

# Plastic instability behavior of bcc and hcp metals after low temperature neutron irradiation

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## Abstract

Plastic instability in uniaxial tensile deformation has been investigated for the body centered cubic (bcc) and hexagonal close packed (hcp) pure metals, V, Nb, Mo, and Zr, after low temperature (60–100 °C) neutron irradiation up to 0.7 dpa. Relatively ductile metals, V, Nb, and Zr, experienced uniform deformation prior to necking at low doses and prompt plastic instability at yield at high doses. Mo failed in a brittle mode within the elastic limit at doses above 0.0001 dpa. V showed a quasi-brittle failure at the highest dose of 0.69 dpa. In the ductile metals, plastic instability at yield occurred when the yield stress exceeded the plastic instability stress (PIS), which was nearly independent of dose. The PIS values for V, Nb, Mo, and Zr were about 390, 370, 510, and 170 MPa, respectively. The coincidence of plastic instability at yield and dislocation channeling cannot be generalized for all metallic materials.

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## 1. Introduction

Metallic materials often experience a complete loss of uniform ductility, or prompt necking at yield, after low temperature irradiation [1–8]. The low temperature irradiation usually results in a strong radiation-induced hardening as well as in a reduction of strain-hardening rate, and both of these radiation effects can cause the loss of uniform ductility [3–9]. The ultimate tensile strength (UTS), or the maximum load divided by the initial cross-sectional area, has been commonly used to describe the uniform deformation limit of the material. Sometimes, however, the radiation effect on plastic instability behavior cannot be correctly described by the UTS because the engineering stress term includes an influence from the contraction in load-carrying area due to plastic deformation. An alternate parameter that has been proposed to provide a better description is the plastic instability stress (PIS), or the true stress at UTS [10,11]. The main goal of the present paper is to analyze

the effects of radiation on the plastic instability behavior, and to suggest the use of PIS for correct description and prediction of plastic instability.

Analysis of tensile test data was performed for four pure metals: V, Nb, Mo, and Zr. The selection of these metals for analysis was because they are the primary components of alloys used for fission and fusion reactor applications at elevated temperatures [12–15]. Discussion is focused on the flow curve behavior after irradiation and on the dose dependencies of the PIS and YS. It is also suggested that the PIS can be an intrinsic property of the material. The relationship between unstable plastic deformation after irradiation and deformation mechanisms is also discussed.

## 2. Experimental

Four bcc and hcp metals: V, Nb, Mo, and Zr were purchased from Alfa Aesar Cooperation and certified impurity analysis results were supplied from the company, as listed in Table 1. Annealing conditions for test metals, which were selected to produce about 10 grains or more over the specimen thickness of 0.25 mm, are also given in Table 1. A custom-designed miniature sheet

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Table 1  
Chemical compositions and heat treatment conditions

Metals	Chemical composition in wt% (purity)	Annealing condition (in vacuum)	Dose range (dpa)
V	V–0.026Si–0.039Mo–0.027O (99.8%)	900 °C, 0.5 h	0–0.69
Nb	Nb–0.3Si–0.01Ni–0.06Ni–0.05Ta (99.5%)	900 °C, 1 h	0–0.37
Mo	Mo–0.036W–0.005O–0.004N–0.002C (99.95%)	1100 °C, 1 h	0–0.07
Zr	Zr–0.0058Hf–0.0056Fe (99.94%)	670 °C, 0.5 h	0–0.63

tensile specimen [7] with gage section dimensions of 1.5 mm wide, 0.25 mm thick, and 8 mm long was used. All tensile tests were conducted at room temperature at a nominal crosshead speed of  $0.008 \text{ mm s}^{-1}$ , which gave a strain rate of  $10^{-3} \text{ s}^{-1}$ . The true plastic uniform strain  $\epsilon_U^p$  was calculated from the uniform elongation, UE (%) using the definition of logarithmic strain  $\epsilon_U^p = \ln(1 + \text{UE}/100)$ . Then UTS was converted to the true stress unit or PIS using the expression  $\text{PIS} = \text{UTS} \times \exp(\epsilon_U^p)$ .

