Materials Science & Engineering A 665 (2016) 171–174

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

Interpretation of unloading tests on nanocrystalline Cu in terms of two mechanisms of deformation

Wolfgang Blum [a,*], Philip Eisenlohr [b], Jiangjiang Hu [c]

[a] Dep. of Materials Science and Technology, Friedrich-Alexander-University Erlangen-Nuremberg, Martensstr. 5, D-91058 Erlangen, Germany
[b] Dep. of Chemical Engineering and Materials Science, Michigan State University, East Lansing, MI 48824, USA
[c] Advanced Materials Research Center, Northwest Institute for Nonferrous Metal Research, Xi'an 710016, China

ARTICLE INFO

Article history:
Received 6 March 2016
Received in revised form
5 April 2016
Accepted 6 April 2016
Available online 7 April 2016

Keywords:
Crystal plasticity
Unloading
Nanocrystalline
Cu
Recovery strain
Dislocations

ABSTRACT

Hu et al. [1] reported the strains measured during unloading of nanocrystalline Cu in the course of deformation at room temperature. The inelastic strain rates during unloading are found to be larger than the quasi-stationary rates. This remarkable result is interpreted in terms of recovery strain acting as a second deformation mechanism in addition to the primary mechanism of glide of free dislocations.

1. Introduction

Recently, Hu et al. [1] reported the results of unloading tests on nanocrystalline (nc) Cu. The results were interpreted in terms of thermally activated glide of dislocations with certain activation volumes and 'internal stress'. The aim of this note is to suggest an alternative explanation in terms of two different, cooperating mechanisms of strain generation, one being related with generation and storage of dislocations, the other one with recovery of defects. The alternative resolves problems in the existing interpretation of unloading test results on the basis of a single mechanism of flow.

2. Quasi-stationary deformation strength

The grain size of the nc Cu investigated by Hu et al. [1] at ambient temperature was measured in transmission electron microscopy (TEM) to be about $w_0 = (25 \pm 5)$ nm $\approx 100\,b$, where b is the Burgers vector length; it accounts for high-angle boundaries and low-angle boundaries, but not for the numerous twin boundaries inside the crystallites. Most probably, the fraction of low-angle boundaries is negligible so that the (initial) grain size d (spacing of high-angle boundaries) in the nc material is $d_0 \approx w_0$.

Fig. 1 displays the quasi-stationary (qs)[1] strength measured at an inelastic strain $\epsilon_{inel} \approx 0.03$ (Fig. 6a in [1]). The other two qs strength curves hold for significantly larger d_0. Although the d-values are not directly comparable due to experimental differences, they vary in the expected order of higher strength at lower d_0. The rate sensitivity of strength increases significantly with decreasing d. This is well known as consequence of increase of the athermal qs strength limit connected with lowering d (compare [2,3]).

3. Deformation strength during unloading

Three states near the qs strength displayed in Fig. 1 serve as starting points of unloading tests of Hu et al. The curves of inelastic strain ϵ_{inel}[2] as function of stress σ at constant time rate -33 MPa s$^{-1} \geq \dot{\sigma} \geq -100$ MPa s^{-1} of unloading reported in Fig. 4 of [1] were evaluated for the inelastic strain rate

[1] qs means that dynamic equilibrium of generation and recovery of crystal defects is nearly established for some, but not all microstructural parameters; in the present case of low strains < 0.05 the intragranular dislocation density and the defect structure at the boundaries presumably are qs, at nearly constant $d \approx d_0$

[2] plastic with possible anelastic contributions

* Corresponding author.
E-mail addresses: wolfgang.blum@fau.de (W. Blum), eisenlohr@egr.msu.edu (P. Eisenlohr), jiangjiangwho@gmail.com (J. Hu).

