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EFFECT OF ELECTRON IRRADIATION ON THE
STRENGTH OF IRON SINGLE CRYSTALS

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by

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"Effect of Electron Irradiation on the Strength of Iron Single Crystals"

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Summary of the Dissertation

The effect of electron irradiation on the plastic deformation of ultrapure iron has been investigated in the temperature range from 4.2°K to 373°K. Special care has been taken to minimize the uncertainty due to impurities, dislocations and sub-boundaries; namely, every single crystal-line specimen grown from the ultrapure iron supplied by the Battelle Memorial Institute was purified in the ZrH_2 system. These specimens exhibited appreciable ductility down to 4.2°K. In addition to these ultrapure specimens, less pure single crystal specimens grown from the Ferrovac-E iron were used to study the effect of the residual impurities.

The total electron dose ranged from 5×10^{16} to 2×10^{18} e/cm^2 with the incident electron energy of 1.3 MeV. Both the electron irradiation and tensile tests were carried out in a liquid helium cryostat. The tensile testing as well as the electron irradiation were always carried out at or below 60°K.

Important observations in this study are summarized as follows:

- (a) The electron irradiation caused a softening effect on the flow stress at 60°K up to the total electron dose of 2×10^{18} e/cm^2 .
- (b) This softening was not observed at 4.2°K.
- (c) In the microyield region, irradiation hardening was observed at 60°K.
- (d) This hardening effect was significantly enhanced at lower temperature, 4.2°K.

- (e) Post-irradiation aging at higher temperature (150°K) enhanced the hardening effect at 60°K .
- (f) Besides these low temperature experiments, the effect of irradiation and aging on the macroscopic deformation was also studied at temperatures from 60°K to 312°K . A temperature-insensitive hardening was observed in the temperature range $233^{\circ}\text{K} \sim 312^{\circ}\text{K}$. Upon testing at 60°K , the specimen showed twinning and ductile fracture.

In order to examine the possible effects of interstitials a calculation was carried out. The dislocation motion through a combined stress field of Peierls potential and misfit strain centers was examined. The effect of the presence of the strain centers appeared to reduce the double kink nucleation energy. By increasing the strength of the misfit strain centers, it was found further that there is a second kind of energy required for the motion of the kinks. These two types of energies were then carefully examined so that the total dislocation motion can be simulated. The results of this calculation explained the overall experimental observations found in the present experiment.

Every aforementioned item in the experimental results can be consistently explained on the ground of the present model of the dislocation motion. Aside from the detailed discussion it should be emphasized that the introduction of point obstacles into pure iron causes softening effects, and that this fact clarifies the role of the inherent lattice resistance in the plastic deformation of pure iron.

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I. INTRODUCTION

One of the characteristic features of the plastic deformation behavior of b.c.c. metals is the large temperature dependence of the flow stress. There have been numerous works attempting to clarify the mechanism by which the plastic deformation is controlled in these metals. In particular, the long standing controversy over the contribution of the interstitial impurities to the rate controlling mechanism has been discussed repeatedly and yet the problem seems to remain unsolved.

Strong evidence in support of the interstitial hardening mechanism was given by Stein et al.,⁽¹⁻⁴⁾ where the low temperature strength and the thermal activation parameters for the plastic deformation of high purity iron single crystals were compared to those for dilute carbon alloys. The interstitial hardening mechanism has also been supported in some other reports. For example, Lawley et al.⁽⁵⁾ found a change in stress vs. temperature curve in molybdenum after zone-refining. The effect of purification on the strength of molybdenum single crystals was also studied later by Stein⁽⁶⁾ and the data again supported the interstitial hardening mechanism. The same type of study has been extended to other b.c.c. metals such as niobium⁽⁷⁻¹⁰⁾ and tungsten.⁽¹¹⁾ Especially in the recent works on purified niobium single crystals, severe hardening has been observed by the addition of a small amount of interstitial impurities.^(8,9)

On the other hand, most of the investigations that have been carried out recently, have emphasized the importance of the inherent nature of the b.c.c. lattice in determining the nature of the plastic deformation at low temperatures. Experimental evidence supporting this inherent

mechanism was again the result of the study of the effect of interstitial impurity atoms on the low temperature strength of b.c.c. metals. Many experimental data have shown the temperature independent hardening or even reduction of the flow stress at low temperatures by the addition of solute interstitials.⁽¹²⁻¹⁴⁾ These results contradict the aforementioned results supporting the interstitial hardening mechanism. Similar studies have been done on substitutional alloys. In most of the alloys, the plots of flow stress vs. concentration have shown monotonic hardening at higher temperatures. At lower temperatures, however, the flow stress decreases to a minimum and increases monotonically for higher solute concentration.⁽¹⁵⁻²⁰⁾ This solution softening effect that appears at low temperatures has often been taken as evidence supporting the inherent lattice hardening mechanism. Some of the explanations put forward in favor of this mechanism are: the reduction of the lattice resistance by solute atoms, the change in mobile dislocation density, and the assistance of local double kink nucleation. In some cases, however, the softening effect has been explained by the scavenging of the residual interstitial impurities.^(8,9,21) In this explanation the interstitial hardening mechanism is inherently assumed. According to the recent work on the Fe-Ti system by Leemans,⁽²²⁾ the flow stress at higher temperatures was lowered by the addition of a small amount of titanium into iron, while at lower temperatures the stress was increased. However, the further addition of titanium caused a decrease of the flow stress at lower temperatures and an increase at higher temperatures. Then the effect of the initial addition of titanium was attributed to the scavenging and the effect of titanium beyond critical concentration was considered as a direct effect of solute atoms.

Another interesting observation which favors the inherent hardening mechanism is the presence of the stress asymmetry in b.c.c. metals. This was clearly demonstrated by the fact⁽²³⁾ that the yield stress measured for a $\langle 111 \rangle \{112\}$ slip system was significantly different for two opposite $\langle 111 \rangle$ directions. This asymmetry cannot be explained by the interstitial hardening mechanism. On the other hand by using the dissociation model^(24, 25) the asymmetry nature of the dislocation motion can be explained in the framework of the inherent hardening mechanism. As a result of extensive studies on b.c.c. single crystals, the dissociation model has gained support in recent years.⁽²⁶⁻³⁶⁾ The application of the dissociation model has, however, been criticized on the ground that the b.c.c. structure has high stacking fault energy and hence the splitting of the partials is expected to be small. A detailed structure of a dissociated screw dislocation in the core region was recently computed by atomistic calculations,^(37,38) and a more sophisticated explanation for the stress asymmetry has been given. As pointed out by Christian,⁽³⁹⁾ this calculation was made in the absence of an applied stress, and hence this model may not be applicable to the low temperature deformation without some modification to take into account the effect of applied stress on the asymmetry nature. Aside from the detailed discussions, the existence of stress asymmetry and the explanation given by the dissociation model strongly support the inherent hardening mechanism.

In conjunction with the effect of impurities on the softening of b.c.c. metals, Christ et al.⁽⁴⁰⁾ pointed out an increase in mobile dislocation density by the interstitial-induced cross-slip. Thus the dissociation model has been widely studied for explaining the characteristic features observed in b.c.c. metals.

The majority of the recent microstrain data on b.c.c. metals also supports the inherent mechanism. By the development of microstrain techniques,^(41,42) plastic deformation can be detected well below the macroscopic yield point. In the early work on iron by Kossowsky and Brown,⁽⁴³⁾ a high work-hardening rate was observed in the initial stage of deformation and this microplasticity was attributed to the motion of screw dislocations. This hypothesis was then supported by Stein⁽⁴⁴⁾ who used a slightly modified expression for the dislocation velocity originally proposed by Johnston.⁽⁴⁵⁾ A few years later, however, Carnahan et al.,⁽⁴⁶⁾ and Arsenault⁽⁴⁷⁾ proposed motion of edge dislocations as being responsible for the appearance of the microplasticity. Most of the recent microplasticity results⁽⁴⁸⁻⁵⁰⁾ have been analyzed on the basis of the edge dislocation mechanism, thus supporting the inherent mechanism. This model has been indirectly substantiated by the electron microscopy observations in Nb⁽⁵¹⁾ and Fe.⁽⁵²⁾ Direct evidence for the motion of edge dislocations in the microyield region was recently presented by Imura.⁽⁵³⁾ In this study the stress-strain behavior was recorded during the direct observation of the motion of individual dislocations in a high voltage electron microscope. Thus a number of recent studies have confirmed the importance of the inherent lattice nature of b.c.c. metals.

On the other hand, a strong argument supporting the interstitial hardening mechanism was recently given by Ravi and Gibala.⁽⁹⁾ The available data does not seem sufficient to refute their arguments, concerning the responsibility of interstitial impurities. Statham and Christian⁽⁵⁴⁾ have recently pointed out the importance of non-screw type dislocations, for the severe hardening effect due to substitutional or interstitial impurities. In this report they have suggested the possibility of

ordering in high solute concentration. In order to explain the drastic increase of the flow stress in Nb single crystals, the difference in the solubility limit was also quoted,⁽³⁹⁾ i.e., the larger hardening effect in Nb can be simply explained by the higher solubility limit of interstitial impurities as compared to the other b.c.c. metals such as iron, tungsten and molybdenum. Thus the striking hardening in Nb can be understood with certainty from the above argument. However, none of the arguments presented to date, can explain the strength of the ideally pure b.c.c. materials, without invoking certain inherent assumptions such as a scavenging effect or a solid solution softening effect. It is, therefore, evident that the problem is not really solved at the present time.

In electron irradiated f.c.c. metals a large increase in the flow stress at low temperatures has been confirmed,⁽⁵⁵⁻⁵⁹⁾ thus indicating that the rate controlling mechanism is strongly affected by the irradiation-induced defects. Moreover, according to their results, the hardening effect is significant only in the as-irradiated stage and it disappears on annealing at slightly higher temperatures corresponding to the defect annihilation. Thus the temperature dependent hardening in electron irradiated f.c.c. metals has been concluded to be due to dispersed interstitial atoms or Frenkel pairs.

There is, however, no report on the effect of electron irradiation on the strength of b.c.c. metals while a few studies have been reported on the effect of the neutron irradiation. Among the limited number of works on b.c.c. metals, there is already a controversy regarding the role of the neutron-irradiation-induced defects in the rate controlling mechanism of the plastic deformation of b.c.c. metals. For example, temperature

dependent hardening has been reported by Seidel⁽⁶⁰⁾ and Kitajima⁽⁶¹⁾ on iron, and by Ohr et al.,⁽⁶²⁾ on niobium, while temperature independent hardening has been reported by Schmidt⁽⁶³⁾ and McRickard⁽⁶⁴⁾ on iron, and by Arsenault and Pink⁽⁶⁵⁾ on molybdenum. On the other hand, according to Oku and Galligan,⁽⁶⁶⁾ the hardening due to fast neutron-irradiation on zone-refined tungsten appears to be strongly temperature dependent at lower temperatures (below 80°K) but at higher temperatures the temperature dependence of the critical resolved shear stress is not altered by the irradiation. Thus the overall situation in the neutron-irradiated b.c.c. metals is rather confusing.

In the case of neutron-irradiated f.c.c. metals the temperature dependent hardening has been observed,⁽⁶⁷⁾ and Koppenaal and Arsenault⁽⁶⁷⁻⁶⁹⁾ attributed this hardening to the irradiation-induced defect clusters by applying the Fleisher model.^(70,71) Mifune⁽⁵⁹⁾ has, however, pointed out that the application of the Fleisher model, as well as the representation of a defect cluster by a dimensionless misfit strain center, is questionable since the nature of the interaction concerned must be entirely different from that for an atomic defect (interstitial or Frenkel pair). The hardening due to neutron irradiation is often interpreted by means of a depleted zone theory⁽⁷²⁾ as it is substantiated by the observations of defect-free zones after deformation in neutron-irradiated⁽⁷³⁻⁷⁵⁾ or quench-aged⁽⁷⁶⁻⁷⁸⁾ f.c.c. metals, and also in neutron-irradiated b.c.c. metals.^(79,80) Thus, besides the aforementioned confusion in the results on neutron irradiated b.c.c. metals, the application of the neutron-irradiated data to the discussion of the importance of interstitial impurities on the rate controlling mechanism is not appropriate.

In order to get a conclusive answer to the question about the

relative importance of the inherent lattice resistance and the resistance due to the dispersed obstacles, a critical experiment must be carried out. Such an experiment has been attempted in the present study. In the present experiment, the effect of electron irradiation on the strength of b.c.c. iron has been studied at low temperatures. The electron irradiation produces atomic scale defects, while the defects produced by the bombardment of heavier particles such as neutrons, protons, and α -particles are generally defect clusters and, as mentioned above, the interaction mechanism with dislocations may not be relevant to the case of interstitial impurities. Thus it is the important merit of the electron irradiation that a controllable number of atomic scale defects (self-interstitials and Frenkel pairs) can be introduced at any stage of the deformation without changing the dislocation structure in a specimen. Many experimental and theoretical studies have demonstrated that the interaction between interstitials (or Frenkel pairs) and dislocations is basically elastic and that the agreement between experimental results and elasticity calculations is good. Therefore the nature of the dislocation interaction with self-interstitials (and Frenkel pairs) can be considered to be similar to that with impurity interstitials. In particular, the strong evidence observed for severe hardening in electron irradiated f.c.c. metals justifies the use of the electron irradiation technique in the study on iron to determine the importance of interstitials on the rate controlling mechanism in b.c.c. metals.

In the course of the present investigation, special care has been taken to minimize the complication due to the residual impurities, dislocation structures and sub-boundaries. To accomplish this, clean single crystals have been grown from zone-refined ultrapure iron supplied by the

Battelle Memorial Institute. Every tensile specimen was subsequently purified by a dynamic ZrH_2 system just before testing. The irradiation effects have been studied in both the micro and macroyield regions at temperatures from 4.2°K to 373°K.

From a theoretical point of view, the impurity hardening has often been interpreted by the tetragonal distortion hardening theory given by Fleisher.^(70,71) In his model a rigid dislocation is considered. Therefore the total interaction energy of a screw dislocation with a defect which has spherical symmetry is zero, but the interaction with a tetragonal defect can cause appreciable hardening for a certain orientation of a tetragonal defect with respect to a dislocation direction. Because of the importance of the tetragonality, this model has been quoted for explaining the nature of the deformation of b.c.c. crystals, where the stress field around an interstitial impurity is considered to be tetragonally distorted. Recently, however, Mifune and Meshii⁽⁸¹⁾ have shown the presence of an interaction between a screw dislocation and a spherical defect by taking account of the splitting of a dislocation, even if a rigid dislocation model is used. The width of the split, giving the maximum interaction, is found to be relatively small (a few atomic distance), according to their results. Thus the tetragonality cannot be the necessary condition for producing a large interaction between a screw dislocation and a point defect.

The first explanation for the increase in the athermal stress component due to the misfit strain centers was given by Mott and Nabarro.^(82,83) They have estimated the elastic interaction between an edge dislocation and a misfit strain around an impurity. This athermal hardening theory was then developed by Friedel,⁽⁸⁴⁾ by making a correction

for the average loop length. Shoenck and Seeger⁽⁸⁵⁾ have also proposed the athermal hardening due to a dragging force exerted on a dislocation by a Snoek ordering.⁽⁸⁶⁾

The aforementioned theories are all about the interaction between dislocations and dispersed obstacles. What follows is a review of the theories which have been developed in order to explain the large temperature dependence by means of the inherent lattice hardening mechanism. A double kink nucleation model was first proposed by Celli et al.⁽⁸⁷⁾ and Dorn and Rajnak,⁽⁸⁸⁾ as the rate controlling process for dislocation motion during plastic deformation. Dorn and Rajnak used a sinusoidal function for representing the lattice potential, and an effect of a slight perturbation on the function was also studied, while Celli et al. assumed a parabolic function. In their theory a double kink nucleation model used by Seeger,⁽⁸⁹⁾ has been developed to be applicable under high applied stress. In their calculation, an infinitely long dislocation line was considered for mathematical convenience. Therefore, their model corresponds to the dislocation motion in a perfect lattice. A number of experimental results have shown good agreement with the theoretical prediction. However the determination of the absolute magnitude of the Peierls stress by theoretical calculations has been somewhat unsuccessful, predicting a range of Peierls stress over several orders of magnitude. Therefore, the agreement found in these works may give self-consistency between the theory and the experiment but it cannot provide any conclusive answer to the question of the role of interstitial impurities.