Irradiation experiments were performed in the Hydraulic Tube facility of the high flux isotope reactor (HFIR) at the Oak Ridge National Laboratory [7]. Fast neutron fluxes ( $E > 0.1 \text{ MeV}$ ) at the irradiation capsules (rabbits) were of order of  $8 \times 10^{18} \text{ n m}^{-2} \text{ s}^{-1}$  and the ratio of fast neutron fluence to displacements per atom (dpa) is approximately  $12 \times 10^{24} \text{ n m}^{-2} \text{ dpa}^{-1}$ . The specimens were irradiated to five different fluences. The dose range for each specimen series is given in Table 1 in the dpa unit. Irradiation temperature was estimated to be in the range 60–100 °C.

TEM pieces cut from the gage section of the deformed tensile specimens were in the form of small square plates,  $1.5 \times 1.5$  (or  $2.0$ )  $\times 0.25 \text{ mm}^3$ . These TEM pieces were ground to a thickness of about 0.1 mm, and were electro-thinned in a Tenupol electropolishing apparatus with a specially modified specimen holder [7]. Deformation microstructures were observed in a JEM-2000FX transmission electron microscope operating at 200 kV.

### 3. Result and discussion

#### 3.1. Engineering stress–strain curve and plastic instability

The engineering stress–strain curves for the test materials are presented in Figs. 1 and 2(a)–(c). The V showed large radiation-induced strengthening; its YS was increased by more than 70% after irradiation to 0.12 dpa. At the highest dose of 0.69 dpa, the specimen experienced quasi-brittle failure in the elastic limit. Lüders regions after yield are present in the curves for 0 and 0.0001 dpa. Yield drops of 20–40 MPa are found on the curves at low doses 0.0001 and 0.0012 dpa. At 0.0012 dpa the engineering strain hardening is negative after the

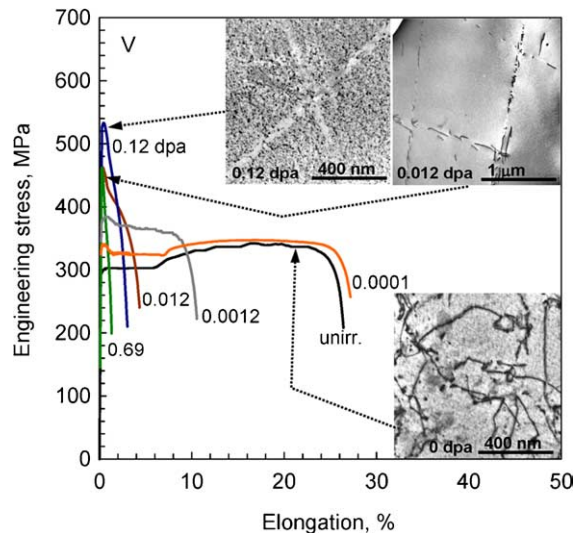


Fig. 1. Irradiation effects on the engineering stress–strain curve for V. Representative deformation microstructures are also presented.

yield drop. At 0.012 dpa an unstable plastic deformation is apparent after a yield drop of 30 MPa. At higher dose of 0.12 dpa, it is not possible to discern the yield drop from the subsequent load decrease due to plastic instability. The plastic instability at yield occurred when the yield stress was higher than the PIS of the material, about 390 MPa. In Fig. 1, the deformation microstructures at 0, 0.012, and 0.12 dpa are also displayed. These will be discussed in detail in Section 3.3.

The dose dependence in the stress–strain curves for pure Nb is similar to that for pure V, as presented in Fig. 2(a). The yield drops, which appeared in the range 0–0.0007 dpa, were less than 20 MPa. The curve at 0.007 dpa is similar to that of V at 0.001 dpa, which consists of a portion of slowly-decreasing engineering stress up to an engineering strain of 9% and an instability failure with accelerated load drop. Again, it is observed that plastic instability at yield occurs when the yield stress is higher than the PIS of the unirradiated specimen, about 360 MPa. The average PIS over the dose range 0–0.01 dpa is about 370 MPa.

