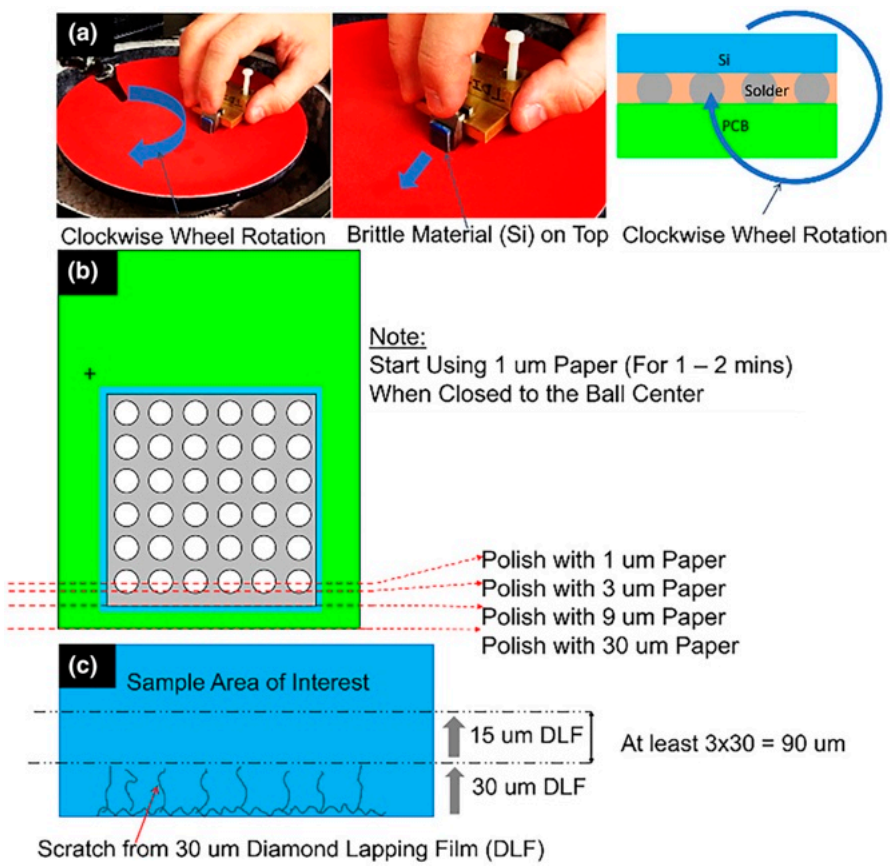

Fig. 4. Guideline for mechanical polishing. (a) Polishing direction on a clockwise rotating wheel, (b) guideline distance for switching from course to fine diamond lapping films as approaching toward indium bumps, and (c) schematic of the scratch-removing process from coarse to fine polish.

subsequent SEM images were captured. (2) Flat Ar milling was employed to enhance the effectiveness of the mechanical polishing process. (3) The Ar mill cross-section mode was tested at room temperature to minimize artifacts induced during the mechanical polishing. (4) Cryo-Ar mill cross-section mode was implemented at a temperature of −70°C to mitigate any potential adverse effects of heat on the indium micro bumps. (5) Cryo-Ar mill conditions were optimized to attain indium micro bumps with clean and smooth surfaces.

Results and Discussion

Fig. 5 displays the SEM image of the cross-section of indium micro bumps obtained immediately after the completion of the fine mechanical polishing procedure (traditional manual polishing method). It should be noted that due to the soft nature of indium, particles originating from the die or cured epoxy materials or loose diamond grit/particles from the lapping film can become trapped on the surface of the indium bump during the mechanical polishing process. This occurrence has the potential to introduce artifacts or cause damage to the structural integrity of the bump, resulting in a significantly roughened surface. The presence of such roughness can obscure the detection of potential cracks, thereby presenting a challenge in terms of reliably assessing the integrity of the bumps.

