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ScienceDirect

Acta Materialia 62 (2014) 225–238

Acta MATERIALIA

www.elsevier.com/locate/actamat

Microshear deformation of gold single crystals

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Received 10 June 2013; received in revised form 3 September 2013; accepted 1 October 2013
Available online 30 October 2013

Abstract

We perform microshear experiments on Au single crystals, directly imposing shear loading on the microscopic crystallographic $\langle 1\,-1\,0\rangle$ $\{1\,1\,1\}$ slip system. We use a focused ion beam machined micro-double shear specimen which we load with a flat punch indenter inside a scanning electron microscope. Our method yields reproducible mechanical data (e.g. critical shear stresses of 63.5 ± 2.5 MPa). We study small-scale plasticity up to high strains (>50%) at constant slip geometry and document localized plastic deformation and sudden plastic deformation events. Strain bursts are observed, which can be related to the formation of new shear bands. Alternatively, they can result from sudden shear strain accumulation events in existing shear bands. Due to the stochastic nature of plastic deformation, the nature and the number of strain bursts can vary. We show and discuss how our in situ test technique captures these effects and how this affects the corresponding load–displacement curves. We discuss the advantages and inconveniences of our microshear test technique compared to other small-scale testing methods and relate our mechanical results to previous results reported for the micromechanical behavior of Au.

Keywords: Microshear specimens; In situ SEM testing; Localized plastic deformation; Strain bursts; Au single crystals

1. Introduction

Today there is a strong interest in the mechanical properties of small material volumes. In the present work we present a new scanning electron microscope (SEM) in situ microshear test technique as an alternative method to other small-scale testing methods. Systematic research on small-scale plasticity started in the 1950s [1,2]. The high strength of small specimens was shown to be associated with low densities of dislocations and/or with specific arrangements of dislocations [3,4]. The progress in micromachining in the 1980s led to the development of microelectromechanical systems (MEMS) for sensor and actor applications in microengineering and in medicine, where mechanical strength is an issue (e.g. [5]). The increasing interest in nanotechnology in the last two decades (e.g. [6]) has been another driving force for a growing interest in small-scale mechanical testing methods. Systematic studies on the mechanical properties of thin films [7] and on nanoindentation [8] have stimulated interest in the materials science community. The rapid success of focused ion beam (FIB) systems since the mid-1970s led to their use for micromachining of small specimens. Especially, the combination of FIB pillar machining with flat punch nanoindentation opened a new field of research [9,10].

Nano- and micromechanical experiments provide important information which cannot directly be retrieved from mechanical experiments on the macroscale. They provide benchmark data for materials modelers, because the mechanical behavior of a tiny specimen like a micropillar can be directly rationalized by molecular dynamic (MD) simulations (e.g. [11]) or by discrete dislocation modeling (DDM) methods (e.g. [12]). Moreover, materials scientists are interested in assessing the local properties of complex multiphase materials using nano- and micromechanical test

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1359-6454/$36.00
http://dx.doi.org/10.1016/j.actamat.2013.10.002

techniques (e.g. [13]). And, last but not least, in situ deformation techniques allow the direct observation of elementary processes which govern dislocation plasticity and fracture events (e.g. [14,15]). Nanoindentation (e.g. [16,17]), micropillar compression (e.g. [9,10,18,19]), microbending (e.g. [20,21]) and microtensile testing (e.g. [21,22]) are well-established micromechanical test techniques. Today, the field is covered in specific textbooks (e.g. [16]) and has been reviewed at different stages [23–25]. Materials scientists are well aware of the fact that the initial dislocation content of a microspecimen is important [26–29] and FIB micromachining was shown to introduce high dislocation densities into microspecimens which can affect the mechanical behavior [30].

The investigation of microspecimens with the μLaue technique had a considerable impact on the field [31–35]. It was shown that this technique is well suited as an in situ method which allows the change of slip geometry to be monitored and the study of the evolution of the dislocation substructure during plastic deformation of micropillars [33], microtensile tests [34] and microbending experiments [35]. However, there is one drawback associated with these microtest methods. During plastic deformation of microtensile experiments and micropillars, they change their slip geometry, as has first been described by Schmid and Boas [36] for macroscopic single crystal tensile specimens ("Wurstscheibenmodell"). Recent evidence for this pronounced change in specimen geometry was presented for micropillar compression [33] and for microtensile experiments [34]. Moreover, at higher accumulated plastic strains, micropillars show barreling [10].

