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Twinning interactions induced amorphisation in ultrafine silicon grains

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ABSTRACT

Detailed transmission electron microscopy analysis on a severely deformed Al-Si composite material has revealed that partial dislocation slips and deformation twinning are the major plastic deformation carriers in ultrafine silicon grains. This resembles the deformation twinning activities and mechanisms observed in nano-crystalline face-centred-cubic metallic materials. While deformation twinning and amorphisation in Si were thought unlikely to co-exist, it is observed for the first time that excessive twinning and partial dislocation interactions can lead to localised solid state amorphisation inside ultrafine silicon grains.

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1. Introduction

Due to the fast development in nanotechnology, ultrafine grained and nano-crystalline materials have been widely used because of their outstanding mechanical [1,2], chemical [3,4] and electrical properties [5,6]. As such, extensive efforts have also been devoted to understanding the deformation mechanisms in nano-crystalline materials [7,8].

Deformation-induced solid-state amorphisation (SSA) in crystalline materials have been observed [9–12], but have not been well understood compare to other mechanisms, such as dislocation slips [13,14], deformation twinning [15–17], and shear banding [18–20]. SSA is a predominant mechanism in ceramic crystalline materials deforming at or below room temperature [9,21,22]. Huang et al. found that high density dislocation accumulations in ball milled crystalline Si can eventually lead to localised SSA. Wang et al. performed high quality in-situ banding of Si nano-wires (NWs), and directly observed the SSA due to the creation of Lomer lock dislocation core and subsequent dislocation accumulation at the core. However, there has not been any evidence of SSA induced by deformation twinning interactions. Moreover, it is difficult to imagine the correlation between deformation twinning and SSA in ceramic materials, because conventionally twinning is only active at high temperatures (~500 °C) close to that around ductile-to-brittle transition [23,24]; on the other hand, amorphous Si are very likely to transform to crystalline structure at ~500 °C or higher temperatures [9,25].

Recent studies have shown that deformation twinning is very sensitive to size effect [8,26]. In many cases, deformation twinning becomes more predominant in nano-crystalline materials compared to their coarse grained counter-parts [16,27]. However, the grain size effect on twinning in ceramic materials is unclear. This paper will show, with direct evidence that deformation twining is an active deformation mechanism in ultrafine grained Si, and excess deformation twinning can eventually lead to localised SSA.

2. Experimental procedures

In this work, Al – 60 vol%Si composite ingot was fabricated by a series of powder metallurgy processes. Al powders (99.9% purity) and Si powders (99.9%) were mixed and cold pressed under the pressure of 100 MPa for 30 min. The mixture was then hot pressed under 50 MPa pressure and 600 °C for three hours in vacuum ($< 10^{-2}$ Pa). The fabricated ingot was cut and lathed into a cylindrical pin of a length of 24 mm and a diameter of 4.5 mm. The pin was loaded onto a CETR Tribometer to slide against an EN26 steel disc at a sliding speed of 0.209 m/s. The dry contact sliding test was performed at room temperature. A vertical load of 1.93 kgf was applied to the pin. The duration of each experiment was four hours to ensure a steady state contact sliding [27]. After the test, the pin was cross sectioned by a diamond saw and subsequently polished by diamond lapping films and slurry (0.4 μm

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grain size) to expose the plastically deformed microstructures parallel and perpendicular to the shear direction. Focused ion beam milling (Carl Zeiss AURIGA) was performed to prepare transmission electron microscopy (TEM) samples which were taken from the pin contact interface, with the aid of an ex-situ lift-out process.

A Carl Zeiss AURIGA scanning electron microscope (SEM) was used to perform electron backscatter diffraction (EBSD) analysis. The operating voltage was 12 kV. The step size was 50 nm. TEM characterizations were performed on a JEOL 2200FS field emission gun TEM operating at 200 kV, and a JEOL 3000F TEM operating at 300 kV.

3. Results and discussion

Fig. 1 is an EBSD image showing the Si grains of the Al-Si composite after a dry contact sliding test. The part above the dotted line is the damaged microstructure due to contact sliding and the part below the dotted line is the unaffected region with its microstructure corresponding to the as-received sample. The black areas between the coloured Si grains are the Al phase. In the as-cast sample, the Si grains have an average size of 1.5 μm within the range of 0.4–15 μm. However, within the subsurface above the dotted line, Si grains are significantly refined to an average grain size of ~400 nm, in the range of 0.1–3.5 μm as has been confirmed by TEM. According to this image, nano-twins are hardly detected by EBSD, because of the relatively large step size of 50 nm.

A detailed TEM analysis confirmed that large quantities of nano-twins and stacking faults (SFs) exist in coarse Si grains, as shown in Fig. 2(a). This is because during the casting process, the material was hot pressed under 50 MPa pressure and 600 °C for three hours in vacuum ($< 10^{-2}$ Pa) which promoted deformation twining and SFs formation in Si [23,24]. Interactions between nano-twins and SFs are also evident as shown in Fig. 2(b). However, the common interactions are SFs transmit across SFs (marked by a white arrow in Fig. 2(b)) and SFs collide onto twin boundaries (marked by a black arrow in Fig. 2(b)). Neither crossing twins nor SF transmit across a twin boundary (TB) was observed. This is because the flow stress during the hot pressing process was not sufficiently high to drive twinning partials transmitting across TBs [28,29].