http://dx.doi.org/10.1016/j.msea.2016.04.013
0921-5093/

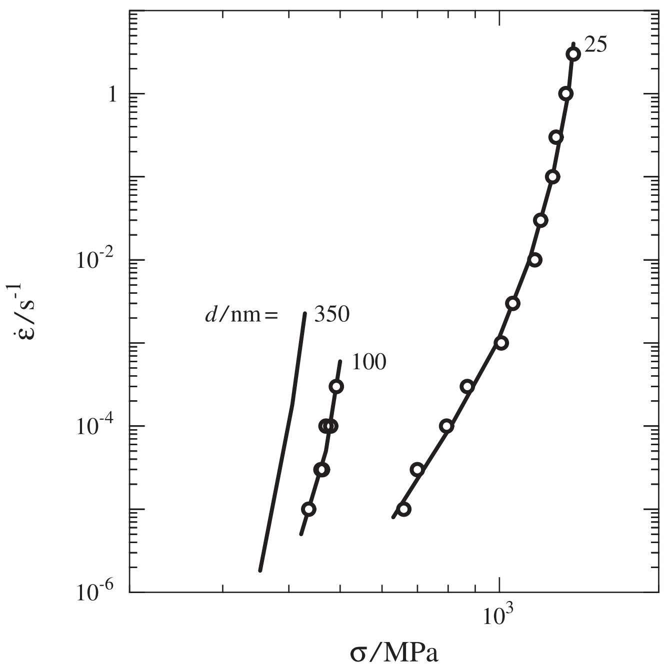

Fig. 1. Quasi-stationary (qs) strength in $\dot\epsilon_{inel}$–σ-field for ultrafine-grained Cu with $d \approx d_0 = 25$ nm evaluated from Fig. 6a in [1] at $\epsilon_{inel} \approx 0.03$ (or at σ-maximum) and constant rate $\dot\epsilon_{tot} \approx \dot\epsilon_{inel}$, $d \approx 100$ nm [4], and $d \approx 350$ nm [5]. The data points were evaluated by digitizing the published figures.

$$\dot\epsilon_{inel} = \frac{\Delta\epsilon_{inel}}{\Delta\sigma}\,\dot\sigma. \tag{1}$$

The curves of this figure were manually digitized such that the differences $\Delta\epsilon_{inel}$ and $\Delta\sigma$ of neighboring points along the curves were sufficiently large compared to the inaccuracies caused by data fluctuations or reading of data. In some cases, bundles of apparently similar curves taken under same experimental conditions were combined to get a reliable average of $\dot\epsilon_{inel}$. In this way the noise of $\dot\epsilon_{inel}$ was reduced to an acceptable level. The uncertainty of $\dot\epsilon_{inel}$ is estimated to be less than a factor of 2.[3] The results of graphical evaluation are shown in Fig. 2. As reported in [1], there is considerable forward inelastic strain when the deformed material is unloaded. This is evident from the positive values of $\dot\epsilon_{inel}$ in Fig. 2. They exceed the quasi-stationary rates (gray), although the material had undergone a maximum of work hardening before the stress reduction. Only at a stress ≈ 400 MPa (labeled $\sigma_{internal}$ in Fig. 2) the rates turn negative due to back flow of dislocations (Fig. 4 in [1]).

Fig. 3 shows the fits to the data plotted versus the relative stress $R \equiv (\sigma - \Delta\sigma)/\sigma$, where σ is the stress acting before the unloading by $\Delta\sigma$. The shape of the curves is very similar to that published by Milička for various materials [7–9]. With decreasing R there is a hump before the rates $\dot\epsilon_{inel}$ drop to zero and change their sign from positive to negative (from net forward to net back flow). It is known that the inelastic deformation after stress reduction to a constant level $0 \le R \le 1$ generally does not cease after the back flow, but becomes positive again for a certain time interval [10–15]. This behavior, found for single crystals and coarse-grained polycrystals, has also been detected in nc Ni [16,17] where it could be directly linked to dynamic recovery with the aid of in situ X-ray diffraction measurements. This behavior is qualitatively similar to what had been found before in materials with