The double kink model has been further modified to explain the asymmetric nature associated with the large temperature dependence of the flow stress. For example, Dorn and Mukherjee⁽⁹⁰⁾ have introduced an

asymmetric nature to the Peierls potential. Duesbery⁽⁹¹⁾ has studied the double kink nucleation on a dissociated screw dislocation. These studies have again been limited to the case of a perfect lattice and any conclusive statement about the role of interstitial impurities has not been given.

Arsenault⁽⁹²⁾ considered a double kink nucleation on a finite dislocation segment and reached basically the same conclusion as given by Dorn and Rajnak.⁽⁸⁸⁾ Ono and Sommer⁽⁹³⁾ recently re-evaluated Arsenault's results by assuming infinite pinning strength at the ends of a dislocation segment, thus introducing the concept of defect concentration into Peierls mechanism. They found an athermal hardening due to a relatively high concentration of pinning points at a higher temperature range, but no effect was found at lower temperatures.

The important assumption made in their calculation was the absolute pinning of a screw dislocation segment at its boundaries, namely, the pinning strength of interstitial atoms is considered to be infinite. This is clearly an unrealistic assumption. Furthermore, this assumption implies that the bow-out of the dislocation from this pinning segment would be the controlling process for the dislocation motion corresponding to the macroscopic plastic deformation. However, the overcoming of the first Peierls potential hill was taken as the rate controlling process in their calculation. Therefore, the results of the calculation are in reality not related to the macroscopic plastic deformation. In order to overcome the above difficulty and to give a realistic model of dislocation motion in the combined field of Peierls potential and the interstitial interaction, a finite and realistic interaction between the dislocation and an interstitial must be introduced.

The explanation of the solid solution softening has also been studied by modifying the double kink nucleation mechanism by Ahmadich et al.⁽⁹⁴⁾ Arsenault⁽⁹²⁾ suggested possibility of decrease in Peierls stress by the introduction of substitutional or interstitial impurities. A localized double kink nucleation model was considered by Weertman,⁽⁹⁵⁾ and recently in detail by Urakami.^(96,97) Urakami examined a preferential double kink nucleation around a misfit strain center by taking account of the interaction between a screw dislocation and a misfit defect. In the results, the softening effect at lower temperatures was qualitatively explained but the hardening in the higher temperature region could not be explained.

None of the existing theories have given a satisfactory conclusion on the relative importance of the interstitial impurities and the inherent lattice. In the present study, the local double kink nucleation model presented by Urakami has been extended to a stronger misfit defect case, where the dislocation motion cannot be controlled only by the double kink nucleation. Using this method, the effect of misfit strain centers on the rate controlling process of plastic deformation in b.c.c. lattice can be studied without invoking the unrealistic assumptions involved in the earlier works, such as infinite strength⁽⁹³⁾ or zero strength⁽⁸⁸⁾ of misfit strain centers. The energetics for the dislocation motion through Peierls potentials and misfit strain centers should give the answer to the relation between the experimental observation and the relative importance of misfit strain centers and inherent lattice. A model for the nature of the concentration dependence, as well as the temperature dependence of the flow stress is presented. The effect of the tetragonal misfit defects is also included in the present study.

II. EXPERIMENTAL PROCEDURE

1. Specimen preparation

The materials used in the present study were Ferrovac-E iron and zone-refined ultra-pure iron supplied by the Battelle Memorial Institute. Ferrovac-E iron is reported to have 150 ppm of non-metallic impurities and 300 ppm of metallic impurities, where the Battelle iron contains less than 5 ppm of total non-metallic impurities and 31 ppm of metallic impurities as listed in Table I.

Three types of single crystals were prepared from these materials; (a) single crystals grown from the Ferrovac-E iron (specimen F), (b) single crystals grown from the Battelle iron (specimen B), and (c) single crystals cut directly from a coarse grained Battelle iron (specimen C). In addition to these single crystals, polycrystalline thin sheet specimens were also prepared from the Battelle iron to study the production rate and the recovery of the electron irradiation-induced defects by electrical resistivity measurement.

a) Single crystal growth:

The usual strain anneal method⁽⁹⁸⁻¹⁰⁰⁾ was used for growing single crystals from the Ferrovac-E iron. Three pieces of 1 x 1/4 x 3 inch blocks were cut from as-received Ferrovac-E iron, and cold rolled down to a 50 mil thickness. The thin sheets were then chemically polished with a mixture of 95% hydrogen peroxide and 5% hydrofluoric acid, to remove the surface contamination. These sheets were subsequently annealed under a dynamic vacuum of better than 10^{-6} mmHg for 1 hour at 800°C. After this recrystallization annealing, a total prestrain of 0.05 inch/inch was given at a strain rate of 10^{-3} per second. These optimum conditions for recrystal-

Table I List of metallic and non-metallic impurities
in the Battelle iron as detected by mass
spectrometer and internal friction.

Non-metallic Impurities

Oxygen	2
Nitrogen	0.1
Hydrogen	0.1
Carbon	1.7
Sulfur	1
<hr/>	
Total	5

Metallic Impurities

Chromium	2
Cobalt	3
Copper	2
Manganese	10
Tin	2
Titanium	2
Tungsten	0.1
Other than listed above	9
<hr/>	
Total	31

lization annealing, strain rate and total strain to grow single crystals were given in the work by Takeuchi.⁽¹⁰⁰⁾

Gradient annealing of the prestrained sample was carried out under a dynamic vacuum of better than 10^{-6} mmHg with the maximum temperature gradient of $800^{\circ}\text{C}/\text{inch}$. The sample was passed through the temperature gradient at a speed of $3/4''$ per hour, by lowering the sample through a water-cooled copper throat into a furnace at 890°C . A schematic drawing of the system is shown in Fig. 1, where the clearance of the throat for a sample pass was $1/8 \times 1$ inch.

By the above-mentioned method, large single crystal sheets of $1 \times (5 \sim 7) \times 0.05$ inch were obtained from most of the samples. Although the as-grown single crystals had many small island grains on their surfaces, all the smaller grains were removed, after removing 15 mil from the surfaces by chemical polishing. The regions where larger grains remained were avoided when specimens were cut out from the single crystal sheets. Thus single crystals which were free from the island grains, were prepared from the Ferrovac-E iron.

In the case of the Battelle iron, cold rolling and subsequent annealing in the temperature range of $600 \sim 890^{\circ}\text{C}$ did not result in a uniform grain size, and hence we did not succeed in preparing a single crystal in sheet form. However, according to the work by Hughes and Barton,⁽¹⁰¹⁾ cold swaging followed by annealing at a relatively low temperature gave rods of a fairly uniform grain size. These rods were then successfully grown into single crystals. A $5/8''$ diameter rod was prepared from the as-received Battelle iron and swaged down to $1/8''$ diameter by a water-cooled swaging machine. After removing the surface contamination by chemical polishing, recrystallization annealing was performed at 640°C

Fig. 1 Temperature gradient furnace for growing single crystals. The single crystal specimens F and B were grown in this furnace. The maximum temperature gradient was $800^{\circ}\text{C}/\text{inch}$ at the tip of the water-cooled copper block. Lowering speed of a sample into the furnace was $3/4''$ per hour.

for 30 minutes, resulting in fairly uniform grain size of approximately 0.2 mm in diameter. Then a prestrain of 0.02"/inch was given at a strain rate of 5×10^{-4} per second, and the specimen was subsequently annealed in the gradient furnace which was used for growing Ferrovac-E iron single crystals. The growing conditions were the same as for the Ferrovac-E iron. By this method single crystals of 2 ~ 6 inches in length were obtained from most of the rod samples.

b) Preparation of tensile specimens from the grown single crystals:

The crystallographic orientations of the grown single crystals were determined by the usual back-reflection Laue method. Two examples of the longitudinal axes for Ferrovac-E iron are given in Fig. 2(a), and traces of possible tensile axes, which can be obtained by cutting the large single crystals, are shown for each single crystal sheet by broken lines. The solid lines with arrows indicate the cutting angle with respect to the longitudinal axes of the large single crystal sheets. The tensile axes prepared from these specimens are given by the cross marks. As seen in this figure, the choice of tensile axis was widely-ranged and most of the grown single crystals could be cut to tensile specimens of the desired orientation.

On the other hand, in order to obtain a tensile specimen of 0.020 x 0.100 x 0.625 inch from the Battelle single crystal rod of 1/8" diameter, the slicing angle to the longitudinal axis was limited to be within 5°. Consequently the fraction of single crystals which could be cut to the desired orientation, was only half of the single crystals grown from the Battelle iron. The longitudinal axes of the Battelle single crystal iron rods are shown in Fig. 2(b), where an example of the possible

choice of tensile axis orientation is given by a shaded area.

Tensile specimens were cut or sliced from these single crystals by using a thin grinding wheel, to give a maximum Schmidt factor for the $\langle 111 \rangle \{110\}$ type of slip system. After polishing these tensile specimens down to 15 mil thick by chemical polishing, each specimen was cut to the final shape with shoulders by using a spark cutting machine. An example showing an as-grown Battelle single crystal rod, a slice cut from the rod and a final specimen, appears in Fig. 3. Two small holes at the ends of the specimen are for hanging the specimen in a vertical furnace during the purification treatment. The final dimension of the specimen was 9 ~ 11 mil thick and 50 mil wide within the gauge length, and the uniformity of the thickness was within ± 0.05 mil.

c) Purification by ZrH_2 :

The purification system used in the present study was similar to, but slightly modified from the one developed by Stein et al.,⁽¹⁾ so that a small vibrational stress can be applied to a specimen during purification process. This vibrational stress is expected to assist the purification process since the small stress applied to a specimen will cause vibration of dislocations and will help the break-away of interstitial impurities from Cottrell atmospheres. A schematic drawing of the entire system is shown in Fig. 4, and the detail of the specimen assembly is shown in Fig. 5. To exert a vibrational stress on a specimen, a 5 gm weight was hung from the bottom of the specimen and the specimen was vibrated by an electromagnet placed above the specimen. The amplitude of the vibration with the frequency of 60 cps was adjusted by controlling the current fed into the magnet to give a slightly lower stress than the yield stress of the specimen at 850°C. The calibration of the stress amplitude was done by

Fig. 3 Preparation of a tensile specimen from the Battelle iron.

- a) A single crystal rod grown by the strain anneal method.
- b) A single crystal sheet sliced from the single crystal rod.
- c) Final shape of a specimen.

Fig. 4 Schematic drawing of the ZrH₂ purification system. Hydrogen was circulated in a closed system. A specimen was hung in a vertical furnace as shown in the next figure.

Fig. 5 Detail of a specimen assembly in ZrH_2 system. A magnet placed above a specimen was used to generate vibrational stress on the specimen with the aid of a light weight hanging from the specimen.

repeated trial and error. When the stress exceeded a critical stress corresponding to the yield stress, the specimen which had undergone the vibrational stress showed significant deformation and polygonization. However if the stress was slightly lower than the critical stress, the specimen did not show any apparent deformation. The power giving this critical stress was 2.8 watt, and the one used during the purification process was 2.3 watt.

The system mentioned above was operated in the following manner:

- (1) The system was pumped down to 20μ of mercury and 99.9999% pure hydrogen was admitted to 100 mmHg into the system. Then the system was again pumped down to 20μ .
- (2) After shutting off the evacuation line, hydrogen was introduced to 540 mmHg and the hydrogen was circulated through a cold trap, ZrH_2 furnace and specimen furnace by a Randolf pump.
- (3) The furnaces were then warmed to $800^\circ C$ for the ZrH_2 and $850^\circ C$ for the specimen, resulting in approximately 790 mmHg, which is slightly higher than atmospheric pressure. The time required to reach the desired temperatures was a half an hour for both of the furnaces.
- (4) Proper current was supplied to the magnet to generate the desired vibrational stress on a specimen and held for two hours.
- (5) In order to reduce the residual dislocations, the purification was continued for .5 hours, after switching off the magnet.
- (6) The specimen was then furnace cooled and it was used within a few hours after taking it out of the system.

2. Irradiation system

The high energy electron beam for irradiation was generated by the 2 MeV High Voltage Engineering Van de Graaff Accelerator. The incident energy of 1.3 MeV was used throughout the present experiments, except for a few preliminary experiments involving electrical resistance measurement. The energy meter was already calibrated by the former worker⁽⁵⁹⁾ by using the reaction $\text{Be}(\gamma, n) \text{B}$ at 1.66 MeV. The stability of the incident electron energy was better than ± 0.02 MeV during all irradiation experiments.

A 5 meter long connecting drift tube between the accelerator and a cryostat was aligned by checking the beam position in the tube. The beam position was revealed by placing a polyvinyl chloride sheet at several connections in the drift tube. The beam position was then centered with respect to the drift tube.

The less uniform regions of the beam around the beam edge were cut off by a 1/8" thick copper collimator which was made adjustable by aligning two plates placed on the collimator. The opening of the collimator had to be as small as possible, within the safety limit for covering the entire specimen area, to minimize the liquid helium consumption rate during irradiation. Thus the opening of 3 mm x 11 mm was used for a specimen of 1.3 mm x 9 mm.

The drift tube, the collimator and the cryostat were all electrically insulated from each other and also insulated from the ground, so that the electrons hitting each section could be measured separately. To determine the electron dose, the electrons passed through the collimator and stopped in the cryostat were counted by an Elcor A 309 A Current Integrator. In the present setup, because of a window placed between a collimator and a specimen, the above-mentioned counting does not give a real value at the

specimen position. However, the electrical resistance measurements of the irradiation-induced defects enabled one to estimate the effect of the window. Correction factors for evaluating the real dosage at the target are estimated in Appendix C on the basis of the multiple scattering theory.

3. Liquid helium cryostat and temperature control

All low temperature studies were done in a stainless steel dewar built by Andonian Associates, Waltham, Massachusetts. A cylindrical specimen chamber of the dewar was open to atmospheric pressure and the chamber was separated from the vacuum by a thin-wall window. A 10 mil thick beryllium window sealed with an indium O-ring was originally built in. However, because of vacuum failure after repeated temperature cycling, the window was replaced by a 1 mil thick stainless steel foil.

The cryostat is equipped with a liquid helium container well which is connected to the specimen chamber through an adjustable throttling system. For a long run of irradiation the total liquid helium consumed exceeded 10 liters. The helium container well could only store 3 liters but it could be refilled easily without emptying the specimen chamber, so that a high electron dosage could be carried out without disturbing the specimen. A schematic drawing of the cryostat is given in Fig. 6.

The specimen temperature was monitored by two copper vs. gold 2.1 at.% cobalt alloy thermocouples, attached on the tensile jig above the upper specimen grip and below the lower grip. In order to keep the specimen temperature below 60°K during electron irradiation, the liquid helium consumption rate was 1.5 liter per hour for the irradiation current of 15 μ A at 1.3 MeV. This high consumption rate was achieved by pressurizing the liquid helium container well and pushing liquid helium into

Fig. 6 Liquid helium cryostat

- A ELECTRICAL FEEDTHROUGHS
- B OFF GAS PORT
- C TOP WORKS
- D HELIUM FILL AND VENT
- E NITROGEN FILL
- F NITROGEN VENT
- G DEWAR BODY OUTER SHELL
- H NITROGEN RESERVOIR
- I HELIUM WELL
- J VACUUM INSULATION
- K NITROGEN TEMPERATURE RADIATION SHIELD
- L OUTER TAIL
- M SAMPLE ZONE
- N DEMOUNTABLE WINDOWS
- O HEAT EXCHANGER/DIFFUSER
- P VAPORIZATION HEATER
- Q CAPILLARY
- R THROTTLE VALVE
- S THROTTLE VALVE STEM
- T THROTTLE VALVE TOP WORKS
- U TEMPERATURE CONTROL HEATER

the specimen chamber through a small hole in the throttling system. Temperature control for tensile tests or post-irradiation aging at below 150°K was accomplished by adjusting the opening of the throttle and by supplying the desired current through a heater which is installed at the outlet of the throttling system. By this method the temperature was controlled within 0.5°K at 30°K and 0.2°K at 60°K.

