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Deformation microstructure of neutron-irradiated pure polycrystalline metals

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Abstract

The effects of neutron-irradiation near 80 °C on the deformation behavior of pure polycrystalline metals vanadium (body centered cubic, BCC), copper (face centered cubic, FCC) and zirconium (hexagonal close packed, HCP) have been investigated. Dislocation channel deformation is observed in all metals, and is coincident with prompt plastic instability at yield. Dislocation pileup was observed at grain boundaries in the deformed vanadium irradiated to 0.012 dpa, indicating that channel formation could lead to dislocation pileup and the resulting stress localization could be a source of grain boundary cracking. TEM analysis suggests that the loss of work hardening capacity in irradiated V, Cu, and Zr at higher doses is mainly due to dislocation channeling in local regions that experience a high resolved shear stress.

1. Introduction

Irradiation can produce dramatic changes in the mechanical properties of metals [1–5]. Early studies on irradiated metals [3,4,6] clearly demonstrated that irradiation at low temperatures (<100 °C) produced pronounced hardening. The hardening is typically accompanied by a severe decrease in uniform plastic elongation as measured in a uniaxial tensile test [1–3]. The decrease in tensile ductility associated with low temperature neutron irradiation was the topic of numerous studies performed in the 1960s, and the phenomenon was commonly referred to as low temperature radiation embrittlement. A more appropriate term for the low uniform elongation typically observed following low temperature irradiation is the loss of strain hardening capacity. A general feature associated with irradiation at low temperature is increased matrix hardness due to the presence of radiation-induced defects that act as obstacles to dislocation motion. A low dose, rapid hardening regime followed by a slowly evolving hardening behavior at higher doses has been observed in several FCC and BCC metals irradiated at low temperatures [6,7]. The present paper will focus on the deformation microstructure, especially dislocation channeling, in neutron-irradiated pure BCC, FCC and HCP metals in order better to understand the deformation mechanisms of materials showing loss of strain hardening capacity.

2. Experimental procedure

High purity polycrystalline vanadium (BCC), copper (FCC) and zirconium (HCP) were obtained from Alfa Aesar Cooperation. The chemical compositions and annealing conditions are shown in Table 1. Custom-designed sheet tensile specimens with a gauge section of 8.0 mm × 1.5 mm × 0.25 mm thick and an overall length of 17.0 mm were irradiated at the range of 65–100 °C in the High Flux Isotope Reactor (HFIR). The irradiation exposures ranged from 1.1×10^{21} to 6.3×10^{24} n m^{-2}, $E > 0.1$ MeV, corresponding to nominal atomic displacement levels of 0.0001–0.92 dpa. Further information on the experiment can be found elsewhere [7]. All tensile tests were conducted at room temperature at a crosshead speed of 0.008 mm s^{-1}, corresponding to an initial specimen strain rate of 10^{-3} s^{-1}. The 0.2% offset

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Table 1
Chemical composition (wt. ppm) and heat treatment of metals used

Metal	Chemical composition (wt%)
V	V–0.026Si–0.039Mo–0.027O (99.8% purity) Annealed at 900 °C for 30 min
Cu	Cu–1.6S–0.09Cr–0.22Ni–0.1P–0.27Fe4.8Ag Annealed at 450 °C for 30 min
Zr	Zr–0.0058Hf–0.0056Fe (99.94% purity) Annealed at 670 °C for 30 min

yield strength (YS), ultimate tensile strength (UTS), uniform elongation (UE), and total elongation (TE) were calculated from the engineering load-elongation curves. A summary of these tensile properties can be found in Ref. [8]. Rectangular pieces were cut from the gauge sections of the tensile-tested (broken) specimens for examination by TEM. Preparation of electrothinned foils from these small pieces in a Tenupol electropolishing apparatus required substantial development work and modification of Tenupol specimen holders, details of which are available in Ref. [7]. Electron microscopy observation was performed at Oak Ridge National Laboratory (ORNL) with a JEM-2000FX transmission electron microscope operating at 200 kV.

