Acta Materialia 122 (2017) 259–265

Contents lists available at ScienceDirect

Acta Materialia

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

Full length article

Formation of dislocation channels in neutron irradiated molybdenum[☆]

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A R T I C L E I N F O

Article history:
Received 9 June 2016
Received in revised form
16 September 2016
Accepted 17 September 2016
Available online 8 October 2016

Keywords:
Neutron irradiation
Dislocation channeling
Defect clusters
Loop-dislocation interactions
Electron microscopy

A B S T R A C T

The formation of deformation-induced dislocation channels in neutron irradiated molybdenum and the loop-dislocation interactions were investigated using transmission electron microscopy. It is suggested that the irradiation-induced defect - microstructure is unstable with respect to the stress. Under the influence of the stress, loops start gliding on their glide cylinders in transition region of a cascade where vacancies and interstitials may coexist. Results indicate that loop-loop interactions have a significant role in the early stage of channel formation; dislocations are not seen either within or at the ends of the embryonic channels. There are two consequences of loop-loop interactions: the reduction in the loop density due to their mutual annihilation in localized regions and the increase in the loop sizes due to the coalescence of loops having the same nature. On further stressing, the segments of the larger loops could serve as the source of mobile dislocations. The yielding may occur when the stress level is high enough to activate the coalescence-produced glide sources. Furthermore, results on the attractive loop-dislocation interactions suggest that they may not have a significant role in eliminating defect clusters. On the other hand, the repulsive interactions may have a substantial role in altering the microstructure of the irradiated metals.

1. Introduction

It is well documented that the neutron irradiation of metals and alloys produces defect clusters. The structural changes in an annealed polycrystalline metal, Fig. 1 (a), on irradiation are schematically illustrated in Fig. 1 (b). An existing dislocation that is near screw orientation assumes a helical form and a large number of defect clusters have formed. The clusters result from the coalescence of excess interstitials and vacancies generated during the irradiation, and the cluster density is very high. An interesting question is how does the damage structure affect the mechanical properties of a material? There is a consensus that the yield points appear at low deformation temperatures; the yield strength is increased; and the ductility is reduced; see the recent review by Byun and Hashimoto [1]. One of the most remarkable features of deformed irradiated metals is that the deformation is not homogeneous, and the strain is localized. These regions are essentially free of defect clusters and are referred to as dislocation channels in the literature, and one of the channels containing a few dislocations and a large size defect cluster is shown in Fig. 1 (c). Cottrell surmised this in 1958 from slip line observations on the surfaces of deformed irradiated metals [2]. He suggested that the channels are defect-free regions and may be produced when the defect clusters are swept away by the gliding dislocations; the subsequent microscopy observations showed that the channels are almost defect-free. What a remarkable insight! The question is how do the channels evolve?

Since the sixties, a large number of studies were carried out to understand the formation of the channels. To delineate their salient features, for example see papers Downey and Eyre [3] and Eyre [4]. Recently, Byun and Hashimoto summarized the salient features of channels [1], and they are: the shear strain is confined to narrow bands; the grown-in dislocations between slip bands show no sign of having moved and are heavily jogged; the traces of channels are compatible with those of slip planes in the material; and the patches of dislocation debris are observed are in the channels, but defect clusters are largely absent. Recently, Brieceno et al. [5] examined the effect of ion irradiation-induced defects on the mobility of dislocations in stainless steel. They showed that the

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[☆] This work was carried out when the authors were associated with the Atomic Energy Research Establishment, Harwell, England.

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http://dx.doi.org/10.1016/j.actamat.2016.09.029

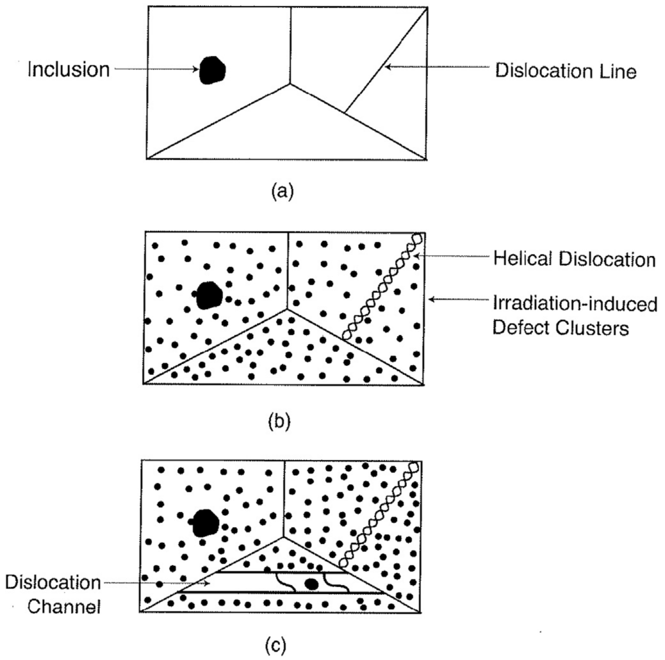

Fig. 1. Schematics showing: (a) an annealed polycrystalline metal with an inclusion and a near screw dislocation; (b) after neutron irradiation; dislocation assumes a helical form due to the absorption of point defects and a large number of defect clusters; and (c) after stressing the irradiated material; note the presence of a clear region that is referred to as a "channel".

dislocations which are mobile prior to irradiation get locked by the irradiation. Also, the mobility and motion of the dislocations are altered by the irradiation. This again implies that the pre-existing dislocations are pinned after irradiation. Furthermore, the study of Sharp [6] on the formation of channels in single crystal copper suggested that the grain boundaries are not needed as the sources of dislocations. The implication of the preceding discussion being that the mobile dislocations probably evolve during the deformation of irradiated metals.