Among the test materials, Mo displayed the most dramatic change of tensile properties due to irradiation,

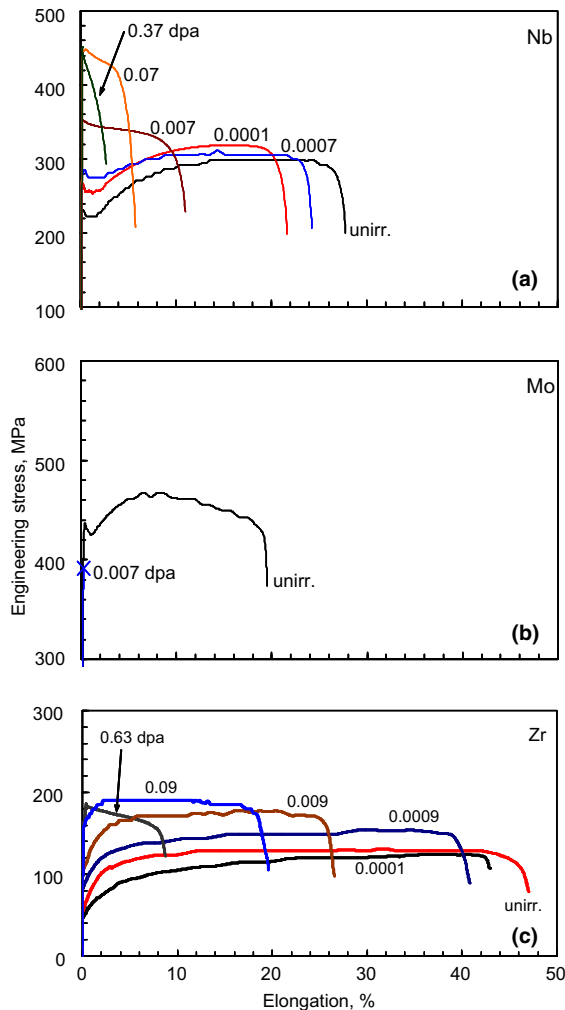


Fig. 2. Irradiation effects on the engineering stress–strain curves for: (a) Nb, (b) Mo, and (c) Zr.

as presented in Fig. 2(b). Of the two specimens irradiated to 0.0001 dpa one sample broke after small uniform deformation without necking, while the other sample failed in a brittle mode within the elastic limit. At higher doses all the specimens failed within the elastic limit, and the fracture stress decreased with dose. Scanning electron microscopy confirmed that those failures occurred by intergranular cracking due to a grain boundary embrittlement by radiation. This irradiation embrittlement is likely due to the powder metallurgy method used for the production of Mo. No specimen after irradiation showed a failure stress higher than the PIS of unirradiated Mo, 510 MPa.

Radiation effects in the pure Zr were milder, Fig. 2(c). The shape of engineering flow curve changes rather progressively as dose increases and plastic instability at yield was not observed in the dose range 0–0.09 dpa. The

uniform deformation regime disappeared only at the highest dose of 0.63 dpa, where the yield stress of 180 MPa was higher than the instability stress of unirradiated material, about 160 MPa.