To enhance the quality of the cross-section, an experimental procedure involving flat Ar milling was employed. The indium bump's cross-section was exposed to the Ar beam at an inclined angle of 80 degrees, and milling was performed for a duration of 10 min (Fig. 6). However, the subsequent results did not yield a significant improvement in the cross-section quality. The procedure merely reduced the roughness observed on the aluminum nickel pad located at the bottom of the bump and its surrounding area. Notably, black particles, which had become embedded in the indium bumps during the mechanical polishing stage, remained visible on the bump's surface. It is postulated that the heat generated by the Ar beam also adversely affects the texture of the indium surrounding the nickel pad.

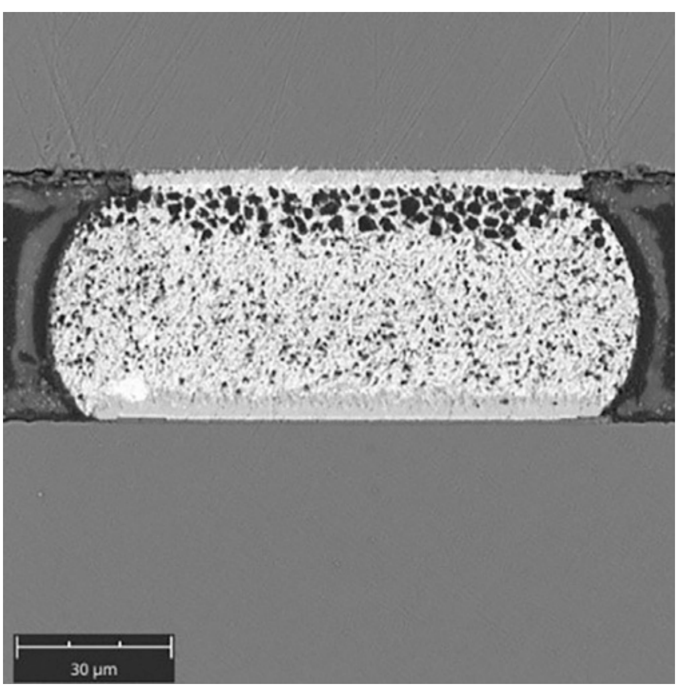

Fig. 5. SEM micrograph depicting the cross-sectioned indium micro bump achieved through manual polishing (traditional polishing method procedure).

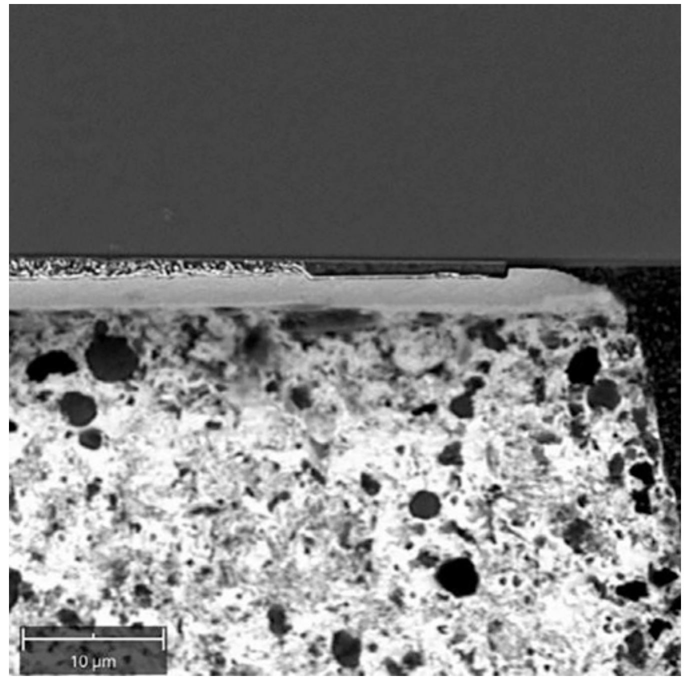

Fig. 6. SEM micrograph illustrating the cross-sectioned indium micro bump that has been cleaned using the flat Ar milling procedure.