Tensile testing and post-irradiation aging above 150°K were not carried out in this helium dewar. For a temperature between 170°K and 0°C, an ethyl-alcohol bath precooled with liquid nitrogen was used, and a regulated glycerol bath was used for a temperature above 24.5°C.

4. Electrical resistivity measurement

Electrical resistance was measured by a standard potentiometric method, by using the assembly of a Hewlett-Packard 147 Nanovolt Null Detector, a Honeywell Rubicon 6 Dial Potentiometer, and a Harrison 6101-A DC Power Supply. The accuracy of the measurement at liquid helium temperature was about $10^{-11} \Omega \text{ cm}$ for a specimen of 1.0 cm in the length between two potential leads and $1.0 \times 10^{-3} \text{ cm}^2$ in cross section. The resistivity measurement was intended only for a preliminary study of the production rate and recovery of the irradiation-induced defects. Therefore only the initial portion of the recovery of the electron irradiation-induced defects was studied. The resistivity increase due to the electron irradiation was measured in liquid helium after the irradiation at the incident energy of 1.2 MeV for a beryllium window and 1.3 MeV for a stainless steel window. The recovery was studied up to 110°K. All resistance measurements were carried out with the specimens immersed in liquid helium.

5. Tensile test and microstrain measurement

The tensile machine used in the present experiment was the same one as used previously by Ono, Mifune and Meshii^(59,102) which consists of a strain gauge cell and an amplifier, a driving motor and a gear box, and an X-Y recorder. By using this system, the change of pulling rate and measurement of load could be done in a way similar to that of a commercial Instron type of tensile machine. The measurement of the load was done with an accuracy of better than 0.5% of the load.

To utilize this system for microstrain measurement, a capacitance type strain measurement device was added. The strain signal from a pair of parallel capacitance plates installed on a tensile jig was fed into the x-axis of the recorder. The capacitance measuring system was the assembly of a 1615-A Capacitance Bridge and a 1311-A Audio Oscillator built by General Radio Company, and a HR-8 Lock-in Amplifier built by Princeton Applied Research. A schematic drawing of the measuring system is given in Fig. 7.

The capacitance plates installed on a tensile jig were made of two parallel brass plates. To insulate these plates from the tensile jig, they were attached on it through Teflon plates. To prevent the Teflon insulators from being damaged by the irradiation, the two capacitance plates were placed 1" above the upper specimen grip and a protector block made of stainless steel was placed above the upper specimen grip. The upper plate unit was attached to the pulling rod through a thread with 25 mil pitch, and the bottom plate was fixed to the outside cylinder of the tensile jig. The arrangement of the specimen grips and the capacitance plates is shown in Fig. 8. The specimen grips were placed close to the window in the cryostat, so that the scattering effect due to the window could be minimized.

Fig. 7 Schematic drawing of the capacitance and load measuring assembly.

Fig. 8 The arrangement of capacitance plates, a specimen and a tensile jig. The gap between the two capacitance plates were made adjustable at the top of the tensile jig.

The capacitance between two parallel plates is theoretically given by the relation $C = 0.0885 \text{ (pF/cm)} kA/d$ where C is the capacitance in pF, k is the dielectric constant given by ϵ/ϵ_0 , A is the area of the plate (cm^2) and d is the spacing between the two plates (cm). A was 1.752 cm^2 , and typical value of d used in the present experiment was $0.35 \sim 0.45 \text{ cm}$. The values of k for oxygen, nitrogen and helium are 1.00048, 1.00054 and 1.055 at 1 atm., respectively. Thus the capacitance must be given by $C = 0.155/d \text{ (pF)}$. However, the measured capacitance did not obey this theoretical relation because the electrode plates were too thick ($1/8''$) and the effect of the plate edges appeared appreciable. The determination of the C vs. d relation in the present setup was made both in helium gas at 60°K and in air at room temperature. The C vs. d curves for both cases were almost identical within the experimental error. The spacing, d , was measured at the cross-head position by an extensometer ($.01 \text{ mm div.}$) attached to it. Since the zero spacing corresponds to a short circuit in the measuring system, the initial level of the cross-head position could be uniquely determined. Then the deviation from the initial level, exactly corresponding to the spacing between the two plates, was measured. Thus, the determined C vs. d curve is given by a solid line in Fig. 9. The value for $\Delta C/\Delta d$ derived from the solid line is also shown by a broken line in the figure. The microstrain as well as macroscopic strain was measured according to Fig. 9. For the microstrain measurement, the total strain was approximately 10^{-2} including an elastic strain. The strain 10^{-2} corresponds to $d - d_0 = 0.090 \text{ mm}$ for a specimen gauge length of 9 mm . The sensitivity and stability of the capacitance measuring assembly was better than 0.00001 pF corresponding to 10^{-6} strain. However because of the mechanical instability and imperfection, the accuracy of the measurement

Fig. 9 Calibration curve for capacitance vs displacement relation in the present setup. The solid line indicates the measured C vs. d relation. The broken line indicates the derivative of the solid line.

was approximately 2×10^{-5} in shear strain. The primary interest concerned in the present study can be achieved with this accuracy.

III. EXPERIMENTAL RESULTS

1. Plastic deformation of ultrapure single crystalline iron

a) Effect of purification on the ductility of iron single crystals at low temperatures

In order to demonstrate the effect of the ZrH_2 treatment, Ferrovac-E iron single crystals (specimen F) oriented for the $\langle 111 \rangle \{110\}$ type slip system were deformed at liquid nitrogen temperature without any prior straining. A nominal strain rate of 5×10^{-4} per second was used for every test in this experiment. The results for the tests after the different annealing treatments are demonstrated in Fig. 10, where resolved shear stress and shear strain were computed by assuming the operation of only the $\langle 111 \rangle \{110\}$ type slip. The slip line observation made on the deformed specimen revealed that the main slip system was actually the $\langle 111 \rangle \{110\}$ system. The specimen treatment is indicated on each curve in the figure. The crystal annealed under vacuum of better than 10^{-6} mmHg broke at the stress of 28 Kg/mm^2 without showing any observable ductility. The crystal treated with ZrH_2 exhibited appreciable ductility. When a vibrational stress was applied to the crystal during the purification, a smooth stress-strain curve without any twinning was obtained. A stress-strain curve determined for the Battelle iron single crystal (specimen B) which was oriented with nearly the same tensile axis as the Ferrovac-E iron single crystals and which was treated with ZrH_2 under a vibrational stress, is also given in Fig. 10. As seen in the figure, the ductility of the crystal increased drastically after the ZrH_2 treatment but the change in the flow stress was not significant. It should be pointed out here that a sharp yielding (usually without a yield drop) occurred in the specimen

Fig. 10 The effect of purification on the ductility of iron single crystals. A drastic change in the stress-strain curves for the Ferrovac-E iron (specimen F) must be noted, due to the purification treatment.

deformed at 77°K without prior deformation. Therefore, the determination of the macroscopic yield stress was relatively unambiguous. This yielding behavior should be compared to those for the specimens prestrained at ambient temperature. In those specimens, the gradual deviation of the stress-strain curve from the linear elastic region occurred considerably below the present yield stress level. This fact is clearly demonstrated in Fig. 15, which appears in the later section (III-1-c, p. 42). Thus a considerable ambiguity exists in determining the yield stress of these specimens. This difficulty partially contributed to the controversy on the low temperature strength mechanism of iron specimens in the past.⁽¹⁾ The different yielding behavior of iron single crystals with or without prestraining will be discussed in conjunction with the movement of edge and screw dislocations at different stress levels in the discussion chapter (p. 127).

b. Temperature dependence of flow stress and strain rate sensitivity

Determinations of flow stress and strain rate sensitivity at temperatures between 4.2°K and 350°K were carried out for single crystal specimens F and B. The flow stress of an unprestrained specimen was taken at the intersection of the extension of the elastic line and the initial portion of work-hardening. This flow stress is plotted against deformation temperature in Fig. 11. The flow stress at 4.2°K was determined after 3% prestrain at 60°K because the specimen did not show any ductility at 4.2°K without prestraining. The flow stress of the vacuum annealed specimen F is also given in the same figure, although data could not be obtained at lower temperatures because of brittle fracture for this less pure specimen.

In this figure it should be noted that the flow stress for specimen F was always higher than that of specimen B. However, the difference in the

Fig. 11 Plots of initial flow stress vs temperature. No prior straining was given except the test at 4.2°K. Circle marks and cross marks indicate the purified specimen B and the vacuum-annealed specimen F, respectively.

flow stress between the two specimens decreased when the temperature was lowered, giving the smaller thermal component of the flow stress to the impure specimen. A similar observation was also reported by Nakada and Keh⁽¹³⁾ in the Fe-N system. The stress-strain diagrams for the ZrH₂ treated specimen B are illustrated in Fig. 12, where the strain rate was cycled between 10^{-3} and 5×10^{-3} , and the testing temperature is noted on each curve. No prestrain was given prior to testing, except for the two tests at 58°K and 4.2°K in which 3% prestrain was given at 24.5°C. These two curves are given here in order to show the effect of prestrain at a high temperature on the initial portion of a stress-strain diagram.

From the strain rate cycling data given in Fig. 12, the strain rate sensitivity, $\Delta\tau/\Delta\ln\dot{\gamma}$, was obtained by taking the average of the values for the different strains. In the case of the tests without prestraining, the value did not show any systematic change with the strain. The results are represented by circles in Fig. 13. For the prestrained specimens the value was smaller in the initial portion of the stress-strain curve, as represented by diamond-shaped marks in the figure, and increased with strain as indicated by dots. There are two interesting features to be noted in addition to the above-mentioned effect of prestraining, namely, the plateau observed for the unprestrained specimen at temperatures between 80°K and 150°K, and the shift of the maximum strain rate sensitivity position to the higher temperature side for the purer specimen. Both the temperature range and the value of $\Delta\tau/\Delta\ln\dot{\gamma}$ at the plateau found in the present study are consistent with the work by Michalak.⁽¹⁰³⁾ The temperatures at which the maximum strain rate sensitivity was obtained were 190°K for specimen F and 250°K for specimen B in the present experiment. The temperature, 250°K, for specimen B is much higher than the temperature,

Fig. 12 Strain rate cycling test at different temperatures. All tests were done without prior straining except for the 4.2°K and 58°K tests.

Fig. 13 Plots of strain-rate sensitivity vs temperature. Circles are for specimen B and cross marks for specimen F. No prestrain was given prior to the test. Diamond marks are for the prestrained specimen B. Dots indicate the increase of the strain-rate sensitivity with strain for the prestrained case.

180°K, given in the earlier works. (13,103,104)

c) The effect of prestrain on a stress-strain curve in the microyield region

The plasticity of iron single crystals in the microyield region was also studied at low temperatures after giving various amounts of prestrain at various temperatures. For a preliminary experiment, the ZrH_2 treated specimen F was used, and the same strain rate of 10^{-3} per second was used for all the microstrain experiments.

The effect of the prestrain temperature on the stress-strain curve is demonstrated in Fig. 14 where each specimen was prestrained to 6×10^{-2} at different temperatures prior to testing. The test temperature was fixed at 77°K for all curves shown in the figure. It is clearly seen in the figure that the plasticity in the microyield region is strongly dependent on the prestrain temperature and that the plastic strain in the microyield region is considerably larger for the higher prestrain temperature.

The amount of prestrain also changes the stress-strain behavior, as observed by several authors. (43,48-50) Such a test in the present experiment is demonstrated in Fig. 15 where the prestrain temperature and the test temperature were fixed at 24.5°C and 77°K respectively, and the amount of prestrain was changed from 0 to 6×10^{-2} . The result obtained here is consistent with the earlier works.

This inelastic behavior in the microyield region has often been explained by the exhaustion-hardening theory. Our purpose here is to investigate the effect of electron irradiation on the plasticity in this microyield region. The results should enable one to examine the mechanism of the interaction between dislocation and electron irradiation-induced defects and to examine various mechanisms proposed for the microplasticity in iron.

Fig. 14 Effect of prestrain temperature on a stress-strain curve in the microyield region. A fixed amount of prestrain, 6×10^{-2} , was given for every test at different temperatures indicated on the curves. Drastic change in the plasticity in the microyield region due to the prestraining should be noted. The accuracy of the strain measurement was 2×10^{-5} . Thus the arrows indicate the appearance of the deviation of 2×10^{-5} strain from an elastic line.

Fig. 15 Effect of the amount of prestrain on a stress-strain curve in the microyield region. Note that the as-annealed specimen also shows the deviation from an elastic line at a relatively low stress level.

2. Production rate and recovery of defects measured by electrical resistivity

The resistivity of a polycrystalline specimen, which was prepared from Battelle iron and was subsequently annealed at 850°C in a ZrH_2 purification system without vibrational stress, was measured to be $9.9 \times 10^{-6} \Omega \text{ cm}$, giving a resistivity ratio, $\rho_{24.5^\circ\text{C}}/\rho_{4.2^\circ\text{K}} = 143$, while the same specimen gave the ratio of 80 when simply annealed under a vacuum of 10^{-6} mmHg . The ratio, 80, obtained for the vacuum-annealed polycrystalline iron prepared from the as-received Battelle iron is considerably low compared to 280, the one reported by the Battelle Memorial Institute. This difference is significant and no one can deny that the specimen must be contaminated in some stage of the preparation or during the period kept in air at room temperature. Since the specimen was never warmed up above room temperature except when it was annealed under vacuum, the most probable explanation for this is that some gaseous impurity (presumably N_2) might have diffused into the specimen when it was kept in air at room temperature. This is the reason why every specimen was purified just before testing throughout the present study. After the ZrH_2 treatment, the ratio of the same specimen increased to 143 although this is still not as high as the reported number. The main reason for this difference is considered to be the error in the resistance measurement at 4.2°K due to elastic strain: Since the resistance was measured on a specimen 2 mil thick in the present experiment, the elastic strain which could occur at 4.2°K might be considerably large. According to the report by Rongstorff,⁽¹⁰⁵⁾ the resistivity ratio measured in a specimen of 50 mil diameter was changed from 270 to 200 only by changing the contact pressure of potential lead wires attached to the specimen. Thus, in order to obtain correct resistance ratio of pure iron, a special

care must be taken to avoid this complication: It is, as suggested in the report, to use longitudinal magnetic field, which gives a saturation value of 700 for zero carbon concentration, and which drops down to 280 by a small addition of interstitial carbon. The resistivity ratio for the vacuum-annealed Ferrovac-E polycrystalline iron was also measured, and $\rho_{24.5^\circ\text{C}}/\rho_{4.2^\circ\text{K}} = 34$ was obtained.

The resistivity change due to irradiation and post-irradiation aging was measured on the above-mentioned purified Battelle iron. The resistivity increase due to 10^{17} e/cm² irradiation at the incident electron energy of 1.2 MeV was $0.39 \times 10^{-8} \Omega\text{-cm}$, when a 10 mil thick beryllium window was used and a specimen was placed at 8 mm from the window. When the beryllium window was replaced by a 1 mil thick stainless steel window and a specimen was placed at 11 mm from the window, the resistivity increase due to the same irradiation was reduced by approximately one half. During the course of irradiation the temperature was always kept below 25°K. The obtained resistivity increase, $\Delta\rho$, for these two runs is plotted against the electron dose in Fig. 16, where the dose given here is essentially counted at the collimator and the scattering effect due to the window is not taken into account. The broken line in the figure is taken from the data (irradiation, 1.4 MeV, 20.4°K) given by Lucasson and Walker.⁽¹⁰⁶⁾

The significant difference in the rate of the resistivity increase seen in the figure is obviously due to the scattering of electrons by the window. The real electron dose at the specimen position was estimated on the basis of the above experimental data and theoretical calculation which is described in Appendix C.