### 3.2. Dose dependence of plastic instability stress

In Fig. 3(a)–(d) the dose dependencies of the stress parameters, PIS, YS, and UTS, are presented for V, Nb, Mo, and Zr, respectively. Fig. 3(a) shows that the PIS of V is nearly constant at 390 MPa, while the YS and UTS increase with dose until the specimen fails in a quasi-brittle mode at the highest dose of 0.69 dpa. At 0.0017 dpa the three parameters, PIS, YS, and UTS, become identical. At higher doses necking initiates at yield. The true stress can continue to increase and can exceed PIS in elastic regime because any plastic instability condition can not be applied to the elastic regime [10]. The necking when  $PIS < YS$  may progress at an accelerated rate. A critical dose at which prompt necking initiates at yield is called the dose to plastic instability at yield,  $D_C$  [10], which is 0.0017 dpa for V. In Fig. 3(b) Nb also shows a dose-independent PIS, which is nearly invariant at about 370 MPa up to a dose of 0.007 dpa ( $D_C$ ). Mo, a relatively brittle material, has a PIS of about 510 MPa. However, the fracture strength FS, which is identical with UTS at 0.0001 dpa or higher, never reaches the PIS and decreases with dose. The PIS for Zr seems to be approximately dose independent. It is noticeable, however, that the PIS data for Zr at 0.01 dpa (two overlapping points) are above the constant PIS line. This may be because the Zr specimens for these data were accidentally subjected to a slight bending during handling. A small bending can erase the yield drop phenomenon and can raise the early strain-hardening rate in subsequent tensile testing; consequently, the uniform strain and PIS would be increased.

Thus far, there has been no criterion proposed to predict prompt necking at yield after irradiation. Recently, the authors found that the PIS is almost independent of irradiation dose in a number of metals [10,11]. A criterion for plastic instability at yield, if it is proposed on the basis of the dose independence of PIS, is that an irradiated material will experience necking at yield when the YS increases above the PIS of unirradiated material. This criterion fits the current bcc and hcp pure metals in Figs. 1 and 2(a)–(c). It is proposed that the PIS be used as a stress criterion for material failure in design and assessment activities for nuclear systems. Once the PIS of the unirradiated material is known, the dose to prompt necking at yield may be predicted from YS values derived from nondestructive hardness measurements.

### 3.3. Channel deformation and plastic instability in V

Fig. 1 includes TEM photographs of the deformation microstructures of V at doses of 0, 0.012, and 0.12 dpa,