To further address the issue of debris on the surface of the indium bumps, a cross-section milling procedure was performed at room temperature. Fig. 7 displays an SEM view of the cross-sectioned indium micro bump following the Ar milling process. The milling procedure yielded a notable improvement in the cross-section profile and successfully removed most of the black particles that were previously adhered to the surface. However, evidence of cracks and delamination persisted around the top and bottom pads, which is likely attributed to the heat generated during the Ar milling process or residual effects from the preceding mechanical polishing stage.

To prevent any adverse effects resulting from heat exposure by the Ar beam, milling was conducted under cryo-mode. As part of this process, the die was cooled down to −70°C using liquid nitrogen following the mechanical polishing stage. The implementation of cryo-milling successfully revealed a clean, smooth, and damage-free profile of the indium bumps (Fig. 8).

Notably, cracks were observed at the top intermetallic interfaces of the indium micro bump (Fig. 9). To gain a comprehensive understanding of the underlying cause behind these observed cracks, an extensive investigation was conducted to ascertain whether they were induced during either the thermo-compression bonding process or the characterization process.

During the mechanical polishing process, debris can adhere to the surface of the indium bumps. When the Ar beam is utilized to remove this debris (Fig. 10), there is a possibility that

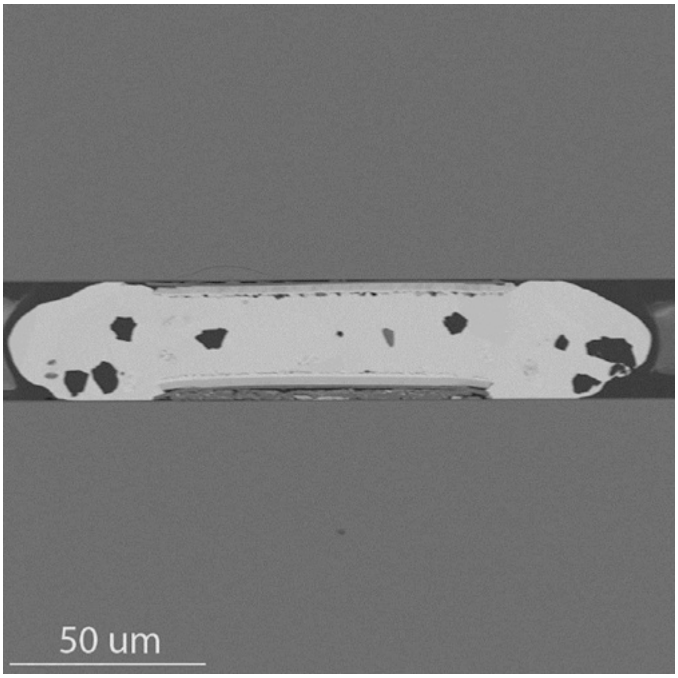

Fig. 7. SEM image showcasing the Ar mill cross-section microstructure of the indium micro bump at room temperature.

hard debris particles exert excessive force on the interface, leading to damage. To investigate this hypothesis, an extended Ar milling procedure was conducted for 10 h under cryo-mode to eliminate any remaining die materials and expose the surface of the indium bumps. This approach aimed to prevent the trapping of hard debris generated during mechanical polishing on the surface of the bumps.