3. Results and discussion

The microstructure of all metals after irradiation to about 0.01, 0.1, and 1 dpa, are shown in Fig. 1. Neutron irradiation at low temperature produced a high number density ($\sim 10^{23}$ m^{-3}) of very small defect clusters ($\sim$3 nm) in all metals. In low stacking fault energy (SFE) FCC metals: Cu, approximately 90% of irradiation-induced defects are stacking fault tetrahedra (SFT), and the average SFT size remains constant at about 2.6 nm at higher doses. The neutron-irradiated BCC metal V in-

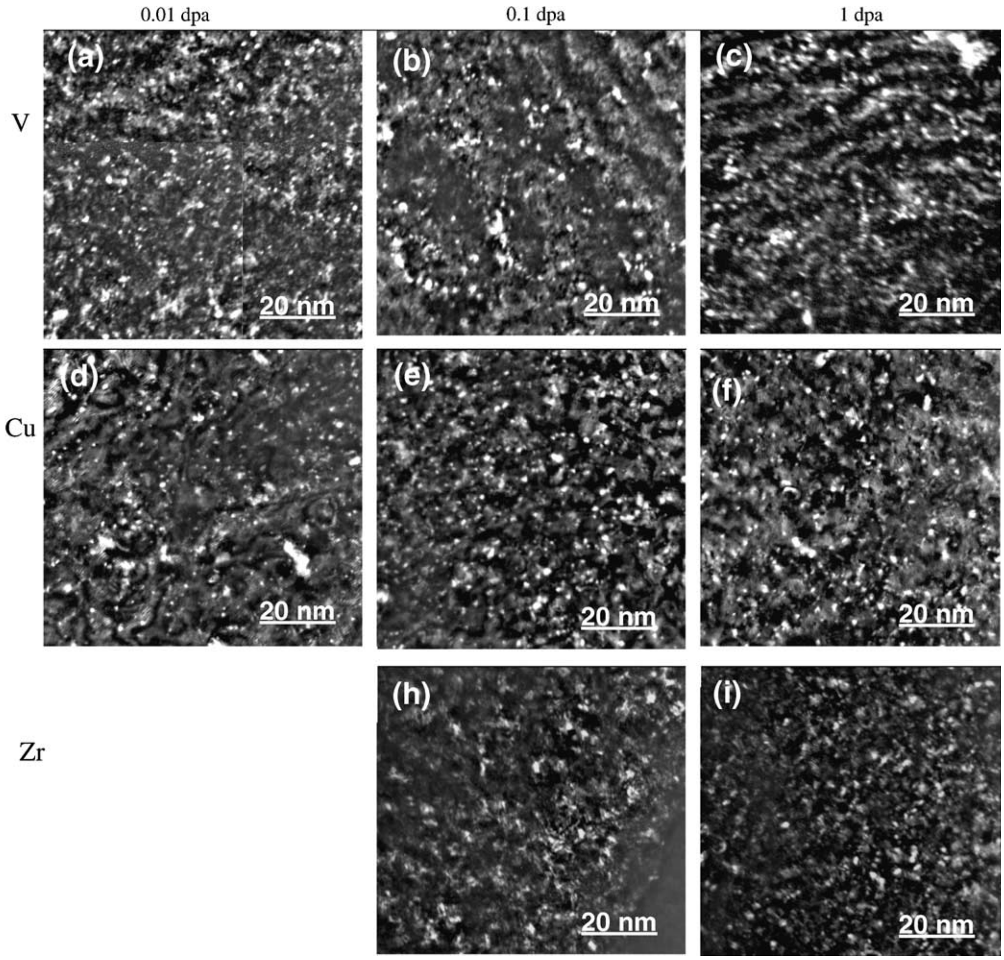

Fig. 1. Microstructure of irradiation-induced defect clusters in pure metals.

Table 2
Summary of defect cluster and tensile property in neutron-irradiated metals

Material	Irradiation	Defect cluster		
	Dose (dpa)	Density (m^{-3})	Size (nm)	$(Nd)^{1/2} \times 10^{-7}$
V	0.012	1.1×10^{23}	1.8	1.42
	0.12	1.9×10^{23}	2.1	2.00
	0.69	2.3×10^{23}	2.1	2.18
Cu	0.013	1.9×10^{23}	1.6	1.90
	0.13	6.7×10^{23}	2.6	6.70
	0.92	6.7×10^{23}	2.6	6.70
Zr	0.009			
	0.09	4.2×10^{22}	1.5	0.80
	0.63	6.5×10^{22}	2.1	1.17

cluded somewhat lower number density of smaller loops compared to Cu. In the case of HCP metal Zr, the number density of small loops (about 2 nm in size) is much lower ($\sim 7\times10^{22}$ m^{-3}) than that of Cu and V. There were no SFT observed in V and Zr. The character of the loops in V and Zr was not analyzed, but they are most probably of interstitial type. Measured defect cluster parameters are listed in Table 2.

