マテリアル先端リサーチインフラ利用報告書
ARIM User's Report

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課題データ / Project Data

課題番号 Project Issue Number	23KU0031
利用課題名 Title	In-situ observation of the deformation of Sn-Bi low temperature solder alloy under tensile loading
利用した実施機関 Support Institute	九州大学 / Kyushu Univ.
機関外・機関内の利用 External or Internal Use	外部利用/External Use
横断技術領域 Cross-Technology Area	計測・分析/Advanced Characterization
重要技術領域 Important Technology Area	次世代ナノスケールマテリアル/Next-generation nanoscale materials
キーワード Keywords	Low temperature solder, Sn-Bi alloys, transmission electron microscopy, in-situ mechanical testing,電子顕微鏡/ Electronic microscope

利用者と利用形態 / User and Support Type

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利用形態 Support Type	技術補助/Technical Assistance

利用した主な設備 / Equipment Used in This Project

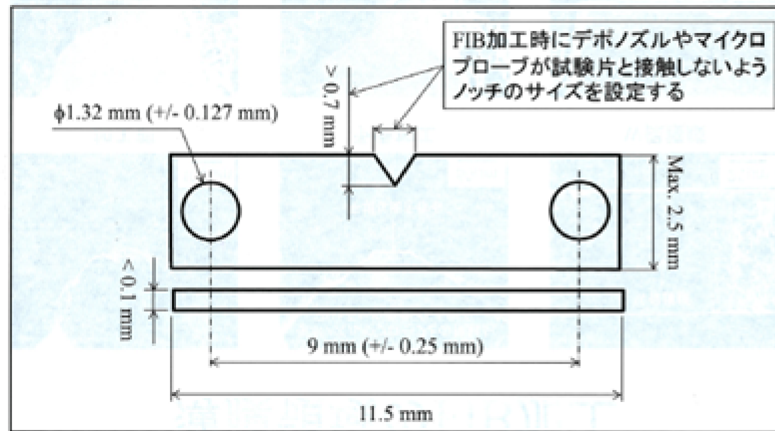
利用した主な設備 Equipment ID & Name	KU-001：電子分光型超高圧分析電子顕微鏡

報告書データ / Report

概要（目的・用途・実施内容） **Abstract (Aim, Use Applications and Contents)**	With low liquidus temperatures, low raw material costs, and non-toxicity, Sn-Bi low temperature solders are promising candidates for the replacement of the currently widely-used lead-free solders in situations in which process temperatures have to be reduced. The project elucidates the mechanical properties of the Sn-Bi alloys at microscales through in-situ electron microscopy. In particular, the failure mechanism of the material under a tensile load was studied. It was found that cracks initiate preferential at phase/grain boundaries perpendicular to the load direction. This is followed by sliding of the Sn/Bi and Sn/Sn boundaries, while the Bi/Bi boundaries are relatively brittle.
実験 **Experimental**	Sample preparation: The near-eutectic Sn-57wt%Bi (Sn57Bi hereafter) alloy was prepared by Sn and Bi ingots (99.9 %, supplied by Northern Smelters Pty Ltd., Australia) of appropriate quantities were melted at 450 ˚C for 1 h to prepare the Sn-57wt%Bi alloy, and casted into an ingot mould preheated to 150˚C. The ingot was remelted and casted into a slightly oversized aluminium mould preheated to 150˚C, with planar dimensions shown in the Fig. 1. The thick cast sample was then filed and polished to the dimensions shown in Fig. 1. A notch was filed into the sample to ensure that the stresses are concentrated in this region. Following this, the sample was sliced into pieces and polished to a thickness of less than 0.1 mm with a final finish of 1 μm. An electron transparent region was prepared at the tip of the notch by an improvised cryogenic focused ion beam (cryo-FIB) technique, shown in Fig. 2. TEM Experiment: The JEOL JEM-1300NEF HV-TEM used for this experiment was set to an accelerating voltage of 1,250 kV, and is equipped with an omega-type energy filter and a Gatan Single Tilt Heating Straining Holder (Model 672). Conventional TEM requires a sample thickness of less than 200 nm, while the HV-TEM enables the use of a 500 nm thick lamellae for the experiment to minimise plane strain and sample damage during FIB. The samples were mounted on the holder bolted down by two screws and inserted in the HV-TEM for in-situ elongation observation. An energy slit of 80.0 eV and an energy shift of 100.0 eV was applied for the best image contrast. The sample was elongated intermittently through displacement of the crosshead with a direct current (DC) motor at a rate of 1.0 μm/s. The real-time tensile load was recorded and shown in Fig. 3c. Elongation was stopped when significant changes in the microstructure were observed, and images were taken for the whole electron transparent region at each of these points, until the crack propagates through the electron transparent region.
結果と考察 **Results and Discussion**	Fig. 3 shows the energy-filtered images of the pristine sample stitched together to observe the whole electron transparent region. Under this condition, Bi which diffracts more electrons has a darker contrast, while the brighter material is Sn. The Bi grain at the top left corner of the electron transparent area showed signs of FIB damage including dislocations and nanosized defects, while other grains had no significant damage. As seen in Fig. 3, crack initiated at 3.8 N at a Sn/Sn grain boundary perpendicular to the load direction. The initiation of cracks perpendicular to the load direction is expected as the stress is greatest in this direction. At 5.2 N, cracks continued to form at Sn/Sn, Sn/Bi and Bi/Bi boundaries near-perpendicular to the load direction. This is followed by sliding of the Sn/Sn and Sn/Bi boundaries near-parallel to the load direction at 5.6 N, while the Bi/Bi boundaries are more brittle than the aforementioned. Grain boundary sliding often occur in polycrystalline materials at homologous temperatures above 0.4 at low strain rates. At 5.9 N, the Sn/Sn detached while the Sn/Bi boundaries continued to slide until at 7.0 N when the crack propagated through the electron thin region. The fracture mode is intergranular. The sample did not break completely as the maximum drive range was 2.0 mm and thus the strain was small. Repeat experiments with intermittent elongation (similar to the experiment above) and continuous elongation confirm that crack initiations are often along boundaries perpendicular to the load direction, with no significant preference of the Sn/Sn, Sn/Bi or Bi/Bi boundary, and followed by sliding of Sn/Sn and Sn/Bi boundaries parallel to the load direction.

