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EFFECT OF ELECTRON IRRADIATION ON THE STRENGTH OF IRON SINGLE CRYSTALS*

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Introduction

The effect of electron irradiation on the strength of fcc metals and alloys has been extensively investigated (1-4) and the results can be satisfactorily correlated to calculations (5-7) based on the dislocation-interstitial interaction. This paper reports the effect of electron irradiation on the strength of a bcc metal, iron. This investigation also intends to establish the role of interstitials in determining the low temperature strength of iron and other bcc metals. The controversy over the mechanism(s) controlling the low temperature strength of these metals has never been resolved (8-12); the mechanisms proposed are often divided into two classes: those related to the presence of interstitials (impurity hardening), and others basically caused by the nature of the bcc crystal structure (inherent lattice hardening).

The use of electron irradiation to study the nature and effect of interstitials has unique advantages: (1) Interstitials can be alloyed into a specimen at a cryogenic temperature without altering the structure and configuration of other defects such as dislocations. (2) These interstitials are distributed in nearly random fashion. (3) The control and determination of the relative concentration of interstitials can be done readily and accurately in the concentration range 0.001 ~ 100 ppm. No other method is so expedient. Since the interstitials introduced by the irradiation are self interstitials one might argue that they are entirely different from impurity interstitials. However, many experimental and theoretical studies have demonstrated that the interaction between interstitials and dislocations is basically elastic and that the agreement between experimental results and elasticity calculations is good. Therefore this report treats the nature of the dislocation interaction with self interstitials as similar to that with impurity interstitials.

Experimental Procedures and Results

Three types of iron single crystals were used in the present study: (a) Single crystals grown from Ferrovac E iron by a strain anneal method, (b) single crystals cut directly from a coarse-grained Battelle zone-refined iron rod, and (c) single crystals grown from the Battelle iron by a strain anneal method and subsequently purified in a ZrH_2 system. The Battelle iron was reported to contain 6 ppm of non-metallic impurities and 48 ppm of metallic impurities. The ZrH_2 treatment was expected to reduce the content of C and N. A reduction to as low as 0.005 ppm C had been previously reported (13). The strain-annealed and ZrH_2 treated Battelle iron single crystals demonstrated significant plasticity down to 4.2°K with the small work-hardening coefficients ($d\tau/d\gamma \approx 25 \text{ kg/mm}^2$). An example of a τ - γ diagram is shown in Fig. 1. Thus the macroscopic flow stress can be readily determined and the effect of electron irradiation on the flow stress can be unambiguously studied.

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FIG. 1

Shear stress-shear strain (τ - γ) diagram of an unirradiated Battelle iron single crystal after Zn treatment, specimen (c).

FIG. 2

Effect of electron irradiation (dose unit e/cm^2) on a specimen (c) showing "irradiation softening". Tensile test at 60°K , irradiation temperature below 60°K . Note the hardening after aging treatment at 30°C .

A Van de Graaff accelerator was used to generate the incident electrons of 1.25 MeV. An electron dose of up to $10^{18}\text{e}/\text{cm}^2$ was used in the present study and an interstitial concentration of ~ 50 ppm was expected to be generated by the irradiation (14). The specimens were irradiated and tensile tested in a modified Andonian liquid helium cryostat capable of yielding any temperature between 4.2° and 300°K . However, the irradiation temperature was normally between 20° and 80°K because no significant recovery of the irradiation-induced resistivity was reported below 80°K (15,16) and because the ease and economy of the experiments were to be optimized. The tensile tests were carried out in an Instron type machine and the uncertainty involved in the determination of the flow stress was about $0.25\text{ kg}/\text{mm}^2$ at $\sim 30\text{ kg}/\text{mm}^2$.

The effect of electron irradiation on a (c) specimen is illustrated in Fig. 2. The specimen was deformed about 5% at 60°K to establish the flow stress and was then irradiated. The same procedure was repeated for the second irradiation. The third interruption of the tensile test was to examine the annealing effect. The important observation here is the decrease in flow stress following each electron irradiation (electron irradiation softening). A number of similar experiments confirmed the reproducibility of this observation.