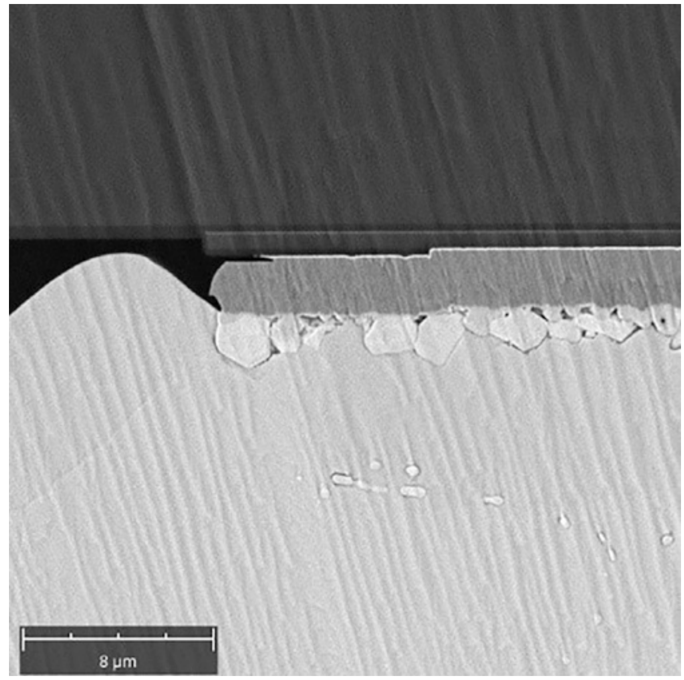

Fig. 9. SEM image depicting a crack observed at the top interface.

Despite the significant improvements achieved through the extended Ar milling process, resulting in smooth and void-free indium micro bumps, cracks were still observed at the top interface between the intermetallic layer and bulk indium (Figs. 11 and 12).

To further investigate the origin of the crack, EDS analysis was performed at the crack interface (Fig. 13). It was observed

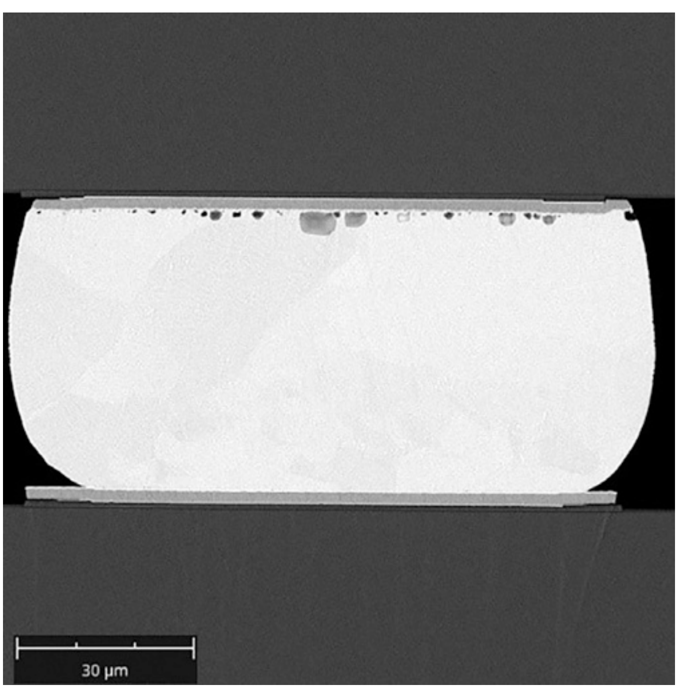

Fig. 8. SEM image illustrating a clean and smooth surface of the indium bump cross-section achieved through cryo-Ar milling at $-70°C$.

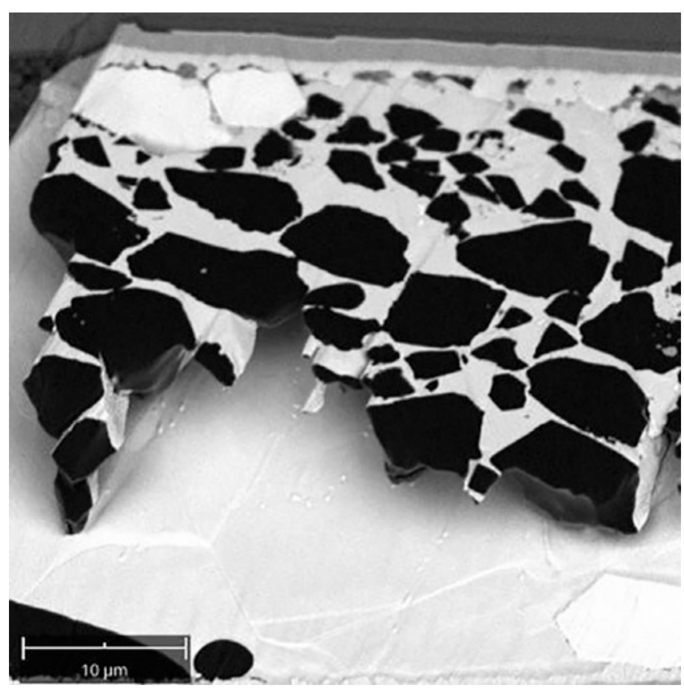

Fig. 10. Tilted SEM image illustrating the presence of trapped particles within the soft texture of the indium bump.

