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Slip in directionally solidified Mo-alloy micropillars – Part I: Nominally dislocation-free pillars

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Abstract

In situ Laue analysis during microcompression reveals plasticity in [0 0 1]-oriented, directionally solidified Mo alloy pillars to start with slip on the {1 1 2}⟨1 1 1⟩ system having the highest Schmid factor followed by slip on the (1 1 0) plane containing the same Burgers vector. The results are interpreted in terms of the microstructure analyzed by scanning transmission electron microscopy and 3-D atom probe.

Keywords: X-ray diffraction; Microstructure; Dislocation; Microcompression; Three-dimensional atom probe

1. Introduction

Slip usually occurs in face-centred cubic (fcc) metals on the {1 1 1} crystallographic planes along ⟨1 1 0⟩ directions, and initiates on the plane with the highest resolved shear stress (i.e. follows Schmid's Law). In body-centred cubic (bcc) metals, however, slip has been observed on {1 1 0}, {1 1 2} and {1 2 3} planes in the ⟨1 1 1⟩ directions [1–3]. Moreover, bcc metals exhibit a strong temperature and strain rate dependence of the flow stress, which has been ascribed to the high Peierls stress resulting from the non-planar structure of the core of the screw dislocation [4].

The temperature dependence of flow stress is well documented in the literature. At temperatures well below a critical temperature T_c, the flow stress and plastic deformation mechanisms in bcc metals are primarily controlled by the glide of 1/2⟨1 1 1⟩ screw dislocations on {1 1 0} planes. Their mobility is low relative to that of edge dislocations having a planar core structure. Above T_c, the description of plasticity in bcc metals is simpler because the mobility of screw and edge dislocations become comparable and the deformation behaviour, including hardening mechanisms, become fcc-like, i.e. the selection of slip system is primarily determined by the Schmid factor.

At intermediate temperatures, the slip plane on which screw dislocations move has been the subject of long-standing debate. Discussions regarding whether it is the {1 1 0} or the {1 1 2} plane have kept scientists busy for many years [5–13]. One of the points of contention has been the influence of the resolved shear stress, or the Schmid factor, on the selection of the slip plane. Recently, slip behaviour in bcc Mo has been extensively addressed by atomistic simulations [5], and the formation of elementary slip steps on {1 1 0} planes has been confirmed. There are, however, studies which show that, depending on the specific boundary conditions applied to the simulation box, a dislocation could preferentially slip on the {1 1 2} planes [8].

With the development of the microcompression technique [14], testing of single crystals became attractive again, especially after a "smaller is stronger" trend was

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revealed in single-crystal pillars with diameters below $\sim$10 μm. This size effect, often represented by a power law between pillar diameter and strength, appears to be different for fcc and bcc metals [15–19]. A study performed on bcc single-crystal samples with the compression axis oriented in the [001] direction suggests that the slope of the power law becomes less steep (approaching that of fcc pillars) when the ratio between the test temperature (298 K) and the critical temperature T_c decreases from 0.82 (Nb), to 0.62 (Mo) and then to 0.32 (W) [15]. The differences in the size effects of fcc and bcc pillars are inferred mainly by comparing their stress–strain curves. This poses some experimental difficulties since strength is determined at strains as high as 5% (because of the uncertainty in measurements at smaller strains), and the stress–strain curves are usually jerky and often exhibit large strain jumps. Analysis of the slip lines on the surface by scanning electron microscopy (SEM) after deformation to large strains is used to characterize their nature [15,16,18]. Such observations have reported wavy slip lines for metals with high T_c or the presence of differently oriented slip lines. No clear correlation could be found between the slip trace analysis and the size effect [18]. Additionally, since a single major slip burst visible on the pillar surface does not exclude the activation of multiple slip systems internally, slip trace analysis can provide only limited information on the deformation behaviour.