Other experiments were also carried out with varying parameters such as irradiation, aging and testing temperatures and dosage, using all three types of crystals. The observations in these experiments are summarized as follows: (i) The softening effect was always found in (c) crystals after electron irradiation below 60°K . The softening effect appeared to increase with the electron dose up to $5 \times 10^{17}\text{e}/\text{cm}^2$. (ii) In (a) and (b) crystals [which always showed a higher initial flow stress than that of (c)], the softening effect could not be detected. (iii) The amount of prior deformation did not appear to alter the magnitude of the softening effect significantly. (iv) No discernible change in the temperature dependence of flow stress was found in the irradiation softened crystals. (v) When an irradiated specimen was warmed up to ambient temperature, a hardening effect was always observed (cf. Fig. 2). This hardening effect was clearly induced by the irradiation,

as shown in Fig. 3, where the increase in flow stress determined at 24.5°C was plotted against the aging time. In this series of experiments, the (b) specimens were irradiated with $5 \times 10^{17}\text{e}/\text{cm}^2$ around 140°K and were aged at 24.5° , 50° , and 100°C for various time intervals. The increase in flow stress, $\Delta\tau$, was defined by the difference between the flow stress prior to the irradiation aging treatment and the lower yield stress after the treatment. The yield drop also appeared after this treatment, but it was not affected by the irradiation.

Dislocation Motion in a Combined Peierls and Misfit Strain Field

In order to examine possible effects of interstitials on the dislocation motion overcoming the Peierls potential, a calculation was carried out. The results of this calculation successfully explained the present experimental results. It also sheds light on the controversy over the mechanism for the low temperature strength of bcc metals and alloy softening.

The formulation of the present calculation explicitly includes both mechanisms which correspond to "inherent lattice hardening" and "impurity hardening" (10). The inherent lattice hardening was represented by a periodic potential function,

$$\Gamma(y) = (\Gamma_c + \Gamma_o)/2 + [(\Gamma_c - \Gamma_o)/2] \cos(2\pi y/b).$$

(1)

The impurity was represented by spherical misfit strain centers at (x_i, y_i, z_i) and the force acting on a dislocation segment at (x, y) was given by

$$F(x, y) = \sum_i 6\mu e b r_o^3 z_i (x - x_i) / [z_i^2 + (x - x_i)^2 + (y - y_i)^2]^{5/2} \quad (2)$$

A screw dislocation lying initially parallel to the x axis at $y = y_o$ was examined with respect to the motion along the y axis. The energy of a given dislocation configuration was evaluated by

$$U = \int_{l_1}^{l_2} u(x, y, y') dx, \quad (3a)$$

where

$$u(x, y, y') = \Gamma(y)(1 + y'^2)^{1/2} - \Gamma(y_o) + \int_{y_o}^y F(x, y) dy - \tau b(y - y_o). \quad (3b)$$

Only the configurations which satisfy the Euler's condition,

$$\delta \left[\int_{l_1}^{l_2} u(x, y, y') dx \right] = 0, \quad (4)$$

were considered here (9). The energies of two consecutive dislocation configurations (position) satisfying Eq. (4) correspond to the minimum and the maximum energies, U_o and U_s . Thus, the difference, $E = (U_s - U_o)$ is the energy required for the screw dislocation to move from this energy minimum position to the next energy minimum position.

It was found that two types of important energies, E , were involved in dislocation movement: E_n which corresponds to the double kink formation in the vicinity of the misfit defect (referred to as "nucleation" in this report) and E_s which corresponds to overcoming the misfit defect (referred to as "sideways motion"). As the stress field of the misfit aided the nucleation, the smallest E_n was found in its vicinity. This E_n also decreased with increasing the strength of the misfit strain center. On the other hand,

FIG. 3

Effect of electron irradiation on age hardening in specimen type (b) tested at 24.5°C. Solid lines show the results after irradiation to $5 \times 10^{17} \text{ e/cm}^2$ around 140°K and dotted lines without irradiation. Aging temperatures are indicated in the figure.

the energy for the sideways motion E_s increased with the strength of the center. Since both nucleation and sideways motion must occur for the dislocation to move continuously, the rate controlling process changes from nucleation to sideways motion as the misfit strength increases. The results of the calculations are summarized in Fig. 4, where the energy, E (E_n or E_s , whichever controls the motion) is plotted against the normalized applied stress (τ/τ_p) for various misfit strains. The curve corresponding to zero misfit strain represents the double kink formation without the misfit strain centers (9). A marked decrease in the energy should be noted as the misfit strain centers are introduced. As the strength of the misfit strain centers increases beyond a certain value (corresponds to the curve for $\epsilon=0.15$ in Fig. 4), however, the energy starts to increase. A considerably larger energy is required for dislocation motion in the case of $\epsilon=0.5$ than in the case without the misfit strain centers, particularly at a small applied stress.