The recovery of the irradiation-induced defects was also studied by an isochronal annealing of 5 minutes at 10°K intervals. The normalized

Fig. 16 Defect production rate measured by electrical resistance measurement. Difference in the production rate is mainly due to the scattering of electrons at the window; namely, the number of the effective electrons hitting a specimen is not the one given in the scale in the figure.

Fig. 17 Recovery of the electron irradiation-induced defects. The main recovery starts at 80°K even in the ultra-pure iron. The solid line shows the data given by Lucasson and Walker. A slight enhancement for the substages below 60°K is noted.

recovery of the resistivity is plotted against the annealing temperature in Fig. 17. The present recovery study was made only in the low temperature region because the main interest here was to confirm the temperature at which the main recovery starts to take place. The decay curve drawn in the figure is taken from Lucasson and Walker.⁽¹⁰⁶⁾ As seen in this comparison, the temperature at which main recovery starts to take place appeared to be the same as for their results within experimental error. On the other hand a small recovery step at around 60°K seems to have been shifted slightly to the lower temperature side as indicated by a broken line for the present experiment. This is perhaps due to the difference in specimen purity since, in their study, vacuum-annealed Johnson Mathy iron ($\rho_{0^\circ\text{C}}/\rho_{20.4^\circ\text{K}} = 110$) was used, which is less pure than the present specimen.

3. Effect of irradiation on macroscopic flow stress

a) Tensile test at low temperatures

An important finding in the present experiment was "irradiation softening", namely the decrease of the flow stress due to electron irradiation. An example of the observation of the softening effect is demonstrated in Fig. 18. The shear stress-shear strain curves shown in the figure were obtained by a tensile test at 60°K and a strain rate of 10^{-3} per second. The ZrH₂ treated specimen B was deformed at 60°K to the first interruption without any prestrain at ambient temperature, and after complete unloading, the specimen was irradiated to the dose of 5×10^{16} e/cm² at or below 60°K. Then, again at 60°K the second portion of the stress strain curve was obtained. In the same manner the third curve was obtained after the total dose of 5×10^{17} e/cm². Throughout this series of experiments

Fig. 18 Electron irradiation softening observed at 60°K test. A specimen B was deformed by 5% prior to the electron irradiation to establish a stress level. The first and second interruptions are due to the irradiation and the third interruption is to study the effect of post-irradiation aging. A dosage-dependent decrease in the flow stress level must be noted. The post-irradiation caused a hardening effect.

the specimen temperature was kept at or below 60°K. As seen clearly in Fig. 18, a slight decrease in the flow stress was observed after the first irradiation of a small dose. This softening effect was significantly enhanced by the second irradiation with the higher dose. On the other hand, when the specimen was aged at 30°C for 15 hours, the irradiated specimen showed twinning, a slight yield drop, and an increase in flow stress, as seen on the fourth stress-strain curve.

The above softening effect was always observed in the B specimens purified adequately. However, the softening was not detected for specimens which showed twinning in the initial deformation. Twinning was always observed for specimen F when vibrational stress was not applied during the purification treatment, or sometimes even for specimens of B which were not purified adequately due to a vacuum failure of the purification system or due to the usage of worn-out ZrH_2 . An example of such a test is demonstrated in Fig. 19. The specimen used here is the ZrH_2 treated specimen F in absence of the vibrational stress.

The amount of the softening observed in a well purified specimen B is plotted against the electron dose in Fig. 20. The circles with a line in the center indicate the case when twinning occurred after the irradiation but not before the irradiation. The decrease in flow stress was determined by comparing the flow stress levels just before irradiation and the flow stress after irradiation where the stress after irradiation was taken at the intersection of the elastic line and the initial portion of the work-hardening.

Clearly the magnitude of the softening increases with the irradiation dosage, namely with the concentration of self-interstitials (or Frenkel pairs) up to the electron dose of $5 \times 10^{17} \text{ e/cm}^2$. Beyond this point, the

Fig. 19 No softening effect was found for a less pure specimen. A result for the specimen F treated with ZrH₂ without vibrational stress, is given here.

Fig. 20 Dosage dependence of the softening effect. The decrease in flow stress caused by electron irradiation is plotted against the logarithm of the electron dose. For the higher electron dose, above 10^{18}e/cm^2 , twinning was observed in some of the specimens.

magnitude of the softening effect appears to decrease. The experimental limitation* prevented us from repeating the high dose irradiation or extending the irradiation to a considerably higher dose than that examined.

It should be pointed out, however, that the mode of deformation changed after irradiations of higher than 10^{18} e/cm². In the case of the 10^{18} e/cm² irradiation, twinning occurred before the yielding and the specimen broke at a stress level lower than that before irradiation. The other example of the appearance of twinning by a high electron dose (1.5×10^{18} irr.) is shown in Fig. 28, which was observed in the microstrain experiment. In the case of the 2×10^{18} e/cm² irradiation, the specimen was tested at an intermediate dose (5×10^{17} e/cm²), and after the additional dose of 1.5×10^{18} e/cm² the twinning was not observed but at a slightly higher stress level than the prior stress level (after 5×10^{17} e/cm² irradiation) necking started. Thus the occurrence of twinning was not systematic in this range of defect concentration. The discussion for this induction of twinning due to the high irradiation dosage and its possible implications will be examined in the Discussion (p. 121). A theoretical reasoning for the existence of the critical concentration for the maximum softening due to interstitial atoms will also be presented in the Discussion.

On the other hand, the softening effect was not detected when a specimen was tensile-tested at 4.2°K as demonstrated in Fig. 21. This nature is an important observation for interpreting the irradiation softening by a local double kink nucleation model as will be discussed in a later section (p. 112).

* The dose of 2×10^{18} e/cm² can be obtained by an irradiation of 10 hours with the liquid helium consumption rate of 1.5 liter/hour.

Fig. 21 No softening was detected for the 4.2°K test. The electron irradiation caused a small yield drop as seen in the figure, but no hardening appeared for the lower yield stress.

b) Tensile test at ambient temperatures after post-irradiation aging

This experiment was carried out on specimen C without a purification treatment since the ZrH_2 system was not ready to use at the time. The orientations of tensile axes for specimen C are plotted in Fig. 22. The size of a grain in the as-received Battelle iron was not large enough to obtain specimens of the same orientation from a limited number of usable grains. In the figure the orientations determined after deformation are also given by the circles with cross marks.

Electron irradiation was always carried out in the cryostat at or below 80°K after establishing the flow stress level at a desired temperature. The irradiated specimens were immediately washed with ethyl alcohol without removing the specimen from the tensile jig, and then transferred into an annealing bath. After the desired annealing treatment, the specimen was removed from the bath and tested at the same temperature as for the initial tensile test. Representative resolved shear stress and strain curves for the tests are illustrated in Fig. 23. The slip was assumed to occur exclusively on a maximum resolved shear stress plane. The broken lines in the figure represent unirradiated cases and the solid line is for the irradiated specimen. It can be seen in the figure that the increase of flow stress (lower yield stress) due to the aging treatment was remarkably enhanced by the irradiation.

The relation of the irradiation with this hardening phenomena was studied quantitatively by choosing various combinations of aging temperature, aging time, and amount of electron dose. The flow stress increase after aging was defined by $\Delta\tau' = \tau_I - \tau_F$, where τ_F is the flow stress just before irradiation and τ_I is the interpolated lower yield stress after

Fig. 22 Tensile axes for specimen C. The direction of the change in the tensile axes determined after deformation are noted by arrows. The specimen numbers from 101 to 109 are for the specimens F.

Fig. 23 The enhancement of the age hardening due to the electron irradiation. Broken lines are for the unirradiated cases and the solid line for the irradiated case. Enhancement in the age hardening for lower yield stress is significant, while that for the upper yield stress is relatively minor. The definition of the parameters, τ_U , τ_I and τ_F , used in the text are also shown. Tensile test at 24.5°C.

irradiation and aging as indicated in Fig. 23. The stress levels of all single crystals were normalized to the initial lower yield stress, so that error due to specimen cross section measurement and uncertainty due to fluctuation of the tensile axis orientation could be removed. Thus, the average lower yield stress was determined to be 3.28 Kg/mm^2 at 24.5°C .

The flow stress increase $\Delta\tau'$ was a function of the electron dose as well as the aging treatment. In Fig. 24 the dosage dependence of $\Delta\tau'$ is demonstrated, where $\Delta\tau'$ is plotted against aging time at 24.5°C . In this figure it is clearly seen that the appearance of the $\Delta\tau'$ is dosage dependent although the value did not saturate within a convenient time period at this aging temperature. Upon aging at higher temperatures, saturation of $\Delta\tau'$ was observed for both the irradiated and unirradiated cases. $\Delta\tau'$ for a fixed electron dose of $5 \times 10^{17} \text{ e/cm}^2$, followed by aging at 24.5°C , 50°C , and 100°C , is plotted against the aging time in Fig. 25. It should be noted here that the saturation value for the irradiated specimen is much higher than that for the unirradiated one and that the saturation values appear to be independent of the aging temperature. An Arrhenius type analysis of Fig. 25 at half of the saturation value of $\Delta\tau'$ gave apparent activation energies of 0.60 eV for the irradiated and 0.46 eV for the unirradiated specimens.

A sharp yield drop was always observed after aging as seen in Fig. 23. The magnitude of yield drop was defined by $\Delta\tau = \tau_{\text{U}} - \tau_{\text{I}}$ which is plotted against the aging time in Fig. 26(a) and (b), for specimen C and specimen F, respectively. The filled marks in Fig. 26(a) indicate the data taken from the irradiated specimens and the open marks indicate those for unirradiated specimens. Here, no appreciable effect due to the irradiation is noted. When Fig. 26(b) is compared with (a), it is noted that the increasing rate

Fig. 24 Dosage dependence of the age hardening, $\Delta\tau'$. $\Delta\tau' (= \tau_I - \tau_F)$ is plotted against aging time. The tensile test was carried out at 24.5°C . The increase in $\Delta\tau'$ due to the irradiation is clearly dependent on the electron dose as well as on the aging time.

Fig. 25 Effect of aging temperature on the age hardening, $\Delta\tau' (= \tau_I - \tau_F)$. The amount of irradiation was $5 \times 10^{17} \text{e/cm}^2$ for all the irradiated cases (filled marks). The tensile test was carried out at 24.5°C . Two points must be noted here. First, the age hardening appeared faster at the higher temperatures. Second, the saturation value of $\Delta\tau'$ was independent of the aging temperatures.

Fig. 26 Comparison of the yield drop. $\Delta\tau$ ($= \tau_U - \tau_F$) is plotted against the aging time.

- (a) Specimen C: Filled marks for the irradiated, and open marks for the unirradiated. The effect of the irradiation appeared to be minor.
- (b) Specimen F: No irradiation. The appearance of $\Delta\tau$ is faster compared to (a).

of $\Delta\tau$ is much faster for Ferrovac-E iron, where the specimen F was not purified but simply annealed under vacuum.

So far, the results for the tensile testing at 24.5°C have been described. What follows is a description of the temperature dependence of the hardening, $\Delta\tau'$. The stress levels at different temperatures were determined from a series of stress-strain curves. The work-hardening term was corrected simply by taking the ratio between the stress just before and after the temperature change, and multiplying the stress before the temperature change by the ratio. After repeating several temperature cycles, the stress at the same temperature as for the initial test was again determined and these two stresses were found to agree within 0.1 Kg/mm². The results are given in Fig. 27, where the stress level for every specimen was normalized to the initial lower yield stress at 24.5°C. The filled marks indicate the results for the irradiated and fully aged specimens, and the results for unirradiated specimens are indicated by open marks. Within the temperature range studied, -40°C ~ 40°C, the increase in $\Delta\tau'$ due to electron irradiation and aging, appeared to be insensitive to the test temperature.

4. Effect of electron irradiation on micro-plastic deformation

In the microyield region, at low temperatures, the electron irradiation caused a hardening effect in a prestrained iron single crystal, while the macroscopic flow stress was decreased by the irradiation. The hardening effect increased with increasing the electron dose, as shown in Fig. 28 where the results obtained at 60°K for ZrH₂ treated specimen B are indicated. The Young's modulus determined from the linear portion of the stress-strain diagram was approximately 5×10^3 (Kg/mm²), which is

Fig. 27 Temperature dependence of the hardening due to the irradiation and post-irradiation aging. Normalized flow stress is plotted against test temperature. The hardening effect appeared to be insensitive to the test temperature.

Fig. 28 Electron irradiation hardening observed in the microyield region. The tensile test was carried out at 60°K. Note the significant hardening in the microstrain region. For a high dosage, 10^{18}e/cm^2 , the appearance of twinning is a characteristic feature in irradiated iron.

much smaller than the theoretically expected value of $1.4 \times 10^4 \text{ Kg/mm}^2$.^{*} This is due to the softness of the present machine.^{**} The elastic line drawn in the figure is not given by the experimentally determined slope but is given by the slope with $E = 10 \times 10^3 \text{ (Kg/mm}^2\text{)}$. The amount of plastic strain was obtained by measuring the deviation from the elastic line on the original chart. The prestrain was given at 24.5°C to a total strain of 3×10^{-2} , at a strain rate of 10^{-3} per second, and the test at 60°K was also carried out at the same strain rate. In a later stage of deformation, which is not shown in the figure, a decrease of the flow stress by the irradiation was again observed, although the measurement of the flow stress change was not as accurate as that of the macroscopic test due to the gradual yielding.

The hardening effect on the microyielding was larger at 4.2°K as shown in Fig. 29. The stress increase due to $5 \times 10^{17} \text{ e/cm}^2$ was 3 Kg/mm^2 at 60°K and was 8 Kg/mm^2 at 4.2°K . The stress was measured at 5×10^{-4} strain offset from the elastic line.

This hardening effect was also more pronounced by aging at a higher

* The Young's modulus for a cubic lattice is given by the relation $1/E = s_{11} - 2(s_{11} - s_{12} - \frac{1}{2}s_{44})(\alpha^2\beta^2 + \beta^2\gamma^2 + \gamma^2\alpha^2)$ where α , β , and γ are the directional cosines of the stress axis with respect to the cube axis. For the tensile axis of the specimen used in the present experiment $\alpha = 0.407$, $\beta = 0.156$, and $\gamma = 0.899$. Then, substituting the values for s_{11} , s_{12} , and s_{44} from Ref. 107, Young's modulus E is calculated to be $1.4 \times 10^4 \text{ Kg/mm}^2$.

** The main reason for this is considered due to the shape of the lower specimen grip (cf. Fig. 8). Since the load is supported by the bottom portions of the grip, elastic bending is expected to affect the measurement of the elastic strain significantly.

Fig. 29 Electron irradiation hardening at 4.2°K. The hardening due to the irradiation was significantly enhanced by lowering the test temperature. (cf. Fig. 28)

temperature, as shown in Fig. 30, where the stress-strain curve after an electron dose of 1.5×10^{17} e/cm is compared between as-irradiated and aged specimens. The aging was carried out in a cryostat at 150°K for 1 hour. The curve for an unirradiated specimen is given by a broken line in the figure.

An interpretation of these hardening effects will be given in a later section by taking account of the dislocation configuration introduced by prestraining at higher temperatures. The mechanism considered for the electron irradiation softening effect of macroscopic flow stress will also be examined in the light of these hardening effects in the microyielding.

Fig. 30 Effect of post-irradiation aging on the electron irradiation hardening. The irradiation hardening in the microyield region was significantly enhanced by the aging at 150°K.