There are three principal microstructural entities in the irradiated metals: isolated point defects; defect clusters and dislocations. The possible interactions that could lead to the change in damage structure are: loop-loop and loop-dislocation. The challenge is how to use these interactions to develop a mechanistic understanding of the formation of the channels. Cottrell's [2] suggestion initiated a number of studies on loop/dislocation interactions. Silcox and Hirsch [7] directly observed the interactions between moving dislocations and loops in aluminum. Several possibilities arose depending upon the relative Burgers vectors and the spatial relationship between the loops and the glide dislocations. Saada and Washburn [8] examined some of the loop-dislocation interactions and suggested that a loop is eliminated because it is chopped up by the moving dislocations. For this mechanism to work, the dislocation must have an attractive interaction with the loop. To date there is no experimental evidence in support of this mechanism.

Arakawa et al. [9] studied loop –loop interactions in iron using *in situ* transmission electron microscopy. They observed that a small loop of Burgers vector $\mathbf{b_1}$ is absorbed by a larger loop of Burgers vector $\mathbf{b_2}$, resulting in a larger loop with Burgers vector $\mathbf{b_2}$. It is conceivable that the loop-loop interactions have a major role in changing the landscape of irradiated metals during the early stages of deformation.

In the present study, the following questions were addressed. First, how are channels nucleated in irradiated materials on deformation? Second, what is the source of mobile dislocations? Third, what is the role of loop-dislocation interactions in the growth of channels? This work was carried out on neutron irradiated polycrystalline molybdenum. The results of this study constitute the present paper.

2. Experimental details

The molybdenum used in the present work had low carbon arc-cast material in stress relieved condition and was procured from the Climax Molybdenum Company. Discs, 3 mm in diameter, were fabricated from the as-received material. These discs were subsequently annealed at 2073 K for 6 h in a vacuum of 5×10^{-5} torr. The annealed samples were neutron irradiated in PLUTO to a fission dose of 5×10^{19} n. cm^{-2} at the ambient reactor temperature 333 K. To study, the loop-dislocation interactions, a coarse loop distribution was produced by annealing the irradiated samples at 1173 K for 1 h in a vacuum of 5×10^{-6} torr.

To study channeling, the irradiated specimens were deformed at room temperature up to the macroscopic yield level of 0.1% offset. The annealed samples were also deformed at ambient temperature to different strain levels.

The discs with different metallurgical histories were jet polished in a 15% nitric acid-water solution, followed by electro-polishing in a static bath containing 23 cm^3 of sulfuric acid and 75 cm^3 of methanol, maintained at 273 K. Thinned samples were examined by electron microscopy.

3. Results

Results of the present study will be divided into two groups. The first group will cover early stages of channel formation, whereas the second group will deal with the loop-dislocation interactions.

3.1. Early stages of channel formation

Fig. 2 shows packets of channels that appear to be in their early stage of formation. This micrograph was obtained from a sample that was strained up to the macroscopic yield point. The well-developed channel J is typical of what has been seen before in deformed irradiated metals. It shows a few dislocations and defect clusters. The features labeled E, F, H, and K that look like braids. The braids are segmented and appear to be on parallel planes that are displaced from each other. Assuming that the braids are precursors to the channels, it appears that the channel may widen by the blending of the braids, see region labeled K.

Fig. 2 (b) shows the same area as in Fig. 2 (a), but the operating reflection is different; it is [01-1] in (a), and [200] is in (b). Assuming that the Burgers vectors of the majority of the clusters are 1/2<111> [4], the 50% of the clusters should be out of contrast in Fig. 2 (a), but all of them should be visible in Fig. 2 (b). Comparing Fig. 2 (a) and (b), it is inferred that the cluster density in the braids is lower than that in the surrounding regions, and the clusters are not fully removed from the braids. Furthermore, dislocations are not observed either within or at the ends of the braids.

Fig. 3 shows a cluster-deficient region in a sample strained up to the macroscopic yield point; this is not a blow up of any of the regions from Fig. 2. Its salient features are: the clearing of defect clusters in an irregular manner; defect clusters of different sizes are observed and some of them are unusually large; and a few dislocation segments that are decorated with defect clusters are also seen. It is emphasized that such large loops were not observed in the as-irradiated samples [4].

Fig. 4 shows channels that are in different stages of evolution. The regions on the left and in the right hand corner represent standard channels that have been studied extensively in the