An earlier work (6) emphasized the importance of the symmetry of a misfit defect such as a tetragonal misfit and a spherical misfit to which "rapid hardening" and "gradual hardening" were attributed, respectively. It was pointed out recently that this distinction is invalid if one considers the dissociation of a dislocation even as small as 1 to 2 atomic distances (7). In the present calculation a tetragonal misfit strain was also used in place of the spherical misfit strain. Although Eq. (2) had to be modified, similar results were obtained. For example, the E vs (τ/τ_p) plot for the y-y tetragonal misfit strain of 1.2 was virtually identical to that for a spherical misfit strain of 0.25 (Fig. 4). Therefore the argument presented in this section is not limited to the spherical defects and the above-mentioned distinction again does not have any significance.

Discussion

The structure of the electron irradiated iron in this experiment is characterized by the interstitial iron atoms displaced from their normal lattice sites and the vacant lattice sites left behind when the temperature of the iron is kept below 80°K during and after the irradiation (15,16). The recombination of some close interstitial-vacancy pairs may have occurred; but this does not alter the argument to be presented here except some overestimate of the interstitial concentration. The interaction of an interstitial atom with a dislocation is usually regarded as at least an order of magnitude greater than that of a vacancy (17). No other structural change is expected to occur due to the irradiation. Furthermore, the interstitial atoms should be distributed nearly randomly and neither interstitial clustering nor significant interstitial segregation to dislocations is expected to occur.

The foregoing irradiation softening effect, therefore, must be caused by those interstitials distributed randomly. As shown by the calculation, the double kink nucleation becomes easier near the interstitials, resulting in the softening. It should be pointed out that this argument applies only to the low interstitial concentration range.

FIG. 4

Energy, E , required for dislocation motion in a combined Peierls and misfit field is plotted against applied stress, τ , normalized with Peierls stress, τ_p . l and Γ_0 ($\approx \Gamma_c$) are dislocation segment length between misfit strain centers and dislocation self energy. Numbers on each line in the figure indicate spherical misfit strain at $r_0 = 2b$. z_i , the distance between the center and the slip plane (x-y plane) is taken to be $5b$.

According to the present calculation, the neighboring misfit strain restricts the double kink formation below a critical dislocation segment, l ; thus the energy E increases with decreasing l . Therefore a hardening effect would eventually be observed if the electron dose had been increased further. A recent calculation (18) utilized this effect to demonstrate that the hardening effect due to dispersed barriers (impurity hardening) can occur in inherent lattice hardening crystals.

— The present experimental and calculated results strongly support the contention that the inherent lattice hardening, namely, the intrinsic lattice resistance to dislocation motion determines the low temperature strength of pure iron and perhaps other pure bcc metals. The effect of the dilute impurities should be the softening effect and the impurity hardening effect can be expected only above a critical concentration.

The electron irradiation softening must be regarded as a simple case of "alloy softening". It has been demonstrated by the present calculation that alloy softening can be explained without invoking a scavenging effect, a reduction in inherent resistance such as Peierls potential or an increase in mobile dislocation density. The presence of misfit strain which normally impedes dislocation motion can promote dislocation overcoming the inherent lattice resistance. It should be noted that the strength of a misfit strain center is determined by the product of misfit strain and defect volume. In the cases of substitutional solid solution softening, clusters of solute atoms or localized regions of ordered solute atoms may have to be considered as the double kink nucleation sites.

— The hardening effect found after the electron irradiation and the aging around ambient temperature is not clearly understood, since the distribution of the irradiation-induced defects becomes uncertain after the aging. It is tempting, however, to propose that the hardening is basically due to the interstitial clustering. The calculation suggested that the resistance to dislocation motion increased with the strength of the misfit strain center due to the increased difficulty of sideways motion. The irradiation enhanced clustering of impurity interstitials, the simultaneous or combined clustering of impurity and self interstitials, or the clustering of self interstitials are some of the possible misfit strain centers.

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