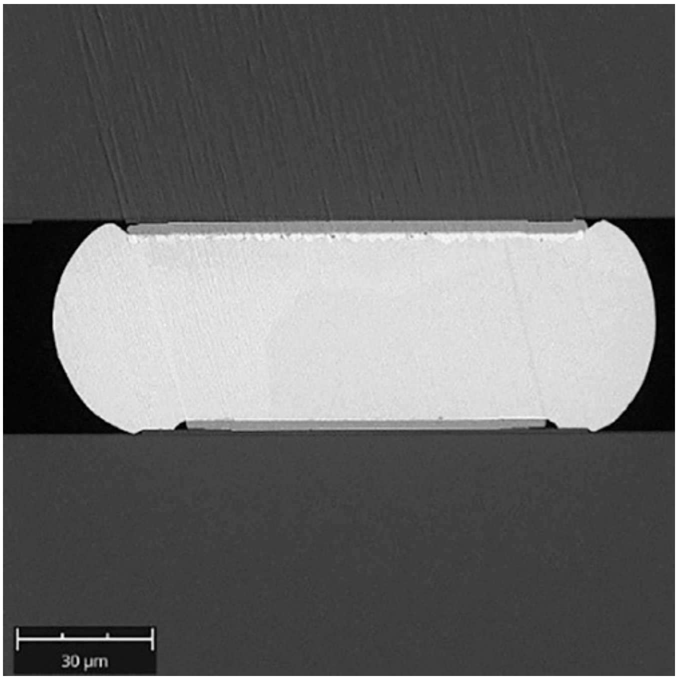

Fig. 11. The extended cryo-Ar milling process notably enhances the quality of the cross-sectioned indium bump.

that the underfill sample exhibited cracking at the gold/indium interface, which can be attributed to the formation of void due to the Kirkendall effect at intermetallic phase between indium and gold [4].

To remove any possible doubts about the impact of (1) thermocompression bonding, (2) mechanical polishing, and (3)

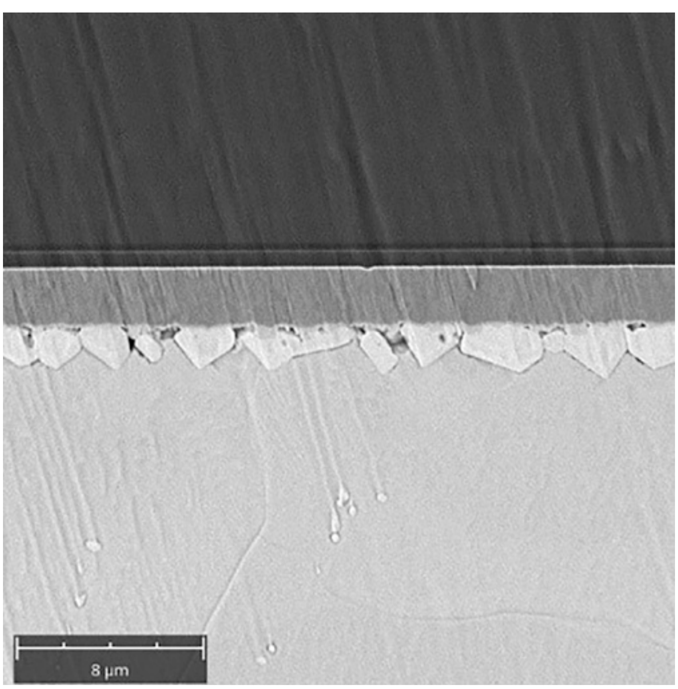

Fig. 12. Identification of a crack at the top interface following the extended cryo-milling procedure.