図・表・数式 1
Figures, Tables and Equations 1

その場引張試験片の形状・寸法



試験片の切り出しには、例えば「放電加工ワイヤーカッター」を使用

Fig. 1. Planar geometry of the TEM lamella.

図・表・数式 2
Figures, Tables and Equations 2

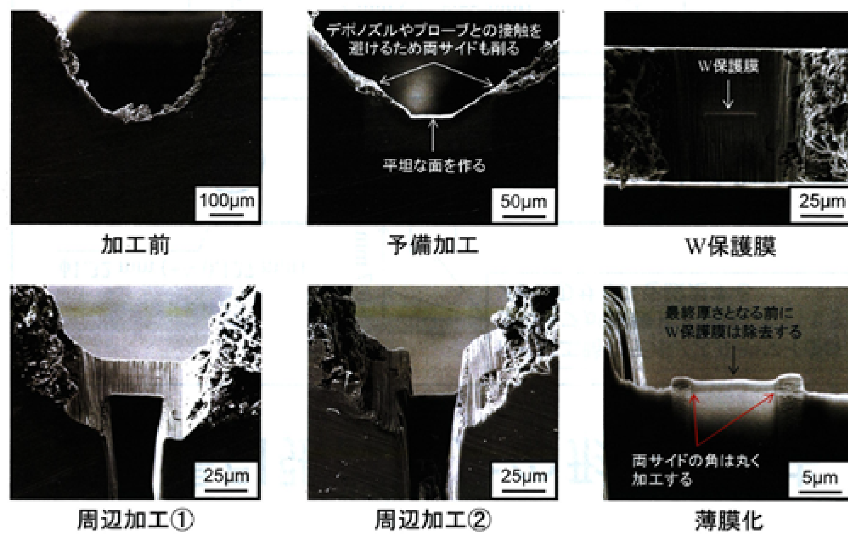


Fig. 2. FIB processing of the tip of the notch into electron transparent region.

図・表・数式 3
Figures, Tables and Equations 3

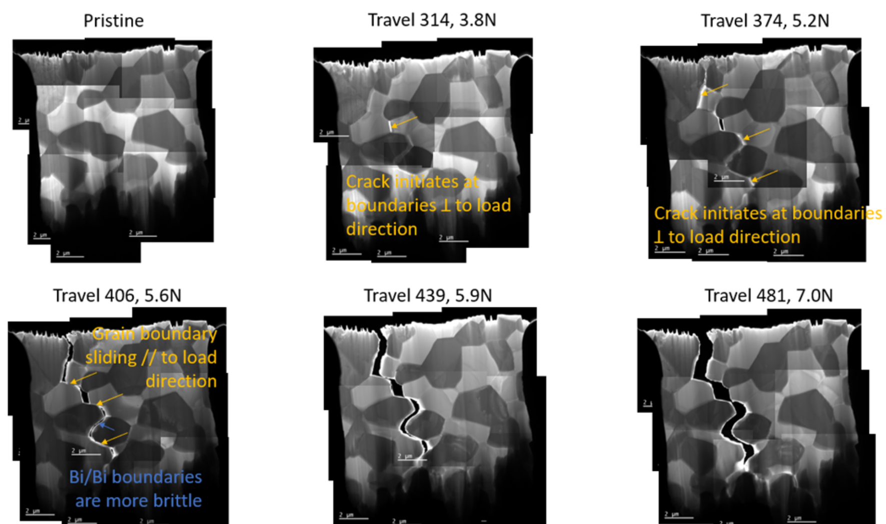


Fig. 3. HV-TEM energy-filtered images of the pristine sample and the sample under various tensile loads, showing the crack initiation sites and crack propagation route.

その他・特記事項（参考文献・謝辞等） Remarks(References and Acknowledgements)	

成果発表・成果利用 / Publication and Patents

DOI（論文・プロシーディング） DOI (Publication and Proceedings)	
口頭発表、ポスター発表および、その他の論文 Oral Presentations etc.	
特許出願件数 Number of Patent Applications	0件
特許登録件数 Number of Registered Patents	0件