IRRADIATION SOFTENING IN PURE IRON SINGLE CRYSTALS

by

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Introduction

Electron irradiation of pure iron specimens at a temperature kept below the major recovery steps of stage I produces primarily single interstitials and single vacancies distributed nearly randomly (except for the close proximity of paired interstitials and vacancies). Therefore, upon plastic deformation following such irradiation, one can study specifically the effect of the interaction between a dislocation and randomly distributed point defects. It has been shown that the effect in fcc metals^(1,2) is a significant hardening and that the hardening results primarily from the presence of interstitials. The magnitude of the hardening can be represented by the equation

$$\Delta\tau = \alpha\mu C^{\frac{1}{2}}, \quad (1)$$

where $\Delta\tau$ is the increase in resolved shear stress due to the irradiation, μ is the shear modulus, and C is the concentration of the point defects. α is called the strengthening efficiency coefficient and varies from one material to another. The magnitude of α is related to the strength of the point defect [size and strain⁽³⁾] and the structure of the dislocation [width⁽³⁾ and effective core size⁽⁴⁾] and is usually large for the electron irradiation, indicating a strong interaction between a dislocation and an interstitial.

There is no reason to believe that this situation (strong dislocation-interstitial interaction and relatively weak dislocation-vacancy interaction) should change drastically in bcc metals. Nevertheless, in iron, the effect of low temperature electron irradiation was found to be opposite to the effects observed in fcc metals.^(5,6) That is, the effect is softening instead of hardening. A theory has been proposed to explain the softening

effect as due to the dispersed point defects with a strong misfit strain field.⁽⁷⁾ The theory suggests that the opposite effect is caused by the difference in the mode of dislocation motion between the two structures. The theory also predicts that the hardening as well as the softening can be caused by point defects with a strong misfit strain depending on the relative strength of the misfit and lattice resistance and the temperature. At the same time, the theory supports the intrinsic mechanism for the low temperature strength of bcc metals and solid solution softening.

On the other hand, support for the extrinsic mechanisms⁽⁸⁻¹⁰⁾ has been reported frequently in recent years; therefore one should not ignore this possibility. Further work must be carefully designed to understand the two sets of possible answers.

Since the irradiation softening phenomenon in iron appears to be a key to understanding the mechanism of dislocation motion in bcc metals, we have investigated the characteristics of this phenomenon further and report the findings here. The important finding is the dependence of the irradiation softening on the crystallographic orientation of the tensile axis. For the largest softening orientation, the temperature and strain rate dependence of the flow stress were also examined. Details of the specimens and of the irradiation have been reported previously.^(11,12)

Orientation Dependence

The dependence of the yield stress of bcc metals on the crystallographic orientation of the tensile axis is well known.⁽¹³⁾ However, the correlation of this orientation dependence with the effects of solutes, or point defects, has not been investigated except in a few cases.⁽¹¹⁾ In the present investigation two directional parameters, λ and χ , are used to describe the

orientation of the tensile axis and they were varied in order to examine the irradiation effect. λ represents the angle between the tensile axis and the primary Burgers vector, $a/2[11\bar{1}]$, and was 45° when χ was varied to examine the effect. χ represents the angle between the $[101]$ direction and the normal to the maximum resolved shear stress plane containing the primary Burgers vector. Therefore the maximum resolved shear stress plane coincides with the $(2\bar{1}1)$, (101) and (112) planes for $\chi = -30^\circ$, 0° and 30° respectively. These three special cases are called the soft $\{112\}$ slip (the slip direction is the twinning direction), the $\{110\}$ slip and the hard $\{112\}$ slip (the slip direction is the anti-twinning direction). Transmission electron microscopy revealed that screw dislocations of at least two Burgers vectors (sometimes four for the specimens oriented near the soft $\{112\}$ slip) coexisted with substantial densities from the onset of the macroscopic deformation in the iron specimens. The variation of the irradiation softening with χ was quite spectacular. At $\chi \cong -30^\circ$ the irradiation effect was hardly apparent in the stress-strain relation; however, at $\chi \cong 30^\circ$ a large softening effect was observed. With the maximum dosage used in this investigation ($1.5 \times 10^{18} \text{ e/cm}^2$) the yield stress (expressed by the tensile stress) was reduced by 60%. The resulting yield stress was $\sim 25 \text{ kg/mm}^2$ at 60° K , the lowest yield stress ever reported in iron at this temperature range. The irradiation softening effect for $\chi = 0$ was mild, a reduction of yield stress by 10% with similar irradiation. Consequently, the post-irradiation yield stress decreases spontaneously with χ from -30° to 30° .

