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Acta Materialia 60 (2012) 4614–4622

Acta MATERIALIA

www.elsevier.com/locate/actamat

Slip in directionally solidified Mo-alloy micropillars—Part II: Pillars containing defects

J. Zimmermann [a,b], H. Van Swygenhoven [a,b,*], S. Van Petegem [a], C. Borca [c]

[a] *Materials Science and Simulations, NUM/ASQ, Paul Scherrer Institut, CH-5232 Villigen, Switzerland*
[b] *Institut of Materials, Ecole Polytechnique Fédérale de Lausanne, CH-1012 Lausanne, Switzerland*
[c] *Swiss Light Source, Paul Scherrer Institut, CH-5232 Villigen, Switzerland*

Abstract

In situ Laue analysis during microcompression is carried out on Mo-alloy pillars containing defects due to focused ion beam milling or pre-strain. Independently of how the defects are introduced, if the diffraction peaks are streaked prior to deformation, slip starts in the direction matching the observed streaking, even if this corresponds to a low Schmid factor. The amount of pre-strain influences the deformation behaviour in terms of hardening and strain bursts.

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

1. Introduction

Plastic deformation in [0 0 1]-oriented, directionally solidified Mo-alloy pillars starts with slip on the $(1\,1\,2)\langle 1\,1\,1\rangle$ slip system having the highest Schmid factor, followed by slip on the $(1\,1\,0)$ plane containing the same Burgers vector. The reason that slip does not start on a $(1\,1\,0)$ plane may be related to an alloying effect reducing the critical temperature T_c or to the presence of nanoscaled face-centred cubic (fcc) precipitates. This was discussed in Ref. [1], the first part of the study. Here the influence of pre-existing defects on the slip behaviour of Mo alloy pillars is studied. The pre-existing defects are introduced either by focused ion beam (FIB) milling or by pre-deformation of the NiAlMo alloy composite prior to making the free-standing pillars.

Many published studies on single-crystal micropillars are performed on pillars that are FIB milled out of a single crystal or out of a large grain in a polycrystal [2,1,3–6]. Transmission electron microscopy (TEM) studies carried out on FIB-milled fcc and body-centred cubic (bcc) pillars have shown that such pillars usually contain defects prior to compression [7–12]. In situ Laue diffraction performed on FIB-milled pillars revealed the presence of rotational strain gradients, low-angle grain boundaries or a misorientation of the pillar body relative to the pillar base [13–15]. A recent in situ TEM study of bcc Mo pillars revealed the presence of long screw dislocations in 100 nm FIB-milled pillars which evolved to a complex dislocation structure when the pillar was deformed [12].

The Mo-alloy pillars obtained via directional solidification are attractive for studying the effect of pre-existing defects/dislocations since they are initially defect free and dislocations/defects can be added by pre-straining or FIB milling. It has been shown that, both after FIB milling and moderate pre-straining, the whisker-like behaviour of the as-grown pillars is lost and the typical scatter of the flow stress usually observed for FIB-milled pillars is reproduced [16,17]. After large pre-strains the scatter vanishes and the pillars exhibit no size effect [16]. These findings show that there is a link between the size effect and the initial microstructure of the pillar and that FIB milling influences this microstructure.

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* Corresponding author at: Materials Science and Simulations, NUM/ASQ, Paul Scherrer Institut, CH-5232 Villigen, Switzerland. Tel.: +41 56 3102931; fax: +41 56 3103131.
E-mail address: helena.vs@psi.ch (H. Van Swygenhoven).

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doi:10.1016/j.actamat.2012.02.042

Here microcompression during in situ Laue diffraction is performed on Mo-alloy pillars that were 11% pre-strained in the bulk composite and pillars that were FIB milled either from an already free-standing pillar or directly FIB milled out of the undeformed composite.