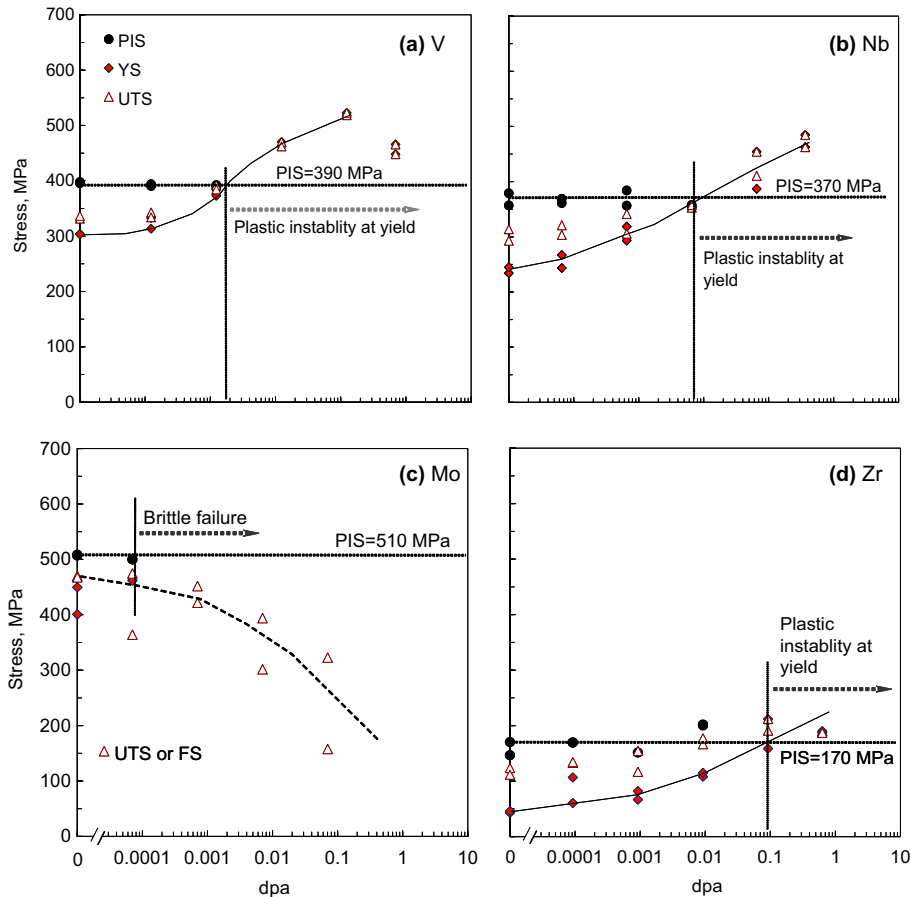


Fig. 3. Dose dependencies of YS, PIS, and UTS for (a) V, (b) Nb, (c) Mo, and (d) Zr. The positions for  $D_C$  and average values for PIS are also indicated.

and the corresponding tensile curves [16] are indicated. For the unirradiated V, TEM was done at a bulk strain of about 5% and shows dislocation tangles. These dislocations must have moved and interacted on multiple planes, typical of the normal work hardening region [11]. The other two micrographs are from the irradiated specimens and they present early deformation at bulk plastic strains of only 0.1% and 0.3%. These latter microstructures show very localized plastic deformation in narrow bands and cleared dislocation channels on few slip systems [4–6,17–19]. Within these narrow bands or channels multiple dislocations can move on each slip plane and can cause large local shear strains, measured in Cu up to 550% [18] and Nb at 220–370% [4,19]. The respective bulk tensile strains are only 4% and 6.6%.

Although the macroscopic plastic strains for the irradiated V specimens in Fig. 1 are very low, 0.1% and 0.3%, the local average strain in a channeled region should be much higher, assuming that the strains in individual channels obey the trend described above. The dimensions of channels are dependent on dose and stress

level [16–18]. In detailed measurements on channel dimensions in V [16] the width of a channel is typically of order 0.1  $\mu\text{m}$ , and the spacing between channels is of order 2  $\mu\text{m}$ . If the shear strain within a channel is assumed to be a modest 100%, the local shear strain averaged over the channeled area should be about 5% ( $=100\% \times 0.1 \mu\text{m} \div 2 \mu\text{m}$ ). This difference between the local and the macroscopic strains may arise because channel deformation in polycrystalline materials, especially at early stage of deformation, occurs in a heterogeneous manner. Since this heterogeneous deformation induces strong interaction stresses among adjacent grains, or long range internal stresses, macroscopic strain-hardening behavior may be affected by the hardening effect from the interaction stress between grains as well as by the softening effect from the defect clearance within channels.

Although the channel deformation frequently coincides with a reduced strain-hardening rate or a prompt necking at yield [1–9], it has not been explicitly demonstrated that channel deformation directly causes

prompt necking at yield. Fig. 1 indicates that the plastic instability at yield coincides with the channel deformation; however, there are plenty of examples that show channel deformation during uniform deformation. In particular, the face-centered cubic (fcc) metals like 316 stainless steels [7], Cu [20], and Au [20] show channel deformation after irradiation in the uniform strain region. Hence channel deformation can be connected to the reduced work hardening only, with or without necking at yield. The existence of a critical stress for channeling and the role of channeling in the plastic instability at yield are unresolved questions.

#### 4. Conclusions

The dose dependence of tensile deformation and plastic instability behavior have been investigated for four bcc and hcp pure metals, V, Nb, Mo, and Zr, after low temperature (60–100 °C) neutron irradiation up to 0.7 dpa. Conclusions drawn from the study are given as follows:

- (1) The ductile metals V, Nb, and Zr showed strong irradiation hardening. The less ductile Mo displayed embrittlement from the lowest dose of 0.0001 dpa.
- (2) Prompt necking at yield occurs when the yield stress is above the plastic instability stress (PIS), which is approximately independent of dose. The PIS values for V, Nb, Mo, and Zr were about 390, 370, 510, and 170 MPa, respectively.
- (3) Although plastic instability at yield and dislocation channeling are simultaneously observed in V, the coincidence of these two phenomena cannot be generalized for all metallic materials.

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