From simple geometric considerations of a dislocation traversing a slip plane which intersects randomly distributed obstacles of diameter d and atomic density N, the increase in the uniaxial tensile stress for polycrystalline specimens ($\Delta\sigma$) in a pure metal is given by the well-known dispersed barrier hardening equation [5,9,10], $\Delta\sigma = M\alpha\mu b(\mathrm{Nd})^{1/2}$, where μ is the shear modulus, b is the magnitude of the Burgers vector of the glide dislocation ($a_0/\sqrt{2}$ for FCC and $\sqrt{3}a_0/2$ for BCC, where a_0 is the lattice parameter), M is the Taylor factor (3.06 for equiaxed FCC and BCC metals), and α is average barrier strength of the radiation-induced defect clusters. Based on this equation and the measurements carried out in this experiment, a value of $\alpha \approx 0.26$ and 0.20 was obtained for pure polycrystalline vanadium and copper, respectively. Recent experimental estimates range from $\alpha \sim 0.18$ to 0.27 for pure vanadium [11–14] and 0.10–0.25 for copper [15,16], austenitic stainless steel [10,17], and V–4Cr–4Ti [18] irradiated at low temperatures. In the case of Zr, $\alpha \approx 0.5$ was obtained ($b = \sqrt{5}a_0/3$ and $M = 3.06$ for HCP).

Dislocation channeling can be observed in Fig. 2 as narrow bands, which have been largely cleaned of the fine defect structure. In general, dislocation channeling begins to occur above a critical dose/hardening level (corresponding to a cluster density of $N >\sim 1 \times 10^{23}/m^3$ for copper tested at room temperature [15]). In V, dislocation channeling occurred even at low dose (0.012 dpa) because irradiation-induced defect clusters were present in sufficient density ($N > 1 \times 10^{23}/m^3$) for dislocation channeling. Deformed Cu and Zr exhibited channels only at higher doses (0.01 dpa) in this study. Dislocation channels tend to be present on $\{1\,1\,2\}$ and $\{1\,1\,0\}$ planes in V, $\{1\,1\,1\}$ in Cu, and $\{1\,0\,\bar{1}\,0\}$, $\{1\,0\,\bar{1}\,1\}$, $(0\,0\,0\,1)$ in Zr. The number of slip systems of BCC, FCC, and HCP is 48, 12, and 3, respectively. Furthermore, in HCP metal it is possible to have pyramidal glide (on $\{1\,0\,1\,1\}$, $\{1\,0\,1\,2\}$, $\{1\,1\,2\,1\}$, and $\{1\,1\,2\,2\}$) and/or prismatic glide (on $\{1\,0\,1\,0\}$) in some cases. Therefore, BCC and HCP metals could have the advantage to deform with cross slip motion compared to FCC metal. Actually, in this experiment, curved dislocation channels were observed in V and Zr (Fig. 2(b) and (i)), but not in Cu. The curvatures appear to arise from cross slip as moving dislocations change from one slip system to another.

Fig. 3 shows that some residual dislocations in V irradiated to 0.012 dpa moving in channels on $\{1\,1\,2\}$ planes still remained in the channels after tensile testing. The channels with dislocations are on $(1\,2\,1)$, $(2\,\bar{1}\,1)$, and $(\bar{2}\,1\,\bar{1})$ planes and the corresponding slip directions are $\langle\bar{1}\,1\,\bar{1}\rangle$, $\langle\bar{1}\,1\,1\rangle$, and $\langle\bar{1}\,\bar{1}\,1\rangle$, respectively. In Fig. 3(b), numerous dislocations lineup on the $(\bar{1}\,\bar{1}\,2)$ plane and their pileup at a grain boundary can be seen. Dislocation channeling has been suggested to induce grain boundary cracking in some irradiated BCC alloys [16]. The microstructure shown in Fig. 3 suggests that channel formation could lead to dislocation pileup and the resulting stress localization could be a source of grain boundary cracking.