2. Methods and samples

In order to prepare pre-deformed pillars, the composite material was cut into discs by electrodischarge machining (EDM) and pre-strained by compression in the longitudinal direction of the fibres to ∼11% plastic strain. In order to avoid extensive cracking of the NiAl matrix, the discs were restrained laterally by being inserted into a copper ring. Free-standing pre-deformed pillars were then prepared using a combination of FIB milling and electropolishing as described in Ref. [1]. In this paper, results from two lamellae, each containing two pillars, are presented (see Table 1).

Two preparation routes were used for preparing FIB-milled pillars from the undeformed composite. The first FIB technique involved milling the pillar directly from the bulk NiAlMo composite using annular milling. First, a thin lamella is produced using mechanical polishing. This lamella is subsequently electropolished in order to remove any artificially introduced defects. The pillars were then stepwise milled while taking care that the centre of the annular shape was always the Mo fibre of interest. Currents ranging from 3 nA to 10 pA were used sequentially for the different steps, as explained in Fig. 1. This figure also shows the final shape of the pillars imaged by scanning electron microscopy (SEM) in side and top view.

It is important to note that when FIB milling is done directly on the bulk composite, the fibres cannot be perfectly aligned relative to the ion beam since their precise orientation below the surface is not known prior to the milling process. This resulted sometimes in a difference between the crystallographic orientation of the longitudinal axis of the Mo fibre and the crystallographic orientation of the vertical pillar axis. Three pillars were prepared from the same lamella using this technique. The in situ deformation behaviour of these pillars was very similar and therefore only the results from one pillar are presented here (FIB-3).

In the second FIB technique, the NiAl matrix was etched in order to obtain a row of Mo pillars prior to FIB milling. From these, a few pillars were selected and aligned relative to the incoming ion beam. The Mo fibres surrounding the selected pillars were milled along with a part of the matrix in order to leave the selected pillars free-standing on a pedestal, as shown in Fig. 2b. Finally the diameter of the Mo pillar of interest was reduced by using an ion current of 10 pA for the entire milling process. A particular technique was developed to reduce the formation of taper (see Fig. 2a). First a thin ring in the pillar interior is annular milled with a depth of approximately 2/3 of the pillar height, resulting in a relatively vertical cut around the pillar. Then the outer part was removed again by using annular milling.

The resulting pillar shape is presented in Fig. 2b for FIB-1, which shows a reduced taper compared to pillars milled with the conventional annular milling shown in Fig. 1. The reduced taper facilitates comparison of the data from the as-prepared and 11% pre-deformed pillars that were made without FIB milling. Two pillars prepared using this FIB technique, FIB-1 and FIB-2, are presented in this study.

Table 1 lists all the samples that were deformed in situ and which are presented in the Results section, along with their sizes and their initial misorientations relative to the [0 0 1] direction. When two pillars are on the same lamella, their names are shown in the same colour. The diameter of the FIB-milled pillars was measured at a point located one-quarter of the pillar height from the top of the pillar, which is also the region where most of the slip traces after deformation were observed. The heights and cross-sectional areas of the pillars are also provided in Table 1.

Note that the pre-deformed pillars have a slight concave top, similar to the as-grown pillars presented in Part I [1], whereas the FIB milled pillars exhibit a flat top.

3. Initial microstructure

3.1. Initial microstructure of 11% pre-deformed pillars

Table 2 provides a summary of the mechanical data and the Laue footprints of the initial microstructure. A large variation in the magnitude of peak broadening and streaking was observed for the four 11% pre-deformed pillars. The second row in Table 2 shows for each pillar the angular plot of the diffraction peaks that exhibit the largest and

Table 1
Overview properties of deformed pillars.

Type	Sample name	Initial offset from [0 0 1]	Cross-section (μm^2)	Height (μm)	Diameter (μm)
11% Pre-deformed	Pre11-1	5.8°	1.76	2.41	1.50
	Pre11-2	6°	2.05	3.50	1.62
	Pre11-3	2.5°	1.69	2.45	1.47
	Pre11-4	4.7°	2.02	3.95	1.60
FIB milled	FIB-1	1.9°	0.36	1.23	0.68
	FIB-2	3.45°	0.31	1.24	0.63
	FIB-3	10°	0.54	2.30	0.83