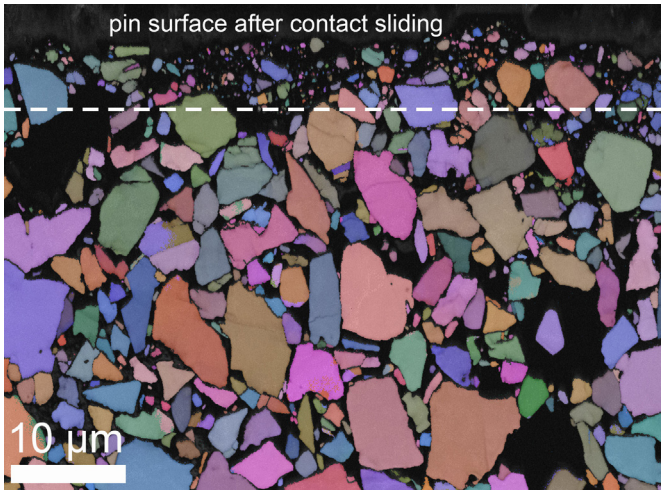

Fig. 1. A typical EBSD image showing the Si grain structures of the Al-Si composite after a dry contact sliding test. The top part of the image shows the contact sliding damaged microstructures. The bottom part of the image shows the unaffected region, where the microstructure corresponds to the as-received sample. The black areas between coloured Si grains are the Al phase.

Fig. 3(a) shows a Si grain located approximately 400 nm beneath the contact surface. This Si grain has a grain size of ~400 nm, surrounded by smaller Si and Al grains. There are three obvious twin bands across the whole grain, one of which is marked by a white arrow. The region pointed by a black arrow is magnified in Fig. 3(b). Twinning activities are very intense in this small region. T1 corresponds to the twin band marked by the white arrow in Fig. 3(a). White parallel lines mark the coherent TBs of T1. T2 is a twin band coming from the bottom of the image (initiated from the lower part of the grain boundary), inclined to T1. T3 is a twin band coming from the top of the image (initiated from the upper part of the gain boundary), inclined to T1. T4 looks like a twin band which is formed by the transmission of T3 across TB1 boundary, because it has almost the same width of T3, and it is adjacent to T3. Moreover, T1 is thinner on the left hand side of T4 than on the right hand side by a few atomic layers. This indicates that de-twinning has somehow occurred to T1 due to three possible causes: (1) the interaction between T2 and T1 [29,30]; (2) the interaction between T4 and T1 [29,31]; and (3) the successive partial dislocation emissions from the grain boundary on the left of the image [32,33]. Although de-twinning has been observed in face centred cubic (fcc) materials, the present finding is the first experimental evidence of de-twinning in diamond cubic Si, the unit cell of which can be regarded as two interpenetrating fcc unit cells, displaced along their body diagonal by one quarter of the diagonal length. Therefore, the de-twinning mechanism in fcc materials can be adopted to the diamond cubic Si.

Fig. 3(c) shows the detailed atomic arrangement at the intersection between T1, T3 and T4. A four-fold twin has formed by the transmission of T3 across the primary TB. The four TBs are marked by four white lines. According to literatures [29,34], the transmission of T3 across the TB will leave a series of sessile stair-rod dislocations on the TB. This is evidenced by the slightly distorted nature of the TB between T3 and T4. However, the distorted TB is not big enough to be recognised as an amorphous region. On the other hand, T4 initiates from the upper TB1 and ends on the lower TB1. Coincidently the end of T4 overlaps the region where T2 meets the lower TB1. Therefore, T1, T2 and T4 formed a junction where excessive partials/dislocations interactions occur. The junction is magnified in Fig. 3(d). The centre of the junction is a node of another fourfold twin. Three twin boundaries marked by the solid lines are clear, but the forth twin boundary marked by a dash line is no longer observable due to strong lattice disordering or amorphisation. It is known that twinning from various directions by a certain sequence will form a fourfold twin or even a fivefold twin in some metallic materials, with the TBs clearly observable [35,36]. This is because metallic materials could accommodate dislocations/interactions at much lower energies or stresses than ceramic materials [23,37,38]. In contrast, from the current observation, fourfold twin formation in nano-crystalline Si could cause amorphisation at the twin node, because extensive dislocations and twinning interactions will largely break the covalent bonds in the local area, and re-bonding could not occur without sufficient thermal energy input [9,38]. Moreover, the amorphised region in Fig. 3(d) is slightly shifted away from the node of the fourfold twin, but it is largely along the lower TB. This indicates that partials/dislocations having Burgers vectors parallel to the coherent TB planes (in this particular case parallel to TB1) play an important role in the amorphisation mechanism. In contrast, as shown in Fig. 3(c) the fourfold twin node has not been piled up with dislocations other than those formed during transmission of partial dislocations; therefore, there was not a distinguishable amorphous region at/near the twin node.

The above observations can lead to a microstructural evolution process of the fourfold twin formation and subsequent amorphisation at the twin node. The microstructural evolution process can

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