The effect of λ was examined at $\chi = 30^\circ$ where the maximum softening took place. The softening effect remained large as λ was increased from

45° to 55°. On the other hand, when λ was reduced slightly from 45°, the magnitude of the softening was less than 20% at $\lambda \cong 39^\circ$.

Temperature Dependence of Yield Stress

The temperature dependence of the yield stress, $\partial\sigma/\partial T$, was increased by the irradiation in spite of the fact that the magnitude of the yield stress decreased. Therefore the fraction of softening was large between 40° and 80° K. A smaller softening effect was observed at 4.2° K.*

Strain Rate Dependence of Yield Stress

Since electron irradiation improved the ductility of the iron, the strain rate cycling could be applied repeatedly to the irradiated specimens and the strain rate dependence could be reliably determined over the temperature range from 4.2° to 80° K. The irradiation increased the strain rate dependence significantly. The strain rate dependence decreased with plastic deformation. The strain rate dependence also increased with the temperature and reached the maximum between 40° and 60° K. It appeared to decrease with a further increase in temperature.

Recovery of Irradiation Softening

When the irradiated iron specimens were annealed at a temperature above 80° K the softening effect was reduced. This recovery peak of the irradiation softening centered at 100° K and the recovery effect appeared to saturate near 150° K.

* The electron irradiation increased the ductility of the iron specimens; therefore no prior deformation was necessary to deform them at 4.2° K. However, without the irradiation the specimens, particularly of the hard {112} slip orientation, must be prestrained at 60° K prior to the 4.2° K deformation.

Discussion

The recovery of electron irradiated iron indicates that no major recovery steps exist below 70° K.⁽¹⁴⁾ Since the 2 MeV electron irradiation produces almost exclusively Frenkel pairs which are expected to distribute nearly randomly throughout the lattice, it is therefore difficult to consider any mechanism other than one based on these Frenkel defects. The magnitudes of the dislocation-interstitial interaction and the dislocation-vacancy interaction were estimated earlier in fcc metals⁽¹⁵⁾ indicating that the former is considerably larger than the latter. If one assumes the interaction to be primarily elastic in nature, a similar situation should exist in bcc metals. Therefore the most logical explanation for the observed effect is that it is caused by the interstitials of nearly random distribution.

An electron microscopic investigation⁽¹⁶⁾ was carried out to examine the dislocation structure of irradiated and unirradiated iron single crystals after deformation at 60° K. There was very little difference in structure between the two types of specimens except that a few times higher dislocation density was found in the irradiated specimens. In both cases, screw dislocations were uniformly distributed throughout the specimens. This structural observation supports a softening mechanism due to an increase in dislocation mobility (or a decrease in the stress sustaining a certain dislocation velocity).

A theory has been proposed⁽⁷⁾ to explain an increase in dislocation mobility by point defects. The formation of double kink is promoted by a local misfit strain, since its stress field exerts a couple force on a screw dislocation. Consequently, the energy to form a double kink is

reduced at one side of the misfit strain center. The softening effect is prevalent in the low temperature region; in fact, the mechanism predicts a hardening effect near and above room temperature. Therefore it has been suggested in the paper that the mechanism of the electron irradiation softening is similar to that for solid solution softening and that solid solution softening and solid solution hardening can arise from the same mechanism depending on the experimental conditions such as temperature, solute (misfit strain center) concentration and relative strength of misfit strain to intrinsic resistance to screw dislocation motion. Unfortunately, it is not possible to examine experimentally the effect of self-interstitials on yield stress in the high temperature regime, since the majority of the interstitials anneal out during stage I recovery and those which survive this recovery stage would not be distributed randomly. The above-mentioned trend has been observed in many solid solutions which exhibit the solid solution softening phenomenon.

The preceding discussion presumes that the low temperature strength of iron (therefore perhaps other bcc metals) arises from the intrinsic lattice resistance to dislocation motion and that the solid solution softening can be explained by an intrinsic mechanism. However, there have been a number of opponents to this point of view. In fact, there are cases in which an extrinsic mechanism such as gettering of carbon and nitrogen atoms in iron by titanium (scavenging mechanism) has been reasonably well established.⁽⁸⁾ It has also been proposed in a series of recent investigations in niobium and tantalum base alloys that the solid solution softening effect occurs exclusively in ternary systems and that the softening effect is attributed to the effect of the solute-solute interaction on

dislocation motion.^(17,18) Therefore there should be a discussion on the possibility or impossibility of an extrinsic mechanism to explain the observed irradiation softening.