In this experiment, rectangular shaped TEM samples (1.5×2.0 mm) were taken from the uniform strain region of the tensile-tested (broken) specimens in order to monitor the tensile axis orientation of the deformed gauge region in the TEM. With the long edge of the rectangular specimen set parallel to the TEM holder, the deformation channel orientation with respect to the tensile direction within the grain could be determined by the diffraction pattern. On the assumption that all the grains in the material deformed uniformly and grain rotation during deformation is negligible, the resolved shear stress for each dislocation channel observed was

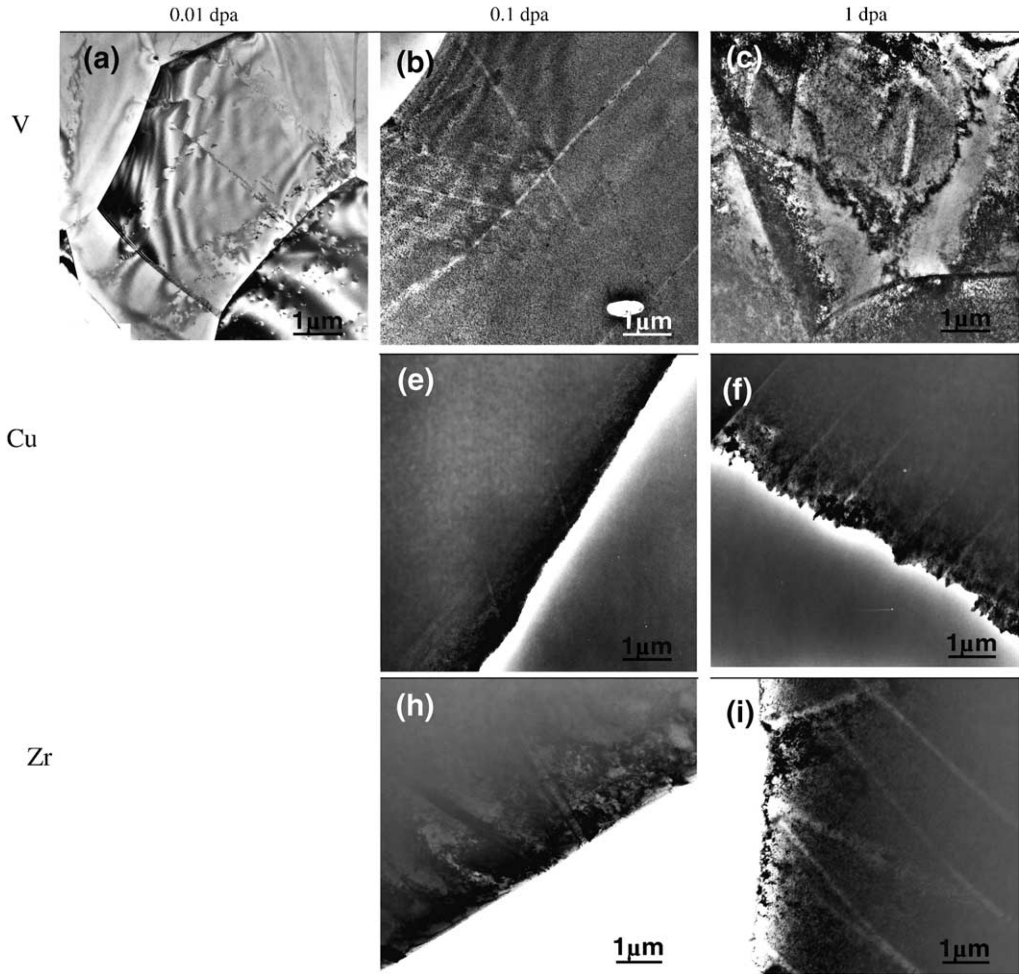

Fig. 2. Microstructure of deformed pure metals irradiated up to 1 dpa. Dislocation channeling was observed in each metal.

estimated using the value of the UTS. In V (BCC), there is a tendency for the resolved shear stress to be greater when the tensile axis is close to [0 1 1]. Furthermore, there is a tendency for the channel width and resolved shear stress to be greater when the angle is around 45°. In addition, channel width increased with increasing resolved shear stress, indicating that the rapid local deformation tends to occur at a high resolved shear stress level. The detail of channeling in Cu and Zr could not be summarized due to the limited number of channels that were analyzed to date. Further details regarding the effect of resolved shear stress on dislocation channel properties will be summarized elsewhere.