IV. DISLOCATION MOTION IN A COMBINED STRESS FIELD OF PEIERLS AND MISFIT STRAIN CENTERS

In order to aid the examination of the experimental results given in the preceding chapter, a mathematical formulation of dislocation motion in the combined stress field of a Peierls potential and point obstacles is given in this chapter. The point obstacle is represented here by a misfit strain center. It will be shown that the strain center which would act as a strong obstacle to the dislocation motion in the absence of the Peierls potential, can aid the dislocation motion⁽⁹⁶⁾ through a relatively high Peierls potential under a certain condition. On the other hand, this strain center can hinder the dislocation motion under a different condition, as one usually expects. The present formulation may be an oversimplification of a real situation to give a quantitative description. However, it will be interesting to note that the present formulation can describe the softening and hardening effect which occurs in one system depending on experimental variables. By varying the obstacle concentration or temperature, both softening and hardening effects can be found in agreement with the present experimental observations as well as those found in many solid solutions.

1. Formulation of energetics

Let us consider that a screw dislocation lies straight, along a Peierls valley (along the x direction) in the absence of any internal or external stress. Since the precise function for the periodic nature of the crystal is not known, in the present calculation, the periodic nature is represented by a simple sinusoidal function,

$$\Gamma(y) = \frac{\Gamma_c + \Gamma_o}{2} + \frac{\Gamma_c - \Gamma_o}{2} \cos 2\pi y/b \quad (1)$$

where Γ_c and Γ_o represent the dislocation line energy at the top and bottom of the Peierls potential, respectively. Therefore the line energy of the screw dislocation varies sinusoidally with the periodicity b as the dislocation moves along the y axis. The periodicity along other crystallographic directions, such as the x direction, is neglected since it is generally accepted that the Peierls potential for the motion of an edge dislocation is much smaller than for a screw dislocation. The corresponding Peierls stress, τ_p , is given by the relation,

$$\tau_p b = - \left. \frac{\partial \Gamma}{\partial y} \right|_{\max} = \pi(\Gamma_c - \Gamma_o)/b \quad (2)$$

The effect of a perturbation on the simple sinusoidal function is small according to the results given by Dorn and Rajnak.⁽⁸⁸⁾ Moreover even a parabolic function gives similar results as shown in the work by Celli et al.⁽⁸⁷⁾ Thus, the choice of the form of a periodic function is not critical for the present calculation to examine the effect of point obstacles on the dislocation motion in a periodic potential field. Basic assumptions used in the present calculation are:

- (i) The material is elastically isotropic.
- (ii) The linear elasticity theory applies.
- (iii) The line energy of a dislocation does not depend on the Burgers vector.

The activation energy (E) required for the thermally activated motion of a dislocation can be evaluated by determining the dislocation energies at successive equilibrium (U_0) and saddle-point (U_s) configurations of the dislocation, during its motion over Peierls barriers and misfit strain

centers, and it is written as

$$E = U_S - U_0 \quad (3)$$

The excess energy of a dislocation segment ($\ell_1 \leq x \leq \ell_2$) on a slip plane (x-y plane) having any shape $y = y(x)$ is given by

$$U = \int_{\ell_1}^{\ell_2} [\Gamma(y) \sqrt{1 + y'^2} - \Gamma(y_0) - \int_{y_0}^y F(x, y) dy - \tau b(y - y_0)] dx \quad (4)$$

where the first and second terms involve the change in the total dislocation line energy due to the Peierls potential and a change in the shape of the dislocation, and the third and last terms are the work done by the internal and external stresses, respectively, during the dislocation motion from $y = y_0$ to $y = y(x)$. U_S and U_0 can be obtained numerically by Eq. (4) after determining the saddle-point and equilibrium configurations of the dislocation by solving Euler's condition, $\delta U = 0$, which is identical to

$$\frac{\partial^2 u}{\partial y^2} \frac{d^2 y}{dx^2} + \frac{\partial^2 u}{\partial y \partial y'} \frac{dy}{dx} + \frac{\partial^2 u}{\partial x \partial y'} - \frac{\partial u}{\partial y} = 0 \quad (5)$$

where u represents the integrand in Eq. (4). Substituting for u from Eq. (4), Eq. (5) can then be written as

$$\Gamma(y)(1 + y'^2)^{-3/2} \frac{d^2 y}{dx^2} - (1 + y'^2)^{-1/2} \frac{\partial \Gamma}{\partial y} + F(s, y) - \tau b = 0 \quad (6)$$

Therefore a dislocation configuration, corresponding to the equilibrium or the saddle-point can be obtained by finding a solution for Eq. (6) with proper boundary conditions. The solution for Eq. (6) was determined with the aid of a computer (CDC 6400 computer at the Vogelback Computing Center, Northwestern University). The method of computation will be presented in the next section. The energy U_S or U_0 corresponding to each solution of

the equation was evaluated by the integration in Eq. (4).

In most of the present calculations the local internal stress was represented by the stress field around a spherical misfit strain center. The force exerted by the strain centers on the dislocation at (x,y) is given by

$$F(x,y) = \sum_i 6\mu\epsilon b r_0^3 z_i (x - x_i) \{(x - x_i)^2 + (y - y_i)^2 + z_i^2\}^{-5/2} \quad (7)$$

where (x_i, y_i, z_i) is the position of the misfit strain center; μ is the shear modulus, b is the Burgers vector, r_0 is the radius of the misfit strain center, and ϵ is the strength of the misfit strain.

For a tetragonal misfit strain center, the formula for $F(x,y)$ must be slightly modified from that of a spherical strain center. The detail of such a modification will be presented in a later section (p. 96).

2. Method of computation and preliminary results without misfit defects

The main part of the computation in the present study was to find a solution for Eq. (6) with a set of logical boundary conditions for the dislocation motion. Once the solution is found, one can determine the energy required to move the dislocation from the reference position $(y = y_0)$ to the new position corresponding to the solution of Eq. (6), by using Eq. (4). If the term in Eq. (6), $F(x,y)$ is zero corresponding to the no-misfit case, then Eq. (6) can be integrated to the expression which involves only a first derivative term of dy/dx , as it was done by the earlier workers. (87,88,92) However, because of the introduction of the term $F(x,y)$ which is the essence of the present study, Eq. (6) had to be solved directly by a numerical method. One of the possible methods is to find the solution by using an iteration method, which was first applied

by Urakami for solving Eq. (6). In the present calculation a similar method proposed by him was used. The method is described briefly as follows: Let us set the boundary conditions as $y = y_A$ at $x = l_1$ and $x = l_2$, where y_A is the position of force balance between the applied stress and the Peierls force. By choosing an arbitrary slope, y' , at (l_1, y_A) and a small increment Δx , slope y' at $x = x_1 + \Delta x$ can be computed by using Eq. (6). y at $x + \Delta x$ is then given by $y = y_A + y' \cdot \Delta x$. This process can be repeated by using the successive new sets of values of x , y and y' . The value of y at $x = l_2$ is then finally given. Changing the initial slope y' at $x = l_1$ so that y approaches y_A at $x = l_2$, a solution for Eq. (6), which satisfies the boundary conditions, $y = y_A$ at $x = l_1$ and $x = l_2$, can be determined.

Some of the preliminary results which were obtained by the above-mentioned method without the misfit strain centers are presented here. The values of y' varied depending on the choice of the dislocation segment length ($l = l_2 - l_1$). For example, the values were approximately 0.2, 0.01 and 0.0001 for the lengths of 50b, 100b and 150b, respectively. The excess dislocation energy was then computed by Eq. (4) for the solutions obtained above. By increasing the segment length from 20b to 200b, it was found that the activation energy became insensitive to the choice of a segment length beyond a critical length. The critical length changed slightly with the magnitude of the external stress, as shown in Fig. 31(a), where the activation energy is plotted against the dislocation segment length ($l = l_1 - l_2$) for different stresses. The line energy used here was $\Gamma_0 = \mu b^2/2$ with $\mu = 7.73 \times 10^3 \text{ Kg/mm}^2$. This result is consistent with the earlier results achieved by using the analytical method for solving the Euler's condition. (92,93)

In the same manner as for Fig. 31(a), the activation energy was

Fig. 31 Activation energy for the double kink nucleation in the absence of misfit strain centers. The dislocation length and applied stress dependence of the energy is compared for the different dislocation line energies in (a) and (b).

evaluated by using a different value of Γ_0 and the result is shown in Fig. 31(b). Here it is noted that by increasing the line energy to $\Gamma_0 = \mu b^2/0.4$ the above-mentioned critical length increased from $\ell = 30b$ to $\ell = 50b$. However, the general shape of the $E - \tau/\tau_p$ relation was almost identical for these two cases, indicating that the choice of Γ_0 is less important for the objective of the present study where the relative strength of the misfit strain center to that of the Peierls potential is the more important factor for our purpose in the study. For all the following calculations $\Gamma_0 = 2.3 \times 10^{-4}$ ergs/cm² and $\tau_p = 60$ Kg/mm² were used, while the strength of the misfit strain center was varied.

3. Introduction of a misfit strain center and choice of the parameters

The force exerted by a misfit strain center on a screw dislocation depends on the parameters such as the misfit strain, ϵ , the misfit volume, $4\pi r_0^3$, and the location of the strain center (x_i, y_i, z_i) as seen in Eq. (7). The effect of the location of the misfit strain center was studied by changing the distance, z_i , holding the other parameters constant. The equilibrium dislocation configurations in the vicinity of a strain center with $\epsilon = 0.15$ and $r_0 = 2b$ were determined by the aforementioned method and the results are shown in Fig. 32, where the center is located on z-axis at various distances from the slip plane (x-y plane). The dislocation energies corresponding to the equilibrium states are also indicated on the curves. It should be noted here that the effect of the misfit strain center depends strongly on the distance z . The effect of changing the misfit strength has a more or less similar effect as studied by Urakami.⁽⁹⁶⁾ For the calculations presented in the following sections the parameter z will always be assumed to be $5b$, and the misfit strength will be changed.

Fig. 32 The equilibrium configuration of a screw dislocation in the presence of a misfit strain center. A misfit strain center is placed at different positions along normal to the paper (x-y plane). The equilibrium energy is given on each curve. It should be noted that the distance scale on the ordinate of the figure is magnified in order to illustrate the shape of the dislocation.

The exact description would require a statistical analysis of the distribution of misfit strain centers and their cumulative effect, but such a treatment is not attempted in the present study.

4. Mode of double kink nucleation in the vicinity of misfit strain centers

As seen in Fig. 32, the equilibrium configuration in the vicinity of a misfit strain center has lower energy compared to the equilibrium dislocation along the x-axis (at Peierls potential valley, $y = y_0$) in the absence of the misfit strain center. Suppose the forward dislocation motion under applied stress is directed towards the positive y direction in Fig. 32, one will naturally expect that the motion must be assisted at the right hand side of the dislocation with respect to the misfit strain center, and that on the other side the motion is hindered. Thus, because of the introduction of a misfit strain center, double kink nucleation is assisted at one side of the strain center and resisted at the other side.

Let us consider the double kink nucleation in the vicinity of two misfit strain centers. Whether the double kink nucleation is assisted or resisted in between the two strain centers, is determined by a combination of the sign of the Burgers vector, the type of the misfit strain (compression or tension) and the location of the strain center (above or below the slip plane). Once the sign of the Burgers vector and the type of the misfit strain are assigned, one will find four combinations for placing the two misfit strain centers with respect to the slip plane. Among those four combinations, the cases where both the strain centers are placed on the same side with respect to the slip plane, are physically identical.

Therefore only the three combinations are considered here. The sign of the Burgers vectors will be taken along the positive x direction, and the misfit strain ϵ will also be taken as positive.

Representative pictures for those three types of nucleation are shown in Fig. 33, indicating that the double kink nucleation is assisted or resisted by the local strain field around the two misfit strain centers. The solid and broken lines correspond to the equilibrium and saddle-point configurations, respectively. Fig. 33(a) is the case where the two strain centers are placed on the same side, below the slip plane. It is clearly seen that the double kink nucleation occurs at the energetically favored side of the strain centers. The required energy for the double kink nucleation was $0.0707 (\Gamma_0)$ as indicated on the broken line (corresponding to the saddle-point configuration) in the figure. It should be noted that the energy decrease due to the introduction of the misfit strain centers can be found by comparing this value to that for $(\tau/\tau_p = 0.1$ and $l = 90b)$ given in Fig. 31(b), where the energy was computed with the same constants as used here. The spacing between two misfit strain centers was taken to be $50b$, and they were located at $5b$ from the slip plane. The boundaries of the dislocation segment were taken at (l_1, y_A) and (l_2, y_A) where l_1 and l_2 were chosen so that y' at l_1 and l_2 became nearly zero to avoid an influence of the boundary condition.

Fig. 33(b) is the case where the left hand side and right hand side strain centers are placed below and above the slip plane, respectively. In this case both the strain centers contribute to assist the double kink nucleation and the nucleation energy is naturally lower than case (a). It is further noted that there are two kinds of nucleation sites, A and B in this case, and that site A has a lower nucleation energy than site B,

Fig. 33(a) Double kink nucleation in the vicinity of misfit strain centers. The positions of the strain centers are indicated by filled circles. They are placed at the same side with respect to the slip plane (below the plane). The double kink nucleation is assisted at the local strain field favored for the nucleation.

Fig. 33(b) The double kink nucleation in the vicinity of the misfit strain centers. Both of the strain centers are placed at the favorable positions for the double kink nucleation between the two strain centers. Two types of double kink nucleation sites were found in this case (A and B).

Fig. 33(c) The double kink nucleation in the vicinity of the misfit strain centers. The two centers are placed at the unfavorable positions. The nucleation energy is naturally higher than the other cases. [cf. Fig. 33(a) and (b)].

as noted in the figure. When the two misfit strain centers in case (b) were reversed, the double kink nucleation was found far away from both the misfit strain centers as shown in Fig. 33(c). The required energy for the nucleation was found to be slightly higher than the case where no strain centers were considered [cf. Fig. 31(b)]. The following calculations will be primarily concerned with the nucleation configuration corresponding to the lowest energy state such as the one at site A in Fig. 33(b).

5. Appearance of the resistance against sideways motion of kinks and determination of a rate controlling process

The discussion given in the preceding section concerned a relatively weak misfit and propagation of a kink after its formation was nearly spontaneous. For a stronger misfit strain center, however, resistance to the propagation of a kink (sideways motion of a kink) increases while double kink nucleation becomes easier. Thus, successive nucleation (second, third, and fourth . . .) can occur, instead of a nearly spontaneous sideways motion of kinks. Such successive nucleations are shown in Fig. 34(a), where the boundary conditions and constants used here were the same as for Fig. 33, except that the misfit strain was increased from 0.05 to 0.25. The activation energy is smallest for the first nucleation, and it increases for the second and third nucleations, because the couple force aiding the double kink formation of the dislocation decreases with increasing the distance between the misfit strain center and the dislocation. In this case, naturally, one must consider the second kind of saddle-point corresponding to the sideways motion of a kink. The dislocation configurations at the equilibrium and saddle-point positions for this type of

Fig. 34 (a) Successive double kink nucleation with the assistance of the misfit strain centers. The periodic nature of the dislocation line energy is represented by a curve given in the right hand side of the figure.

(b) The presence of the strong defect causes a dragging force for the motion of the kinks. The saddle point configurations for the sideways motion of a kink is demonstrated.

motion are shown in Fig. 34(b), where all constants used for this calculation were the same as for Fig. 34(a).

For the calculation of the sideways motion of kinks the dislocation boundaries were slightly modified: the left hand side boundary was extended to a larger distance from the strain center so that the end effect at $x = \ell_1$ did not appear, and at the right hand boundary, y_A was taken at a different potential valley corresponding to the succession of double kink nucleation. It was found that the energy was the highest after the first nucleation and decreased with the successive double kink nucleations. Thus, the energy required to overcome a misfit strain center had to be determined by examining the energetics of these two types of dislocation motions.