The purity of the specimen must be examined first as the impurities may still be the primary cause of the low temperature strength. The initial material, the Battelle-AISI iron, is reported to contain less than 5 wt. ppm of non-metallic impurities and about 30 wt. ppm of metallic impurities. The single crystal growth and the subsequent annealing were carried out in the ZrH_2 purified hydrogen atmosphere; therefore the interstitial content should have been substantially reduced. Earlier experiments using less pure specimens (insufficient purification by hydrogen or Ferrovac E iron as the starting material) indicated that the irradiation softening effect appeared to decrease with increasing impurity content.⁽⁵⁾ In conjunction with the fact that the substantial softening occurs in the range of self-interstitial concentration ~ 100 ppm and the fact that the interstitials do not migrate substantially below $80^\circ K$, it is highly unlikely that the electron irradiation softening is caused by the interaction (neutralization) of the impurities present in the specimens with the irradiation-induced defects.

Other experimental observations support this conclusion. It was reported that the electron irradiation increased the microyield stress (determined as 10^{-4} plastic strain), although the macroyield stress was reduced. If the irradiation is to reduce the resistance of the impurities to dislocation motion, it is difficult to explain the hardening effect on the microyielding. However, the intrinsic mechanism proposed in this report provides a logical explanation for this seemingly contradictory

observation. As the predominant presence of screw dislocations indicates, there is a mobility differential between edge dislocations and screw dislocations. The edge dislocations move at a stress considerably lower than that for the screw dislocations. Since no couple force is exerted on the edge dislocations by a misfit strain, the presence of any point defects only increases the resistance to the edge dislocations. Thus an increase in microyield stress occurs. A decrease in macroyield is to be expected as the latter is determined by the motion of screw dislocations. It is equally difficult to explain the orientation dependence of the electron irradiation softening effect by the solute-solute interaction mechanism or any other extrinsic mechanism. Although no details have been worked out, it is possible to explain the orientation effect by the intrinsic mechanism. The orientation dependence of the yield stress of pure bcc metals can be understood by the asymmetric core structure of screw dislocations or by the asymmetric distribution of the potential for the dislocation motion to different directions. The core configuration or the potential distribution may be extremely sensitive to the local atom configuration (or the local strain field). The presence of interstitial atoms would change the configuration significantly and may cause the observed orientation dependence of the softening effect.

The recovery of electron irradiation softening also tends to negate an extrinsic mechanism. The long range diffusion of interstitials should enhance the interaction and clustering with the impurity atoms. As the observation showed, the softening effect almost completely disappeared after the recovery stage where the interstitial migration is thought to take place.

The electron irradiation also changes the shape of the stress-strain curves. For the specimen orientation which exhibited the largest softening effect, the work-hardening is relatively small in the unirradiated state. On the other hand, in the irradiated specimens a rapid work-hardening follows the yielding. This may be interpreted to mean that a different dislocation-dislocation interaction is in operation; therefore the mode of plastic deformation is different between the irradiated and the unirradiated specimens. However, upon annealing of the specimens irradiated and deformed, the flow stress does not exceed the flow stress of the unirradiated specimens deformed similarly. Therefore the apparently high work-hardening on the irradiated specimen must be due to the annihilation of the interstitials promoting the dislocation motion. This conclusion is in agreement with the electron microscopic observation discussed earlier.

The irradiation softening is not a phenomenon limited to iron single crystals irradiated with electrons. The softening was reported also in the electron-irradiated polycrystalline iron,⁽⁶⁾ in the neutron-irradiated iron single crystals⁽¹⁹⁾ and in the neutron-irradiated polycrystalline iron.⁽⁶⁾ The common observation among these experiments is that the softening effect was observed when the specimens were irradiated and tested at a temperature below the major recovery in stage I (stage I_c and I_d at $\sim 80^\circ$ and $\sim 100^\circ$ K respectively).⁽¹⁴⁾ This suggests the dispersed interstitials as the common cause for the softening.

In conclusion, the observed irradiation softening effect can be explained by the intrinsic mechanism, namely, the interaction of screw dislocations with randomly dispersed interstitials. At least some of the solid solution softening phenomena observed in alloys can be explained by the same mechanism.

However, the alloying may be accompanied by an additional effect such as solute segregation to dislocations which may also strongly affect the yield stress. This effect may mask the softening effect partially or totally. Changes in the dislocation structure of deformed specimens caused by alloying, which are often reported in electron microscopic investigations, support this contention. The alloying, therefore, may not be as good as the low temperature irradiation in studying the effect of random solutes on dislocation motion and yield stress.

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