On the other hand, several studies have demonstrated that factors other than just the size of the pillar influence the deformation behaviour of single-crystal pillars, e.g. the presence of defects before deformation, including dislocations or defects introduced by the focused ion beam (FIB), the method most frequently used for making these pillars. A study of Mo-alloy pillars obtained by directionally solidifying a NiAl–Mo eutectic alloy [20] has demonstrated that such pillars show a whisker-type deformation, reaching an yield stress of $\sim$9 GPa and no size effect on strength for pillars in the size range 360–1000 nm [21]. This high yield stress decreased dramatically when the material was pre-deformed [22] or when the surface was treated by FIB [23].

As a contribution to the understanding of plasticity in bcc micropillars, in situ Laue microdiffraction during compression has been performed to investigate the character of slip activated during the early stage of plastic deformation. This paper reports the slip systems activated in nominally defect-free Mo pillars, obtained by directional solidification. They are made free standing using two procedures, the first based on a combination of FIB and etching, including a final electropolishing step as described in Ref. [24], and the second using a lithographic technique. The pillars were deformed in situ during Laue diffraction [24,25]. The observed slip systems are discussed in terms of the microstructure of these pillars explored by transmission electron microscopy (TEM) [26] and 3-D atom probe. How plasticity is affected by the presence of defects introduced before the in situ deformation is presented in a companion paper. In the second paper Mo pillars that have either a pre-existing dislocation content due to deformation of the bulk composite, or contain defects because they were made by FIB, are studied in situ.

2. Experimental

2.1. Sample preparation

Mo-alloy single-crystal pillars with cross-sectional areas between 0.8 and 1.2 μm^2 and a height of $\sim$2.3 μm were produced from a directionally solidified NiAl–Mo eutectic alloy [20–22] utilizing two procedures, the first based on a combination of FIB and etching, including a final electropolishing step as described in Ref. [24], and the second using a lithographic technique.

The sample preparation method reported in Ref. [22] was used to produce the NiAl–Mo composite material. As discussed in Ref. [22], the in situ Laue microdiffraction is performed in the transmission mode, which requires the presence of a single row of pillars. Briefly, the procedure used to produce such pillars is as follows. Discs $\sim$0.4 mm thick were cut out from the composite by using electrodischarge machining followed by polishing on both sides. Wedge polishing was used in order to reduce the top width down to $\sim$20 μm. Using FIB milling, free-standing islands containing seven fibres each were created on this thinned surface. Since the Mo-alloy fibres grow in an approximately hexagonal pattern [20], the seven-fibre islands consist of six outer fibres and a central fibre in the middle of the hexagonal arrangement. During this step FIB redeposition occurred, leading to a NiAlMo amorphous-like layer on the surfaces of the islands. This layer prevented chemical etching of the NiAl matrix, which is what has been used in the past to obtain free-standing Mo pillars [21,22]. Therefore, a 300 ms flash electropolishing was applied using a solution of 12.5 H_2SO_4 and 87.5 methanol (vol.%) to remove the redeposited layer. This polishing step, while it removed the redeposited layer, unfortunately also preferentially attacked the Mo-alloy fibres, resulting in them having concave tops. As a last step an etching solution of 74 H_2O, 18 HCl and 8 H_2O_2 (vol.%) was used to chemically remove the NiAl matrix and obtain a Mo pillar aspect ratio close to 1/2.5 (diameter/height). In each island containing seven such pillars, only the pillar in the centre was kept; the outer ones were bent out of the way with a micromanipulator operating in situ in a high-resolution scanning electron microscope.

The electropolishing step described above resulted in pillars with a concave top as shown in Fig. 7a due to faster removal of Mo than the NiAl matrix. For comparison, pillars with flat tops were produced by a multistep lithography technique. Starting from a lamella of the NiAl–Mo composite with a thickness of 300 μm, the top width of the lamella is reduced down to 3 μm by reducing sequentially the size of the mask from 100 to 20 μm. Between each step the lamella was electropolished and its thickness controlled by transparency through the mask in the light