A characteristic feature that accompanies the pronounced hardening in metals irradiated at low temperatures is the loss of work hardening capacity. Early studies of radiation hardening produced competing models for the hardening mechanism, based on either dislocation source locking or lattice friction. Several researchers have proposed that both mechanisms operate, with source hardening responsible for the upper yield point and lattice hardening responsible for the lower yield stress observed in metals irradiated at low temperatures [19,20]. The loss of work hardening capacity produces sharp decreases in uniform elongation. Both effects have been shown to be generally due to dislocation channeling [2,18]. Twinning has also been observed to cause a pronounced loss in strain hardening capacity in austenitic stainless steel irradiated at 290 °C and tested near room temperature [21], however, no twinning was observed in the present experiment. From these results, it is suggested that the loss of work hardening capacity in V, Cu and Zr irradiated at low temperature is mainly due to dislocation channels that are formed under a high local resolved shear stress, leading to the observed localized deformation.

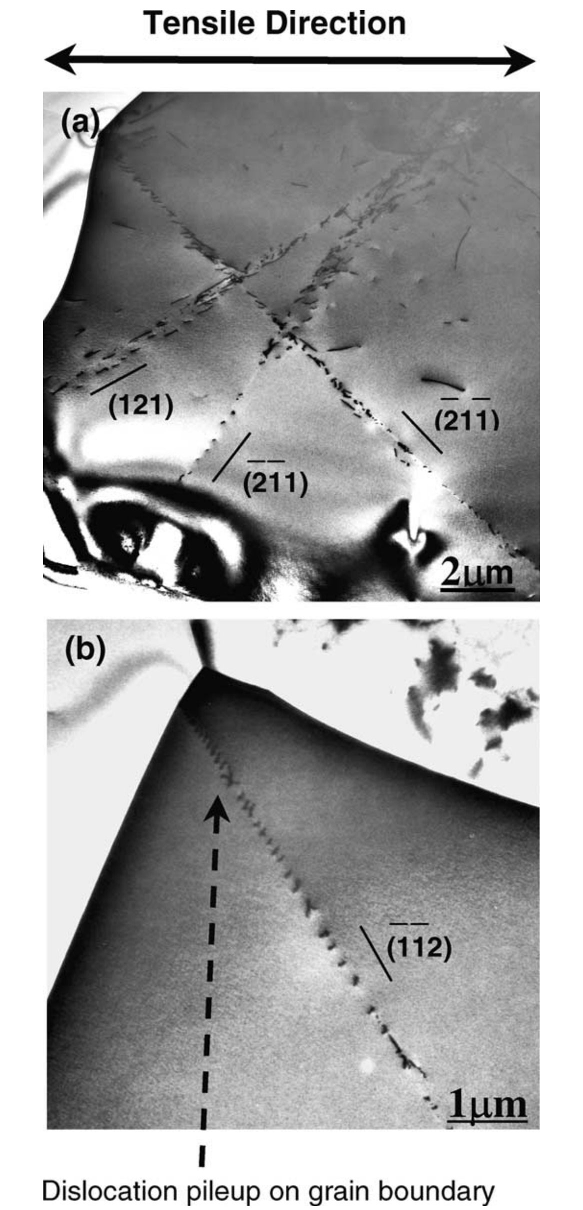

Fig. 3. Microstructure of dislocations in channels in V irradiated to 0.012 dpa after tensile test.

4. Conclusions

The deformation microstructure of irradiated pure vanadium, copper, and zirconium has been investigated by transmission electron microscopy in order to understand the deformation mechanisms of materials showing a loss of strain hardening capacity.

Irradiation-induced defect clusters, consisting predominantly of dislocation loops in V and Zr, and SFT in Cu, were observed in all irradiation conditions. The estimated barrier strength of each defect cluster was in good agreement with previously reported value. Dislocation channeling occurred in all deformed metals, and is coincident with prompt plastic instability at yield. In addition, dislocation pileup was observed at grain boundary in the deformed V irradiated to 0.012 dpa, indicating that channel formation could lead to dislocation pileup and the resulting stress localization could be a source of grain boundary cracking. It is suggested that the loss of work hardening capacity in irradiated V, Cu, and Zr at higher doses is mainly due to dislocation channeling in local regions that experience a high resolved shear stress.

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