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Sample size effects on grain boundary sliding

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ABSTRACT

Grain boundary sliding is an important deformation mechanism that contributes to creep and superplastic forming. In tin-based lead-free solders grain boundary sliding can make significant contributions to in service performance. Novel micro-compression tests designed to isolate individual grain boundaries and assess their mechanical resistance to sliding were conducted on tin. The boundary sliding deformation was more obvious for smaller sample cross-sections and made a larger contribution to the overall deformation. As with dislocation and twinning mediated plasticity there was a significant size effect in which the resistance to grain boundary sliding increases as the sample size is reduced.

1. Introduction

Grain boundary sliding can be a damaging process when it occurs during service loading but is also of great advantage when employed in superplastic forming in metals and ceramics [1–5]. It is well established that grain boundary sliding requires elevated homologous temperature and a relatively fine grain size. Tin-based alloys mostly based on silver and copper additions are the major group of materials developed as replacements for lead–tin eutectic based solders which are being replaced as a result of environmental concerns. At room temperature tin is already at ~55% of its relatively low melting point (505 K) and of course operating temperatures are even higher. β-Sn makes up the largest volume fraction in the majority of lead free solder alloys and grain boundary sliding has been identified as a significant deformation mechanism [6,7] especially when the grain size is small. Thermal cycling experienced by ball grid arrays in-service can lead to considerable changes within the initial microstructure leading to very inhomogeneous deformation and failure linked to strain localisation in regions where grain boundary sliding operates.

Significant grain size effect on the creep rate in grain boundary sliding and superplastic deformation of fine grained polycrystals is a well-known phenomenological observation with grain size exponents of −2 to −3 reported in metals and ceramics [2,4,5]. Observations on polycrystal ensembles prevent a clear separation of local driving force at a particular grain boundary and its resistance to sliding. The constraints of the three dimensional grain boundary network are prohibitively complex to unravel. However the observations of cooperative movement of groups of grains [4,8] give a clear indication that different grain boundary types have strongly differing resistance to grain boundary sliding. Work on grain boundary engineered Ni has shown that altering the geometrical nature of boundary types can dramatically enhance the overall resistance to creep and grain boundary sliding [9], while chemical doping of geometrically similar grain boundaries in ceramics has been shown to affect their propensity for boundary sliding [1]. The importance of grain boundary sliding in the deformation and failure of solder joints motivates this fundamental study of sliding at individual grain boundaries in β-Sn. Here we demonstrate the possibilities of using a focused ion beam to isolate a grain boundary segment within a small volume (few micron to sub-micron) which can be tested in compression (and shearing) using a nanoindenter.

Since the seminal work of Uchic et al. [10] there has been a huge growth in the use of mechanical testing at the micron and sub-micron length scales. The vast majority of the work has used compression pillar geometries [10–22], though work on cantilevers in bending [23–28], and tensile testing [29,30] at these length scales has been reported. Dislocation mediated plasticity in single crystal test pieces has been the most highly studied process for which face centred [10,13,14,17–19,22,28–32], body centred cubic [11,12,16], and hexagonal close packed metals [26,27] have been investigated. Deformation twinning has also received some attention in two hexagonal close packed metals Ti and Mg [20,21]. For dislocation mediated plasticity strong size effects are evident with the flow stress values increasing as test diameters d get smaller with exponents of approximately −2/3 often reported for the face centred cubic metals [13] while the exponent is −1 for both FCC and HCP in bending [27]. The stress required for the onset of deformation twinning has also been shown to be size dependent with a size exponent of −1 being reported in Ti [21]. Grain boundary sliding has not previously been tested using such small material volumes but length scale effects are to be expected.

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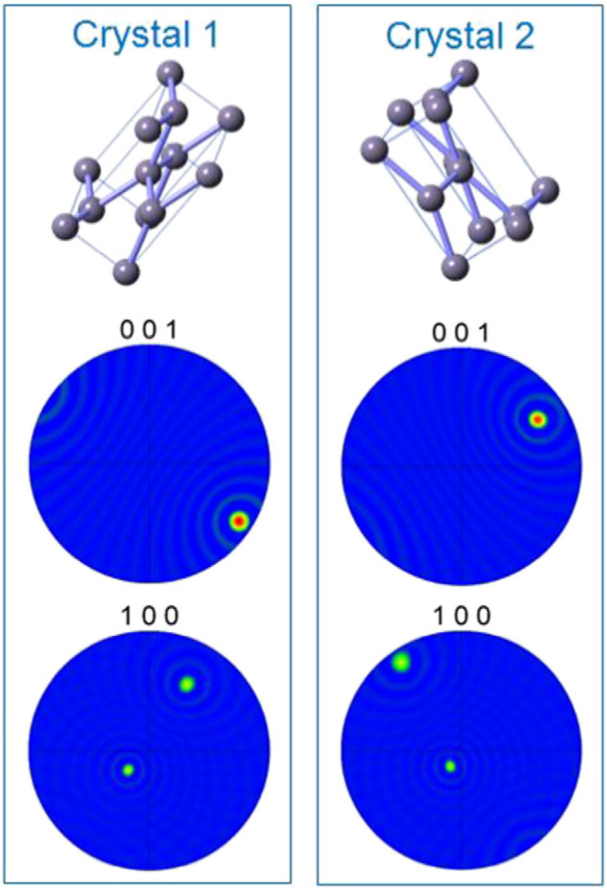

Fig. 1. Orientations of the two body-centred-tetragonal β-tin crystals. The unit cells (top) are shown as viewed along the compression axis which is at the centre of the pole figures (bottom) calculated from EBSD measurements and presented in stereographic projection.