Here we explore microshear testing as a method which has so far not received much attention in the micromechanics community. This appears unfortunate for two reasons: (1) from a mechanical point of view, shear testing represents a simple case of multiaxial loading with $\sigma_1 = -\sigma_3 \neq 0$, $\sigma_2 = 0$ (e.g. [37]). Shear data can therefore serve as benchmark data for constitutive models and for calculations performed with the finite element method (FEM). (2) And from a physical point of view, dislocation glide processes are driven by shear stresses which act in crystallographic glide planes and therefore microshear tests allow microscopic crystallographic slip systems to be directly activated (e.g. [38]). Recently a micro-double shear specimen geometry was proposed [39] and a first feasibility study was performed using FIB machined Cu single crystals. One microscopic crystallographic slip system could be directly activated and it was shown that the slip geometry does not change during loading. However, tip positioning on the shear specimen was challenging; the quality of the resulting load–displacement curves was not sufficient to interpret the mechanical behavior on a microstructural basis. In the present study, we use the specimen geometry proposed previously [39] to perform in situ microshear experiments in the SEM using FIB milled gold single crystals. We pay attention to the effect of the FIB milling parameters comparing high and low acceleration voltages. We directly load the microscopic crystallographic slip system $\langle 1\,{-}1\,0\rangle\ \{1\,1\,1\}$ and document the reproducibility of the mechanical data. We interpret the resulting load–displacement curves on the basis of a scenario which invokes stochastic sudden localized deformation events. We discuss the advantages and inconveniences of our microshear test technique as compared to other small-scale testing methods. And we use our results to contribute to a better understanding of the stochastic nature of microplasticity of gold and discuss our findings in the light of previous results.

2. Experiments and methods

2.1. Microshear specimen design

Pure shear corresponds to a stress state where a specimen volume is subjected to two equal and opposite forces without bending. In reality, pure shear loading cannot be fully realized because a shear specimen always represents a beam [40]. It is convenient to use double shear specimens because it is easy to introduce shear stresses through the application of vertical loads. Mayr et al. [41] developed a double shear specimen for shear creep testing of super alloy single crystals at temperatures of up to 1100° (Fig. 1a). They explained that there are a number of conflicting requirements which need to be considered. A large shear width W, for example, is desirable because it yields large shear displacements. But this benefit must be paid for by an increase of superimposed bending stresses. A large shear zone dimension, H, in the direction of loading minimizes the superimposed bending stresses. But, at the same time, it results in higher shear areas and increases the force which is required to deform the specimen. Mayr et al. [41] found a best compromise for a double shear specimen geometry, which is shown in Fig. 1a (all dimensions in mm). Each shear zone had a depth D of 3 mm, a height H of 8 mm and a width W of 2 mm. Applying a load P resulted in an engineering shear stress τ_0 given by:

$$\tau_0 = \frac{P}{2 \cdot H \cdot D} \tag{1}$$

With the measured shear displacement Δ, the shear γ is obtained as:

$$\gamma = \frac{\Delta}{W} \tag{2}$$

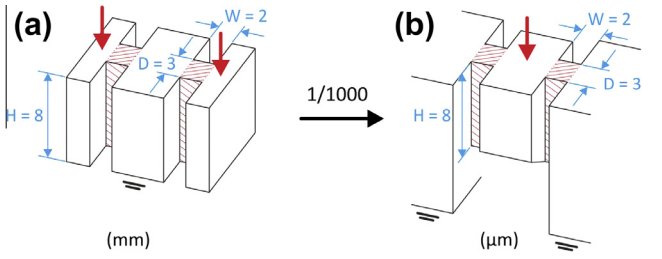

Fig. 1. Shear specimen geometries. (a) Macro-double shear specimen for high temperature designed by Mayr et al. [41] (dimensions given in mm). (b) Micro-double shear specimen used in the present study (dimensions given in μm).