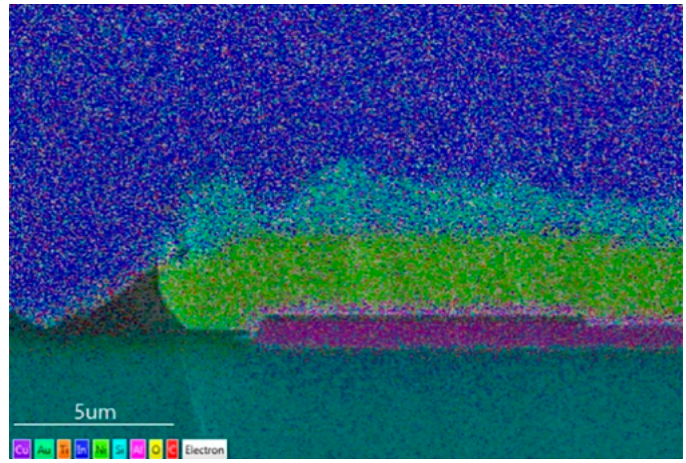

Fig. 13. EDS analysis at the crack interface.

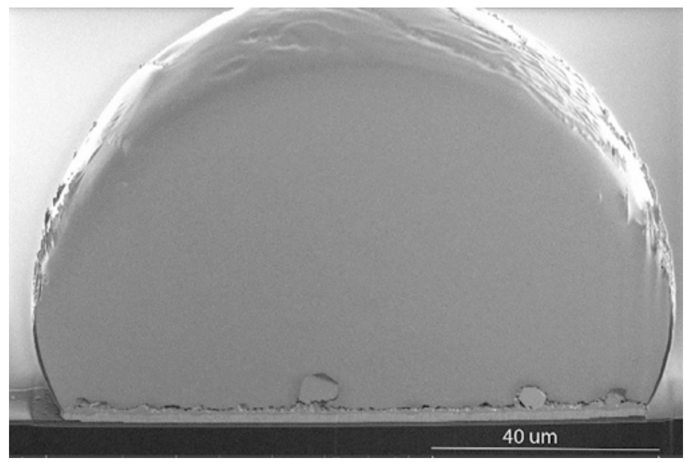

Fig. 14. SEM image displaying the cross-section of the indium micro bump obtained through the PFIB technique.

cryo-Ar milling processes on the observed crack, we obtained a cross-section of an unbounded sample using the plasma FIB (PFIB). It is important to note that, at this stage, the sample had not been subjected to any thermocompression process

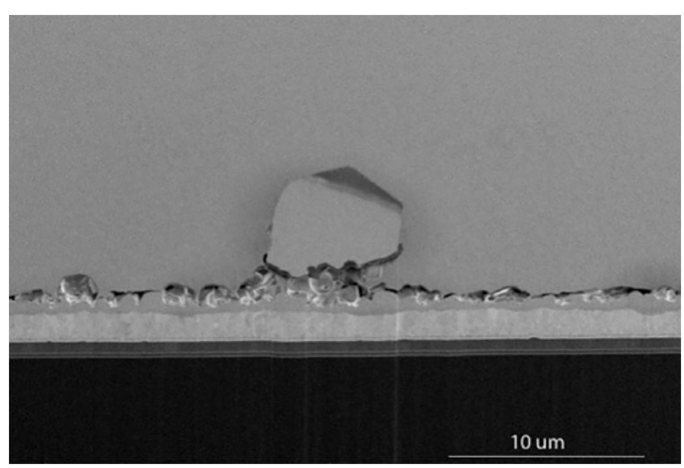

Fig. 15. High-magnification SEM micrograph highlighting the presence of a crack at the indium/intermetallic interface.