An example of the determination of the activation energy required for overcoming the strain center is illustrated in Fig. 35, where the misfit strain used was 0.35. The activation energies for double kink nucleation and sideways motions are plotted against the normalized external stress, τ/τ_p . The variation of energy of the dislocation configuration during the double kink nucleation process is also plotted against the propagation distance of the top of the double kink along the y-axis, at the right side corner in the same figure. As shown in this small figure, the total dislocation energy at equilibrium position was lowered by the successive double kink nucleation to the limit of the bow-out distance, which is determined by the balance between the line energy increase and the work done by the applied stress. The limitation in the bow-out distance naturally changed with the external stress. At $\tau/\tau_p = 0.1$ the above limitation occurred after the fourth double kink nucleation as indicated by a star mark in the figure. For the higher stresses this limitation

Fig. 35 An example of the determination of the rate controlling process. The energies required for the successive double kink nucleations are plotted by open circles and those for the sideways motions are given by cross marks. The small figure shows the variation of dislocation energy by the successive double kink nucleations. The * mark indicated on the fourth nucleation implies this nucleation process is restricted by the increased line energy. For the detail of the discussion on determining the rate controlling process, see the text.

occurs for the more advanced stages of the double kink nucleation than the fourth nucleation.

Having the above-mentioned nature in mind, let us consider the overcoming process of the strain center. As far as the dislocation energy decreases with the successive double kink nucleation and the activation energy required for the sideways motion of the kink is reasonably larger than that of the next double kink nucleation, the probability of the occurrence of the sideways motion of the kink is negligibly small* compared to that of the next double kink nucleation. Therefore, the activation energy for the overcoming process can be defined in the following manner: For example, at $\tau/\tau_p = 0.3$, the occurrence of the third sideways motion is negligibly small compared to the fourth nucleation and the fourth nucleation must occur before the third sideways motion takes place. After the fourth nucleation the fourth sideways motion becomes easier than the fifth nucleation. Therefore, the misfit strain center must be overcome with the activation energy corresponding to the fourth nucleation. From a similar consideration the third sideways motion must be taken as responsible for the overcoming of a strain center at the stress $\tau/\tau_p = 0.5$. However, for a lower applied stress the situation is slightly different: although the fourth nucleation is energetically easier than the third sideways motion at $\tau/\tau_p = 0.1$, the third sideways motion must be considered as the responsible process. This is because the equilibrium energy after the fourth

* Even with the energy difference of 0.001 (Γ_0), the probability of the occurrence of the lower energy event was 10 times larger than that of the higher energy.

nucleation is higher than that after the third nucleation as explained in the description of the small figure in Fig. 35. Thus, the backwards motion is more predominant than the fourth nucleation and hence the dislocation stays mostly in the third equilibrium position until the third sideways motion occurs.

In such a situation it is possible that in the neighboring segment of the dislocation, another double kink can be formed with a smaller energy than the third sideways motion, and that the new kink will spread out to join with one of the prior double kinks. Then the dislocation becomes able to overcome the misfit strain center with a different mode as shown in Fig. 36. In this case, the activation energy is much smaller than that for the sideways motion, (cf. Fig. 35). Therefore, in such a case the misfit strain center will be overcome by a completely different mode from those considered in Fig. 35, and the sideways motion obtained in Fig. 35 is not required to overcome the strain center. However this kind of dislocation motion will not significantly contribute to the total energetics of a dislocation motion, since along the total dislocation line there will be a site where double kink nucleation is resisted between two adjacent strain centers, as in case (c) in Fig. 33. Therefore the dislocation will be hindered at such a site until the sideways motion will occur.

From the above-mentioned considerations, a combination of the nucleation of type A in Fig. 33(b) and the boundary conditions for the sideways motion given in Fig. 34(b) is considered to be a reasonable choice for determining the rate controlling process.

Fig. 36 Overcoming of the strain center by kink collision. The left side kink were assumed to have moved from the other double kink nucleation site. The energy required for this type of process is lower compared to the sideways motion (cf. Fig. 35).

6. The activation energy vs τ/τ_p profiles

a) Effect of misfit strength

In the same manner as described in the previous section, the activation energies corresponding to the rate controlling processes were determined at various applied stress levels for different misfit strengths. The results are summarized in Fig. 37, where the activation energy, E , for the double kink nucleation or for the sideways motion, whichever controls motion, is plotted against the normalized external stress τ/τ_p . The spacing between the two misfit strain centers was taken to be $50b$ for all calculations. The line corresponding to zero misfit strain represents double kink nucleation without the misfit strain centers. A marked decrease in the energy should be noted as the misfit strain centers are introduced. The largest reduction in energy was achieved by a relatively weak strain center. As the strength of the misfit strain center increases beyond a critical value (corresponding to the curve for $\epsilon = 0.15$ in Fig. 37), the energy starts to increase. A considerably larger energy is required for the dislocation to overcome the misfit strain center in the case of $\epsilon = 0.5$ than that in the case without the misfit strain centers, particularly at low stress levels.

b) Effect of the separation between two strain centers

Concentration dependence is an important characteristic in the experimental study of the interaction between dispersed misfit strain centers and dislocations. Although a precise description cannot be given without a statistical analysis of a random distribution of the strain centers, semi-quantitative information can be obtained concerning the energetics of the overcoming process by simply changing the spacing between the two strain centers in the present model.

Fig. 37 Energy, E , required for dislocation motion in a combined Peierls and misfit field. $l = 50b$ is the spacing between the two strain centers. $\Gamma_0 (\approx \Gamma_c)$ is the dislocation self energy. Numbers on the curves indicate the strength of misfit strain centers. A marked decrease in the energy should be noted as the misfit strain centers were introduced. For a stronger defect, however, the energy increased beyond 0 misfit case at lower stresses.

The same type of calculations as in the previous sections were carried out for the different spacings between the two strain centers. The results for the two different lengths, $\ell = 50b$ and $90b$, are compared in Fig. 38, where all the constants used for this calculation were the same as the case for Fig. 37. It should be noted from this figure that the higher energy for a dislocation motion is required for the smaller spacing, especially at lower stress levels, while the energy does not change for the higher stresses. This energy increase is essentially coming from the limitation in the bow-out distance. As we have seen in the small figure in Fig. 35, the successive double kink nucleation is limited within the bow-out distance. This limit was found to change with the defect spacing, namely, the limiting distance is smaller for the smaller spacing. Therefore in the case of smaller spacing the sideways motion must operate at the earlier stage of the multiple double kink formation, thus resulting in higher energy. At higher stress levels, however, the sideways motion can occur within the limitation of the bow-out distance because of the increase in the limit.

To visualize the limitation in the bow-out distance, a simple calculation was carried out by assuming perfect pinning at the ends of the dislocation segment and also by neglecting the effect of the Peierls potential. For calculating the energy change due to the bow-out, the shape of the bow-out dislocation was always assumed as a part of a circumference. The energy change due to bow-out was then given by

$$E = 2\Gamma_0(R\theta - L) - \tau bA \quad (8)$$

where the first term represents the increase in the line energy and the second term represents the work done by the applied stress. The parameters

Fig. 38 The energy, E , is dependent on the spacing between the two strain centers. The cases for $l = 50b$ and $l = 90b$ are compared. The fact that higher energy is required for the smaller spacing, especially at lower stresses, should be noted.

R, θ , and A were given by the bow-out distance, Y, as follows:

$$R = (Y^2 + L^2)/2Y \quad (9-a)$$

$$\theta = \cos^{-1} (Y - R)/R \quad (9-b)$$

$$A = R^2 + (Y - R)L \quad (9-c)$$

where L is the dislocation segment length. The energy E was computed by using $L = 200b$ and $\Gamma_0 = 2.3 \times 10^{-4}$ erg/cm which is the same as the one used for the computation in the preceding sections. The result is shown in Fig. 39, where the variation of the dislocation energy is plotted against the bow-out distance Y. The limitation of the bow-out occurs for $\partial E/\partial Y > 0$ where the further bow-out process causes increase of the dislocation energy. It should be noted here that the limiting distance corresponding to $\partial E/\partial Y = 0$, increases with the applied stress, and that only beyond a critical stress $\tau = \tau_F$ corresponding to $Y = L/2$, the dislocation segment can bow out contributing to the macroscopic deformation. This stress level is extremely high even with this large dislocation segment length ($L = 200b$), as clearly demonstrated in the figure. Therefore the assumption of the perfect pinning made by Ono and Sommer⁽⁹³⁾ appears to encounter the difficulty for explaining the magnitude of the athermal hardening given by them. As we have seen in the present calculation, this difficulty was completely overcome by introducing a finite interaction between a point defect (a misfit strain center) and a dislocation. Furthermore, the general trends in the experimental observations are well understood from the present calculation as it will be discussed in the Discussion chapter (p. 102).

Fig. 39 Relation between bowing-out distance and the dislocation energy, calculated from a simplified expression for the dislocation energy. Dislocation segment length is taken to be $200b$. Note that the bowing-out distance corresponding to $\partial E/\partial Y = 0$ increases with the increase of the applied stress. It should also be noted that the stress required for moving the dislocation segment ($\tau = \tau_F$) is more than half of the Peierls stress ($\tau_p = 60$ Kg/mm²).

7. Activation volume

The area surrounded by a pair of dislocation segments corresponding to saddle-point and equilibrium configurations was also computed for every activation event at the time when the activation energy was determined. The activation volume was then given by the product of the Burgers vector, b , and the computed area. The activation volumes thus determined for the double kink nucleation and for the sideways motion are plotted against the normalized stress in Fig. 40, by solid lines for the nucleation and by broken lines for the sideways motion.

As seen in this figure the calculated activation volume for double kink nucleation was always smaller in the presence of misfit strain centers than in the perfect lattice, while the activation volume for the sideways motion was much larger than that for the nucleation. This is seen especially at the lower stress levels, and with moderate strength of the strain centers.

The alternative way for defining the activation volume is to approximate it by taking the quantity, $-\partial E/\partial \tau^*$. From E vs. τ/τ_p plot given in Fig. 37, this quantity must be evaluated in principle. However, as the determination of the activation energy E was made with relatively large stress intervals (cf. Fig. 35), the determination of the slope for the E vs. τ/τ_p , which is equivalent to evaluating the quantity, $-\partial E/\partial \tau^*$, could not be made with reliable accuracy. Therefore we cannot compare the activation volumes which can be defined in two different ways. However, in the zero misfit case, the activation volume determined here by taking the product between the swept area and the Burgers vector showed good agreement with the results given by Dorn and Rajnak⁽⁸⁸⁾ where they used the approximation, $v^* = -\partial E/\partial \tau^*$. This agreement would substantiate the application of the present results to the case of $-\partial E/\partial \tau^*$ expression, which can be related

Fig. 40 Activation volume vs. τ/τ_p plot. The volume was determined by taking the product of the area swept by the thermal activation event and the Burgers vector. Broken lines are for the sideways motion and solid lines for the double kink nucleation. Note the larger activation volume for the sideways motion, especially at lower stress levels.

to an experimental observation through a rate theory.

An important finding in Fig. 40 is that the stronger strain center gives a larger activation volume at a low stress level where the sideways motion is the predominant rate controlling process. On the other hand, at higher stress levels or for the weaker strain centers, the double kink nucleation is the rate controlling process and hence the activation volume appears to be relatively small.

8. Effect of tetragonal misfit strain centers

The effect of the tetragonal misfit strain center is studied in this section. A tetragonality is assumed along $[x,0,0]$, $[0,y,0]$ or $[0,0,z]$ direction where x , y and z are the dislocation coordinates. Displacement around a strain center of an arbitrary shape is expressed by using Eshelby's expression, (142)

$$U_i^C(\bar{X}) = V \epsilon_{jk}^T g_{ijk} / 8\pi(1 - \nu)r^2 \quad (10)$$

where U_i^C is the displacement, V is the volume of a misfit strain center, ϵ_{jk}^T is the stress free strain, r is the distance from the strain center, and $\bar{X}$ is the point of observation. g_{ijk} is given by a relation,

$$g_{ijk} = (1 - 2\nu)(\delta_{ij}l_k + \delta_{ik}l_j - \delta_{jk}l_i) + 3l_i l_j l_k \quad (11)$$

where ν is the Poisson's ratio, $\bar{l}(l_1, l_2, l_3)$ is the direction cosine and δ_{ij} is the Kronecker delta. After finding the formula for the displacement due to the tetragonal strain centers defined above, the stress function was derived by the general transformation from the displacement function to a stress function. The three types of stress functions are then written as follows:

i) for ϵ_{xx}^T type of strain centers (tetragonality along $[x, 0, 0]$)

$$\sigma_{zx} = \sum_i K \epsilon_{xx}^T (x-x_i) z_i \{ (x-x_i)^2 + (y-y_i)^2 + z_i^2 - 5(x-x_i)^2 \} \{ (x-x_i)^2 + (y-y_i)^2 + z_i^2 \}^{-7/2} \quad (12-a)$$

ii) for ϵ_{yy}^T

$$\sigma_{zx} = \sum_i K \epsilon_{yy}^T (x-x_i) z_i \{ (x-x_i)^2 + (y-y_i)^2 + z_i^2 - 5(y-y_i)^2 \} \{ (x-x_i)^2 + (y-y_i)^2 + z_i^2 \}^{-7/2} \quad (12-b)$$

iii) for ϵ_{zz}^T

$$\sigma_{zx} = \sum_i K \epsilon_{zz}^T (x-x_i) z_i \{ (x-x_i)^2 + (y-y_i)^2 + z_i^2 - 5z_i^2 \} \{ (x-x_i)^2 + (y-y_i)^2 + z_i^2 \}^{-7/2} \quad (12-c)$$

where K is equal to $6V\mu/8\pi(1-\nu)$ and (x_i, y_i, z_i) is the location of a misfit strain center. The details of the derivation of these stress functions are given in Appendix B. The forces exerted on a dislocation by the strain centers are then given by $F^T(x, y) = \sigma_{zx} b$.

The interaction between a screw dislocation and a misfit strain center is compared for the three types of tetragonal misfit strain centers and a spherical misfit strain center in Fig. 41. The misfit strength is given by the stress free strains*, $\epsilon_{xx}^T = \epsilon_{yy}^T = \epsilon_{zz}^T = 0.40$ and $\epsilon^S = 0.34$. $\epsilon^S = 0.34$ used here is identical to $\epsilon = 0.25$ used in the previous sections, where the strain was defined as it was relaxed in the matrix. The general shapes of the equilibrium configurations are rather similar, although their sizes are different. A similar effect, therefore, can be simulated by changing the strength of a misfit strain center.

* "Stress free strain" is the transformation strain which the misfit strain center would undergo in the absence of the matrix material.

Fig. 41. The equilibrium shape of a screw dislocation in the presence of a tetragonal misfit strain center. Three types are compared to the spherical case (broken line). The interaction energy and the detail of the dislocation shapes are not identical to one another, but the general nature is similar.

For these three types of tetragonal misfit strain centers, the same type of calculation as for the spherical misfit strain centers was carried out, and the result is shown in Fig. 42. Here the misfit strength of $\epsilon_{xx}^T = \epsilon_{yy}^T = \epsilon_{zz}^T = 1.2$ is used for the three cases. For ϵ_{xx}^T misfit strain centers at lower stresses the energy was higher than the case with ϵ_{zz}^T defect as seen in the figure. The nature of this crossover at lower stress levels is similar to the case of spherical misfit strain centers.

For the different strengths of the strain centers, the overall nature of the increase or decrease of the activation energy due to the tetragonal defects was similar to that in the spherical case (cf. Fig. 37) for the two-dimensional motion of a dislocation. An example of the strength dependence is shown for the ϵ_{yy}^T type of strain centers in Fig. 43. This figure appears to be quite similar to Fig. 37. Especially for the $\epsilon_{yy}^T = 1.2$ case, the E vs. τ/τ_p relation coincides with the spherical case of $\epsilon = 0.25$ in Fig. 37.