2. Methods

Square cross section samples were prepared by focused ion beam (FIB) for compression testing using a nanoindenter with a flat diamond tip. The raw material was a high purity (99.99%) tin bar supplied by Goodfellow. The bar was cut into small blocks (5 mm × 5 mm × 10 mm), which were then mounted in resin. These pure tin samples were heated in the resin mounts to the molten state, and then slowly cooled down to the solid state to remove the effect of pre-deformation and cutting damage. Directional solidification was implemented to control the alignment of grain boundaries inside the sample blocks. After solidification, the sample surface was carefully polished using colloidal silica. Electron backscatter diffraction (EBSD) was used to map the crystal orientation and grain boundaries. Rectangular shaped micro-pillars with accurate controlling of the dimensions and the position of a boundary inside a pillar were machined on the selected grain boundary using a Zeiss Nvision 40 FIB-SEM system. The boundary was selected on the basis that it ran at ~45° to the sample surface normal which would become the compressive axis in the test pillars and so would have a large shear stress resolved upon it to promote grain boundary sliding. Micro-pillars of various dimensions were cut in each of the two crystals and spanning the grain boundary. The micro-pillar compression tests were conducted on a NanoXP nano-indentation platform equipped with a flat diamond punch which had been produced by FIB machining the end of a blunted Berkovich indenter tip. Although the indenter system is inherently load controlled the tests were conducted with the feedback system set to maintain a constant displacement rate during the tests. A target displacement rate of 20 nm/s was used for all but one slower rate test (2 nm/s) on a 2 μm wide bi-crystal pillar. A separate temperature control system was implemented to ensure that all the tests were conducted at 20 °C. The pillars were imaged using the SEM column of a Zeiss Nvision 40 FIB-SEM instrument.

3. Results and discussion

The test pieces had the selected grain boundary oriented at ~45° to the load axis and multiple test pieces of sizes from 5 μm, 2 μm, 1 μm, and 500 nm where prepared from the same grain boundary. The crystal orientations determined using EBSD are illustrated in pole figures (Fig. 1) which show that the c-axis was close (within 82°) to perpendicular to the load axis for crystal 1 and somewhat further away from this (within 73°) for crystal 2. The two crystals have a-axes that are within 18° and 26° of the load axis and these a-axes are only 8° apart. The rotation between the two crystals can be described approximately by a 65° rotation about an axis close to the almost shared [100] axis.

Micro-pillars of similar sizes were also cut from each of the two grains making up the bi-crystal pillars. Fig. 2(a and b) shows example engineering stress-displacement curves for compression tests for different pillar sizes on both single crystals and bi-crystal pillars. We prefer to use displacement rather than strain as deformation at the base of the pillars is not negligible and can lead to significant over estimation of the strain. The stress at the onset of major displacement events were obtained from such curves. For the smaller test piece sizes (2 μm, 1 μm, and 500 nm) abrupt load drops were seen as a result of significant displacement bursts but these are supressed at the largest pillar size. This is true

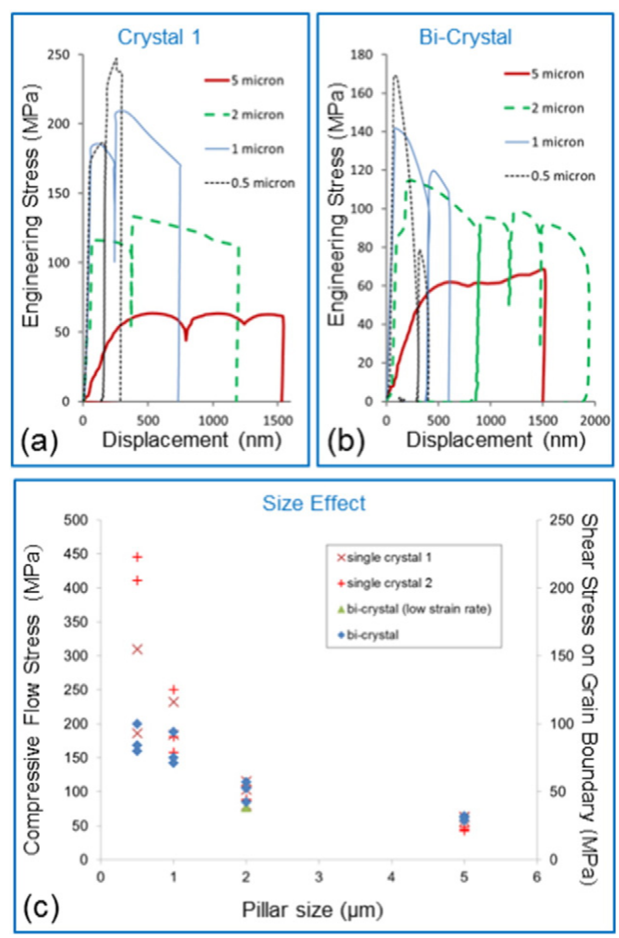

Fig. 2. Micro-mechanical data from single crystal and bi-crystal compression tests. Example engineering stress versus displacement curves at different sample widths for (a) single crystal 1, and (b) bi-crystals. (c) Compressive stress (left hand axis) required for plastic deformation as a function of sample width for single crystal and bi-crystal samples. For bi-crystals shear stress on the grain boundary is also indicated (right hand axis).