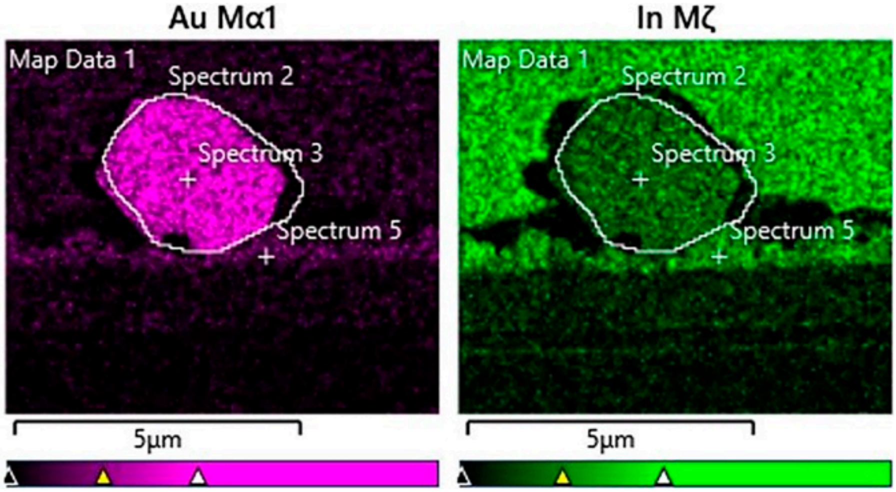

Fig. 16. EDS mapping confirming the formation of a crack at the indium/intermetallic interface.

(eliminating the possible potential factors for crack formation due to force during the process). Also, no sample preparation, such as mounting using epoxy/resin or mechanical polishing, was done before the PFIB process. Fig. 14 shows the cross-section of the micro indium bump (as received from the vendor) captured using the PFIB technique. Despite the absence of the thermocompression process and sample preparation steps, a crack was still observed at the indium/gold intermetallic interface (Fig. 15). This evidence confirms that the crack formation cannot be attributed to either our thermocompression bonding procedure or the cryo-Ar milling cross-sectioning techniques. In essence, the components received from the supplier were mostly defective, with indium bumps delaminated from the substrate at the indium-gold interface.

The EDS analysis performed confirmed the formation of a crack at the indium/intermetallic interface, which is likely attributed to the presence of Kirkendall voiding at the interface (Fig. 16) [4]. The formation of Kirkendall voids in the indium-gold interface is primarily due to the diffusion of atoms across the interface. When indium and gold are in contact, there is a diffusion of atoms from one material into the other. However, the diffusion rates of indium and gold atoms may not be equal. In this case, if the indium atoms diffuse faster into the gold than the gold atoms diffuse into the indium, it leads to a net migration of atoms toward the gold side. As a result, voids or vacancies form on the indium side, known as Kirkendall voids. This phenomenon occurs due to the difference in atomic mobility or diffusion rates between the two materials [13].

For future work, it is crucial to conduct additional investigations to grasp the mechanism behind crack formation during the manufacturing process. Detailed knowledge of manufacturing process steps is essential, as the development of voids is primarily linked to processing stages like plating, diffusion, and the control of intermetallic phase growth.

Conclusions

Millimeter-wide and damage-free cross-sections of arrays of indium micro bumps were obtained using the cryo-Ar milling technique. Optimization of the process was carried out through a series of experimental processes, including manual polishing, followed by flat Ar milling, cross-section Ar milling at both room temperature and cryo-milling at $-70°C$.

The main challenge in achieving clean and smooth microstructures of the indium micro bumps lies in the presence of polishing particles originating from the die or epoxy materials or loose diamond grit/particles from the lapping film. These hard particles tend to adhere to the surface of the bumps, posing difficulties for their removal. To mitigate damage, it is recommended to use both mechanical and cyro-milling to avoid any artifact for the indium cross-section process. Upon observing a crack on the unbounded indium sample before the thermocompression process or any sample preparation, it can be deduced that the crack did not occur during the mechanical polishing process or as a result of excessive force from a black particle during the cryo-Ar mill process. It is presumed that the formation of Kirkendall voids within the intermetallic phase between indium and gold is the primary factor contributing to crack formation. Further investigation is warranted to gain a deeper understanding of the precise mechanism underlying crack formation during the manufacturing process.

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