These results clearly indicate that the tetragonality is not the necessary condition for having a strong interaction with a screw dislocation. However, it will be interesting to extend the present calculation to the three-dimensional motion of the screw dislocations in the presence of the tetragonal defect.

Fig. 42 The energy required for overcoming the tetragonal defect. The direction of the tetragonality is indicated on each curve. It is found that the ϵ_{xx}^T type of tetragonality is most effective. Note that the general trend is the same as for the spherical defect case.

Fig. 43 The energy vs. τ/τ_p plot for the different strength of defects. The tetragonality is assumed to be along the y-axis. The curve for $\epsilon_{yy}^T = 1.2$ coincides with the case of $\epsilon = 0.25$ for the spherical defects.

V. DISCUSSION

1. On the plastic behavior of ultra-pure iron single crystals

As demonstrated in Figs. 10 and 12, the iron single crystals (both Battelle iron and Ferrovac-E iron) oriented for the $\langle 111 \rangle, \{110\}$ type slip system and purified in the ZrH_2 furnace under vibrational stress, exhibited significant plasticity down to 4.2°K with a relatively small work hardening rate, $(\Delta\tau/\Delta\gamma \approx 25 \text{ Kg/mm}^2)$. As discussed in the earlier work by Stein et al.,⁽¹⁾ a thermodynamic analysis on the ZrH_2 treatment is supposed to give a very low concentration of non-metallic impurities. This analysis is reviewed in Appendix A. In their study, single crystals grown from Ferrovac-E iron were used and the stress-strain curve after the ZrH_2 treatment was similar to that of the specimens prepared in the present study without vibrational stress (cf. Fig. 10). The vibrational stress applied during the present purification treatment clearly improved the ductility of the same specimen. The enhancement of the purification by applying a vibrational stress may be explained as follows: the break-away of interstitial impurity atoms from Cottrell atmospheres can be assisted by the applied stress, because the binding energy between an edge dislocation and an interstitial impurity is around 0.5 eV⁽¹⁰⁸⁾ which should give a so-called condensation temperature of 6400°K.

On the other hand, the reduction in flow stress cannot be directly attributed to the reduced interstitial content, since in less pure and unprestrained specimens the plastic deformation tends to be more local^(13,16) and hence the effective strain rate may increase. When high strain rate sensitivity of iron is taken into account, the increase in the strain rate can cause the increase in the stress. Moreover, the shape of the stress-

strain curve changes with the appearance of a yield drop and twinning. This makes it difficult to determine the stress levels corresponding to the same dislocation structures. Therefore, a direct comparison of the stress levels at low temperatures among specimens of different purities may not necessarily provide conclusive results to the role of interstitial atoms.

The temperature dependence of the yield stress of the ZrH_2 purified Battelle iron is shown in Fig. 11, where no prestrain was given prior to testing at each temperature except the testing at 4.2°K . In the case of the 4.2°K test, the specimen did not exhibit any ductility without prestraining and we could not determine the stress level of the unprestrained specimen. However, after giving 3% prestrain at 60°K the specimen showed significant ductility. The stress level determined in such a test is given in the figure at 4.2°K . In this case the prestrain temperature was low and the deformation in the microyield region was small enough to determine the flow stress level unambiguously. The temperature dependence of the flow stress is a smoothly decreasing function with temperature from 4.2°K to 350°K as seen in the figure. The stresses for vacuum annealed specimen F are also given in the same figure. The flow stress of unprestrained specimen F could be determined only in the higher temperature region because this specimen showed twinning or brittle fracture at lower temperatures. However, it should be noted that within the temperature range studied here, the present results where no prestrain was given reproduced the behavior observed in a prestrained specimen by Nakada and Keh.⁽¹³⁾

The strain rate sensitivity was also determined as shown in Fig. 13. As described in the previous section, the temperature corresponding to a strain rate sensitivity maximum shifted to a higher temperature for a

purier specimen. As noted from the results of the earlier work,⁽¹⁴⁾ this can be taken as an indication of improved specimen purity, due to the present purification treatment.

It was also noted that the strain rate sensitivity at low temperatures was decreased by the prestraining at higher temperature as shown in Fig. 13. This decrease must be closely related to the difference in the dislocation structures. The dislocations introduced by the prestraining at higher temperatures have often been observed to be tangled, while the specimens deformed at lower temperatures contain mostly straight dislocations along $\langle 111 \rangle$ direction. This feature has been reported in the electron microscopy study by several authors.⁽⁵¹⁻⁵³⁾ Thus the decrease of strain rate sensitivity in the initial portion of the deformation due to prestraining is explained by taking account of the difference in the dislocation structures. The increase in the number of edge dislocations may directly affect the strain rate sensitivity, because the activation volume for the motion of an edge component through a misfit defect was calculated to be larger than that for the double kink nucleation of a screw dislocation, as shown in Fig. 40. Thus the low strain rate sensitivity for the prestrained specimen can be explained by the reciprocal relation between an activation volume and a strain sensitivity.

From the strain rate sensitivity data and the temperature dependence of the yield stress one can calculate the activation energy and the activation volume by assuming a single thermally activated deformation process. In a rate equation representing the thermally activated deformation,

$$\dot{\gamma} = \dot{\gamma}_0 \exp(-\Delta G/kT) \quad (1)$$

if the pre-exponential term, $\dot{\gamma}_0$, is assumed to be constant and the temperature dependence of entropy and of internal stress is neglected, the

activation energy, E , and the activation volume, v^* , can be given by the following equations, (109-111)

$$E = -kT^2 (\partial \tau^* / \partial T)_{\dot{\gamma}} / (\partial \tau^* / \partial \ln \dot{\gamma})_T \quad (2)$$

$$v^* = -(\partial E / \partial \tau^*) = kT / (\partial \tau^* / \partial \ln \dot{\gamma})_T \quad (3)$$

where $\tau^* = \tau - \tau_\mu$ is the effective stress. Figs. 44 and 45 are the results of the calculations of the activation energy and activation volume, respectively. In the present calculation the effective stress τ^* was taken by simply subtracting the value of the stress at 70°C from the stress at each test temperature. As seen in the figures, both the activation energy and volume appeared to be insensitive to the impurities and to the dislocation structures, especially at higher stresses. However, this result does not necessarily mean that the interstitial impurities are unimportant for the thermally activated deformation. At lower stresses both of the parameters are larger for less pure specimens. This fact implies that thermally activated deformation must be related to the presence of impurities, at higher temperatures.

The interpretation of the athermal increase in the flow stress has been given in terms of Mott and Nabarro's theory^(82,83) by Nakada and Keh.⁽¹³⁾ In their interpretation, however, the transient from Peierls mechanism to the athermal hardening mechanism due to the dispersed obstacles cannot be clearly explained. If the present local double kink nucleation model is applied, the appearance of the transient can be well understood, as seen in Fig. 37, in the case of $\epsilon = 0.35$. A simple explanation for the transient can be seen in Fig. 35, where the activation energies for the nucleation and the sideways motion are compared. The stress dependence of the activation energy for the sideways motion is

Fig. 44 Activation energy calculated from the strain rate cycling experiment at different temperatures (cf. Figs. 11 and 12). The energy determined is insensitive to the specimen purity. At lower stresses, however, the less pure specimen seems to show higher energy.

Fig. 45 The activation volume calculated from Fig. 12. The volume is again found to be insensitive to the specimen purity.

larger than that for the nucleation, especially at lower stress levels; namely, the temperature dependence of the flow stress for the sideways motion is smaller than that for the double kink nucleation. Therefore the temperature insensitive hardening at higher temperature regions can be explained by the interaction between non-screw type dislocations and the interstitials. However, the results of the calculation given in the preceding chapter do not directly indicate the experimental parameters. Conversion from E vs. τ/τ_p relation to τ/τ_p vs. T relation will help to find out the correlation between the experimental results and the results of the calculation. This will be discussed in a later section (p. 112).

2. Concentration of irradiation-induced defects and their recovery

a) A theoretical estimate of the defect production rate

In the relatively low concentration range, the defect production rate is generally observed to be proportional to the electron dosage rate.⁽¹¹²⁾ The defect concentration is then given by $C = \sigma_d \phi$, where σ_d is the displacement cross section and ϕ is the electron dose.

The displacement cross section is generally given by an integration⁽¹¹³⁾

$$\sigma_d = \int_{T_d}^{T_m} P(T) d\sigma_d(T) \quad (1)$$

where $P(T)$ is a threshold probability function, $d\sigma_d(T)$ is a differential displacement cross section, T_m is the maximum recoil energy which can be transmitted to an atom, and T_d is the threshold energy. The differential cross section $d\sigma_d$ in the McKinley and Feshbach⁽¹¹⁴⁾ approximation is given by

$$d\sigma_d = (0.25 \text{ barns}) z^2 (1 - \beta^2 / \beta^2) (T_m / T^2 [1 - \beta^2 (T / T_m) + \alpha \beta \{ (T / T_m)^{1/2} - (T / T_m) \}]) dT \quad (2)$$

where the meaning of the symbols are as follows:

z = atomic number of the target

β = ratio of the electron velocity to that of light

$\alpha = z/137$

T = recoil energy transmitted to an atom

The threshold probability function $P(T)$ is a simple step function equal to unity above T_d and zero for $T < T_d$, in any particular crystallographic direction at 0°K. However, in reality, electrons coming into a specimen are scattered and the probability function should be a more complicated function. For polycrystalline iron a staircase function was found by Lucasson and Walker⁽¹⁰⁶⁾ to be a good approximation for the probability function:

$$\begin{aligned} P(T) &= 0 & 0 \leq T < T_1 \\ P(T) &= 0.6 & T_1 \leq T < T_2 \\ P(T) &= 1.0 & T_2 \leq T < T_m \end{aligned} \quad (3)$$

where $T_1 = 22$, $T_2 = 66$ eV and T_m is given by the formula⁽¹¹⁵⁾

$$T_m = [2mE(E + 2mC^2)]/[MmC^2] \quad (4)$$

Here, E is the incident electron energy. In the present experiment the incident electron energy is dissipated by a window placed in front of the specimen as well as by the specimen itself. A correction for the electron energy has to be made. This was done by using a Landau approximation.⁽¹¹⁶⁾ The energy loss was calculated to be ~ 0.02 MeV for a 1 mil thick stainless steel window or for a 10 mil thick beryllium window and 0.21 MeV for a 10 mil thick specimen for an incident electron energy of 1.3 MeV. This result yields an electron energy of 1.28 MeV at the front of the specimen

and 1.07 MeV at the back. Taking the arithmetic mean of these values and substituting into Eq. (4), the maximum recoil energy T_m was calculated to be 98.6 eV. By substituting Eqs. (2) and (3) into Eq. (1) and integrating over T , the total displacement cross section is given by

$$\sigma_{\alpha}(\text{TOTAL}) = (0.25 \text{ barns}) z^2 \frac{1-\beta^2}{\beta^2} \left[0.6 \left\{ \left(\frac{T_2}{T_1} - 1 \right) - \beta^2 \ln \left(\frac{T_2}{T_1} \right) + \pi \alpha \beta \left(2 \left(\frac{T_2}{T_1} - 1 \right) - \ln \left(\frac{T_2}{T_1} \right) \right) \right\} \right. \\ \left. + \left\{ \left(\frac{T_m}{T_2} - 1 \right) - \beta^2 \ln \left(\frac{T_m}{T_2} \right) + \pi \alpha \beta \left(2 \left(\frac{T_m}{T_2} - 1 \right) - \ln \left(\frac{T_m}{T_2} \right) \right) \right\} \right] \quad (5)$$

By using the values of $T_1 = 22$ eV, $T_2 = 66$ eV and $T_m = 98.6$ eV the value of σ_d was calculated to be 40.3 barns for the incident electron energy of 1.3 MeV.

As was mentioned in the previous chapter (p. 43), the window placed in front of the specimen caused scattering of the electrons. Therefore besides the correction for the electron energy, the electron dose had to be corrected. The detail of such a correction is given in Appendix C, where an estimate is made by using a Gaussian distribution for the electron distribution. For a specimen placed 7 mm away from a 1 mil thick stainless steel window, the fraction of electrons coming into the specimen was calculated to be 0.7 for the present set-up of the collimator and the specimen geometry.

b) Production rate of defects

The two experimentally measured production rates given in Fig. 16 can be explained by taking account of the scattering of electrons by the window. As discussed above, the energy dissipation due to the window is negligibly small for both materials. However the scattering angle is significantly different between these two materials, as given in Appendix C. The scattering parameter for the stainless steel window was calculated to

be $\sim 20^\circ$ while that for the beryllium window was calculated to be $\sim 3^\circ$. Therefore from Fig. 48 in the Appendix, the fraction of electrons coming into the specimen was obtained to be ~ 0.95 for the beryllium window and ~ 0.5 for the stainless steel window. Thus the resistivity for Frenkel pairs is estimated to be $11.5\Omega\text{-cm/at.}\%$. This value is in agreement with the value of $12.5\Omega\text{-cm/at.}\%$ given by Lucasson and Walker.⁽¹⁰⁶⁾ The defect concentration in the present set-up for the tensile test, is then given by a relation, $C = 2.82 \times 10^{-23} \phi$ where ϕ is the total electron dose measured at the collimator.

c) Recovery of irradiation-induced defects

The major recovery stage of the electron irradiation in iron was reported to be above 80°K .⁽¹⁰⁶⁾ This recovery feature of iron is significantly different from other f.c.c. and b.c.c. metals such as Al, Cu, Ag, Ni, Nb, , where the major recovery occurs above $20 \sim 30^\circ\text{K}$, and was one of the advantages in choosing iron as the specimen material in the present experiment. It is probably needless to point out that the maintenance of 60°K is far more economical and easier than the maintenance of 20°K . This peculiar recovery feature of iron is itself an interesting problem to investigate further. However, there was no irradiation experiment performed on an iron specimen which was as pure as or purer than the present specimen. Therefore, it was necessary to examine the effect of purity on the recovery and to assure the optimum choice of the temperature for our experiment.

As mentioned in Section III-2, the recovery was studied only up to 110°K with isochronal annealing. As shown in Fig. 17 the results confirmed that a major recovery stage was above 80°K , even in the ultrapure iron. The main recovery peak around 80°K was interpreted as due to free migration

of self-interstitials by Lucasson and Walker,⁽¹⁰⁶⁾ from the analogy to the f.c.c. case. However, Neely and Keefer⁽¹¹⁷⁾ presented a different interpretation where the long range migration of interstitials was attributed to the recovery stage at 120 ~ 150°K. In a recent work by Bourret,⁽¹¹⁸⁾ where the irradiation damage under a high voltage microscope is studied, the low production rate of secondary defects in iron is explained by the higher concentration of closed pairs in iron than in other materials. This observation indirectly supports the interpretation given by Neely and Keefer.

The slight enhancement of the second substage observed in the present experiment around 60°K must be due to the improved purity of the present specimen. The total recovery in the substages below 80°K was less than 13% of the total resistivity increase due to the irradiation, and must be due to the recombination of the Frenkel pairs of the closest proximity. No long range migration of interstitial atoms could take place during the irradiation and the subsequent mechanical test in the present experiment, as the specimen temperature was always kept below 60°K. Therefore, the observed irradiation effects must have been caused by nearly randomly distributed interstitial atoms. The majority of these interstitials were probably accompanied by the corresponding vacancies at some distance away.

3. Interpretation of electron irradiation softening

The irradiation softening observed in the present experiment is well explained by a local double kink nucleation model in the presence of misfit strain centers, which was first proposed by Urakami in his Ph.D. thesis.⁽⁹⁶⁾ This local double kink nucleation model was developed further by taking account of the sideways motion of kinks in the present study. An immediate

interpretation for this softening effect is seen in Fig. 37, where the energy required for overcoming a misfit strain center is shown to be lower than that of double kink nucleation in a perfect lattice especially at higher stress levels.