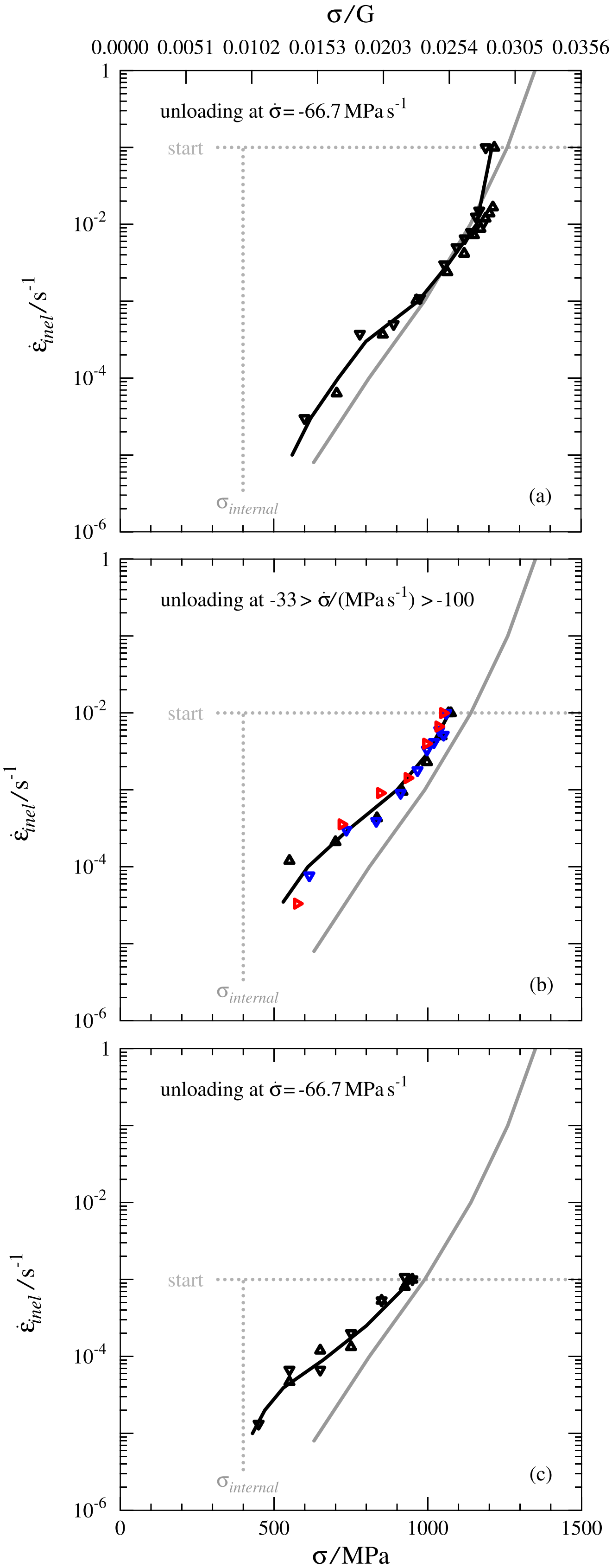

Fig. 2. Inelastic strain rate $\dot\epsilon_{inel}$ as function of stress σ during unloading from $\dot\epsilon_{inel}$ = (a) 10^{-1} s^{-1}, (b) 10^{-2} s^{-1}, (c) 10^{-3} s^{-1} at different $-\dot\sigma/(\text{MPa s}^{-1}) \approx 33.3$ (blue), 66.7 (black) and 100 (red); different symbols correspond to different unloadings; gray lines: quasi-stationary (qs) strength at $\epsilon_{inel} \approx 0.03$ and constant rate $\dot\epsilon_{tot} \approx \dot\epsilon_{inel}$ from Fig. 1. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

[3] A somewhat more precise and more objective method of getting noise-reduced $\dot\epsilon_{inel}$ from the original data is described in [6].