However, for a precise description, the energy vs. stress relation determined by the numerical calculation in the previous chapter must be converted to a stress vs. temperature, or a stress vs. defect concentration relation through a formula representing the thermally activated deformation process. The calculation of the energetics of the overcoming process of the misfit defects was carried out on a short dislocation segment. In order to describe the macroscopic deformation, the dislocation density and the defect concentration must be taken into account. A theoretical estimate for the expected concentration dependence of the effect of the misfit defects is given in the following calculations.

In order to simplify the calculation a regular distribution was assumed for the dislocations and a random distribution was assumed for the defect distribution. The additional assumptions were further made for the defects as follows:

- a) Only the defects within $5b$ from a dislocation line were taken as to interact with the dislocation.
- b) These defects were then assumed to be distributed randomly at $5b$ from the dislocation with equal strength.
- c) The average spacing $\bar{l}$ between the two adjacent defects along a dislocation was defined by

$$\bar{l} = \frac{1}{\pi(5b)^2 N} = \frac{1}{\pi(5b)^2} / \left(\frac{\sqrt{3}}{2} \cdot \frac{C}{b^3} \right) = 0.147b/C \quad (6)$$

where N is the number of the defects per unit volume and

where C is the defect concentration.

For the regular array of the dislocations, the dislocation segment length, L , between the forest dislocations will be given by

$$L = \sqrt{\frac{3}{\rho}} \quad (7)$$

where ρ is the dislocation density.

Let us consider a mean dislocation velocity $\bar{v}$ through the randomly distributed defects. A local dislocation velocity must be closely related to the local defect concentration. We take into account this local defect dislocation by dividing the local concentration into three groups. In the region where the defect concentration is very low (the defect separation, $l > 2L$) the motion of a dislocation segment of length L must occur mostly by a double kink nucleation in a perfect lattice. In such a region the local dislocation velocity will be given by

$$v_o = (aL v_o / w) \exp(-E_o / kT) \quad (8-a)$$

as proposed by Dorn and Rajnak.⁽⁸⁸⁾ The symbols in the equation are:

a = taken to be the same as b

$v_o(\frac{b}{w})$ = the attempting frequency for the double kink nucleation
(w = double kink width)

E_o = the activation energy for the double kink nucleation in
a perfect lattice

k = Boltzmann constant

T = absolute temperature

In a slightly higher defect concentration region ($2L > \ell > L/2$), we expect the presence of a small number (one or two) of misfit defects along a dislocation segment of length L . Therefore the double kink nucleation will occur with the aid of a misfit strain center and once the defect is overcome the kinks will spread out spontaneously to the end of the dislocation segment without encountering other defects. Thus the local dislocation velocity will be given by

$$v_1 = (naLv_o/w) \exp(-E_n^{(S \text{ or } N)}/kT) \quad (8-b)$$

where $E_n^{(S \text{ or } N)}$ is the energy required for overcoming a defect as determined in Fig. 37, n is the number of steps in a double kink which is the same number as the subscript of $E_n^{(N \text{ or } S)}$, and the rest of the symbols are the same as for Eq. (8-a). In the region of high local defect concentration ($\ell < L/2$), the kinks will encounter other defects before they reach the end of a dislocation segment. Therefore the local forward dislocation velocity will be controlled by a kink collision. In such a region the local velocity is given by⁽⁸⁷⁾

$$v_2(\ell) = na(2v_n^N v_n^S \ell)^{1/2} = (\sqrt{2} nabv_o/w) \ell^{1/2} \exp\left(-\frac{E_n^N + E_n^S}{2kT}\right) \quad (8-c)$$

as a function of a defect separation of ℓ . By assuming that the number of dislocation segments having a length between ℓ and $\ell + d\ell$ is given by $\bar{\ell}^{-2} \exp(-\ell/\bar{\ell}) d\ell$ per the unit dislocation line,^(121,158) the local mean velocity obeying the kink collision mechanism will be given by

$$\begin{aligned} \bar{v}_2 &= \frac{\int_0^{L/2} v_2(\ell) \bar{\ell}^{-2} \exp(-\ell/\bar{\ell}) d\ell}{\int_0^{L/2} \bar{\ell}^{-2} \exp(-\ell/\bar{\ell}) d\ell} \\ &= \left\{ \frac{\sqrt{2} nabv_o/w/\bar{\ell}}{1 - \exp(-L/2\bar{\ell})} \right\} \exp\left(-\frac{E_n^N + E_n^S}{2kT}\right) \int_0^{L/2} \ell^{1/2} \exp(-\ell/\bar{\ell}) d\ell \end{aligned} \quad (8-c')$$

The mean dislocation velocity through the randomly distributed defects can then be given in terms of v_0 , v_1 and $\bar{v}_2$ by

$$\bar{v} = \frac{1}{f_0/v_0 + f_1/v_1 + f_2/\bar{v}_2} \quad (9)$$

where f_0 , f_1 and f_2 are the fractional sites where the dislocation velocities are given by the local velocities v_0 , v_1 and $\bar{v}_2$, respectively, and they can be approximated by the following relations:

$$f_1 = \frac{\int_{L/2}^{2L} \bar{l}^{-2} \exp(-l/\bar{l}) dl}{\int_0^{\infty} \bar{l}^{-2} \exp(-l/\bar{l}) dl} = \exp(-L/2\bar{l}) - \exp(-2L/\bar{l}) \quad (10-a)$$

$$f_2 = \frac{\int_0^{L/2} \bar{l}^{-2} \exp(-l/\bar{l}) dl}{\int_0^{\infty} \bar{l}^{-2} \exp(-l/\bar{l}) dl} = 1 - \exp(-L/2\bar{l}) \quad (10-b)$$

$$f_0 = 1 - (f_1 + f_2) = \exp(-2L/\bar{l}) \quad (10-c)$$

The average defect spacing along a dislocation, $\bar{l}$, which appeared in the above equation is related to the defect concentration through Eq. (6).

Since the macroscopic strain rate is generally given by $\dot{\gamma} = \rho_m b v$ where ρ_m is the mobile dislocation density, $\dot{\gamma}$ in the present model can be written by

$$\dot{\gamma} = \rho_m b \bar{v} = \frac{\rho_m b}{f_0/v_0 + f_1/v_1 + f_2/\bar{v}_2} \quad (11)$$

Then the stress vs. concentration or the stress vs. temperature relation can be computed numerically, by using Eqs. (11), (10) and (8), if we can write the energy term in Eq. (8) as a function of the stress.

As pointed out by Mifune,⁽¹¹⁹⁾ the activation energy for overcoming

a point defect in f.c.c. metals has been given by a formula,

$$E(\tau/\tau_p) = E^*[1 - \{(\tau/\tau_p)/(\tau_c/\tau_p)\}^\alpha]^\beta \quad (12)$$

in any case of the models given in the literature. This type of function was also used in the case of double kink nucleation⁽¹²⁰⁾ as a good approximation. In the calculation given in the previous chapter the energy required for the overcoming of a defect or the double kink nucleation was determined at the stress levels ($\tau/\tau_p = 0.1, 0.3, 0.5$ and 0.7) as shown in Fig. 35. Eq. (12) contains four unknown constants, E^* , τ_c/τ_p , α and β . Therefore the four constants in Eq. (12) can be computed by using the four sets of values of E and τ/τ_p , giving the best fit to the curves shown in Fig. 37. The constants thus determined are listed in Table II. The constants determined from Fig. 35 for $E(\tau/\tau_p) = \frac{E_n^N + E_n^S}{2}$ are in parentheses.

Let us return to Eq. (8). Now, since we know the analytical relation between E and τ/τ_p , we can calculate the velocities v_0 , v_1 and $\bar{v}_2$ for a given set of constants such as dislocation segment length, stress, temperature and so on. By substituting Eqs. (8) and (10) into Eq. (11) one can compute the right hand side of Eq. (11). A stress level giving a desired strain rate $\dot{\gamma}$ was determined by an iteration method by varying the stress.

The stresses thus determined are plotted against the defect concentration in Fig. 46, where the misfit strength is assumed to be $\epsilon = 0.35$. The parameters and constants used in this computation are given below:

$\dot{\gamma} = 10^{-3}$	$v_0 = 10^{13}$
$\rho = 10^8$	$w = 50b$
$\rho_m = \rho/3$	$k = 0.862 \times 10^{-4} (\text{eV}/^\circ\text{K})$
$a = b = 2.48 \times 10^{-8} \text{ cm}$	$n = 3$
	$\epsilon = 0.35$

Table II. Constants, E^* , τ_c/τ_p , α and β determined from Figs. 35 and 37 by curve fitting.

ϵ	$E^*(\text{eV})$	τ_c/τ_p	α	β
0	0.641	1.00	0.77	1.24
0.05	0.453	0.89	0.80	1.37
0.15	0.391	0.90	1.00	2.08
0.25	0.399	0.95	1.30	2.94
0.35 (0.35)†	1.046 (0.800)	0.78 (0.79)	0.52 (0.54)	1.35 (1.58)
0.50 (0.50)†	1.760 (1.134)	1.10 (1.30)	0.51 (0.51)	1.81 (1.59)

† The parentheses indicate the parameters for the kink collision mechanism, $E(\tau/\tau_p) = (E_n^N + E_n^S)/2 = E^*[1 - (\tau/\tau_c)^\alpha]^\beta$.

Fig. 46 Concentration dependence of the flow stress. The results for $c = 0.35$ case. The open circles are for a perfect crystal. Note the monotonic hardening at higher temperature, and the softening due to low concentration of defects. At 4.2°K the softening effect is not expected for a defect concentration below 200 ppm.

The temperatures used for the calculation are indicated on the curves in the figure. The zero misfit cases are indicated by open circles on the ordinate axis.

As seen in the figure, the irradiation softening at relatively low temperatures can be explained by the local double kink nucleation model. At the same time the present results of the calculation can give a description for the appearance of the hardening effect, due to a small amount of interstitial impurities at higher temperatures, or due to a relatively high concentration of defects at lower temperatures. Since the exact strength of the misfit strain centers is not known, the quantitative discussion cannot be made here. However, the nature presented in the foregoing discussion is also true for the different strengths of the misfit strain centers from $\epsilon = 0.05$ to 0.35 studied here. This fact clearly indicates that the nature derived in the present calculation will apply for general cases.

At very low temperatures, however, the defect concentration range giving the observable softening effect is expected to be relatively high ($C = 10^{-3}$) compared to the present case of electron irradiation (5×10^{-5}). Thus, the softening effect due to the dispersed barriers is not expected at the 4.2°K test with the defect concentration of the present study. Therefore the experimental observation (no softening at 4.2°K) shown in Fig. 21 is in agreement with the present model.

4. Comments on the alloy softening

The occurrence of the softening due to the addition of impurity atoms has often been observed in b.c.c. metals at low temperatures. However, the interpretations of these softening effects have been given in various ways. As reviewed in Chapter I, they were categorized into five groups:

- (a) As reported by Conrad,⁽¹²²⁾ and by Nakada and Keh,⁽¹³⁾ the pre-exponential term in the rate equation is considered to be responsible for the softening effect; namely, the mobile dislocation density is assumed to increase by impurities. The increase in the dislocation density in less pure iron single crystals has been reported by etch pit study.^(123,124)
- (b) As proposed by Arsenault,⁽⁹²⁾ the addition of impurities can cause reduction in the Peierls potential.
- (c) A scavenging effect is also considered as a responsible mechanism by several workers.^(8,9,21) Especially in the study on Ta-Re-N system by Simi-alek et al.,⁽²¹⁾ the presence of the scavenging effect is well demonstrated.
- (d) The interaction of impurities with the dissociated screw dislocation can be considered to lower the flow stress as proposed by Christ, et al.⁽⁴⁰⁾
- (e) Local double kink nucleation in the presence of misfit defects can also explain the alloy softening.^(96,97)

Although the first mechanism is possible, there is no direct evidence for such an observation, namely, the quantitative evidence relating the flow stress decrease with the increase in mobile dislocation density. The irradiation softening observed in the present experiment strongly suggests that the softening can be caused by the direct effect of interstitials on dislocation motion, since the introduction of interstitials by electron irradiation is not expected to change the dislocation structures. The second mechanism may operate in some alloy systems as discussed by Arse-

nault,⁽⁹²⁾ although this mechanism does not apply to the present case.

The third mechanism should also be true in some cases, where the concentration of the interstitial impurities is relatively high, and where the tendency of the alloying element forming a compound with the interstitials is high. However, the fact that the softening appears only at lower temperatures but not at higher temperatures in some alloy systems may not be explained by the scavenging effect as pointed out by Arsenault.⁽⁹²⁾

Clearly, this mechanism does not apply to the present case. On the other hand, the irradiation softening has been successfully explained by a local double kink nucleation model. When this model is applied to some solid solution cases, the solid solution softening can also be explained and at the same time the monotonic hardening at higher temperatures can be explained without invoking additional assumptions. It should be noted here that the strength of a misfit strain center is determined by the product of misfit strain and the defect volume. Therefore, for a substitutional solid solution or for a case of weak misfit strain, localized regions of ordered solute atoms associated with strain or solute clusters should be considered as the local double kink nucleation site. In such a case, accordingly, the concentration range giving the observable flow stress decrease will shift to the higher concentration. Thus the difference in the softening rates between the Fe-N and Fe-Ni systems⁽⁴⁰⁾ can be explained. Furthermore, in the present model, the double kink nucleation and the overcoming of defects are considered within a slip plane. Therefore the present model does not conflict with the slip line observation where the coarser and straighter slip lines⁽¹³⁾ were reported in less pure specimens, while the impurity-induced cross slip model may not explain the nature of this slip line observation.

5. On the appearance of twinning

For a high electron dose corresponding to ~ 50 ppm of self-interstitials, twinning was observed upon testing right after the irradiation (cf. Fig. 28). This twinning was also observed in the less pure specimen (specimen F) as demonstrated in Fig. 10. The stress levels for the occurrence of this twinning were, however, always lower than the macroscopic flow stress of the purest specimen (specimen B) in which the twinning was never observed. These facts indicate that the occurrence of twinning is closely related to the presence of interstitials, and that there is a critical concentration for this under a given test condition.

According to the present calculation, the presence of interstitials causes a softening effect for the plastic deformation at low temperatures to a critical concentration. The experimental observation of the softening effect due to the electron irradiation, is in agreement with the results of this calculation. If we assume that a similar type of softening can be applicable to the threshold stress for the nucleation of a twin, the threshold stress is expected to be lowered by the presence of interstitials. Thus in the presence of interstitials the appearance of twinning can be observed at a lower stress than in the case for the purer specimen.

6. Hardening due to post-irradiation aging

As seen in Figs. 18 and 25, the post-irradiation aging produced a significant increase in the flow stress. The temperature dependence of this hardening effect was also studied as shown in Fig. 27, resulting in a temperature independent hardening. The athermal increase of flow stress was also obtained by McRickard⁽⁶⁴⁾ in the neutron irradiation experiments

at ambient temperatures. On the other hand, according to Seidel,⁽⁶⁰⁾ Kitajima⁽⁶¹⁾ and Ohr et al.,⁽⁶²⁾ the hardening due to neutron irradiation at ambient temperatures is reported to be temperature dependent; namely, the hardening is enhanced at lower temperatures. Seidel⁽⁶⁰⁾ studied the effect of neutron irradiation on both the upper and lower yield stresses in zone-refined iron. The data presented in the study clearly indicated that the neutron irradiation caused temperature dependent hardening for both the upper and lower yield stresses. Ohr et al.⁽⁶²⁾ also showed the temperature dependent hardening in zone-refined niobium, and they attributed the discrepancy observed in the temperature dependence to the difference in the specimen purity. Thus the temperature dependent hardening in the heavily irradiated b.c.c. metals appears to be well established. However, as pointed out in Chapter I, the source for the appearance of this hardening is hardly considered to be relevant to that of interstitial hardening. Therefore the observation of the temperature dependent hardening in the neutron irradiated case may not be inferred to the discussion of the strength of the ideally pure b.c.c. metals.

However, in the present study, the electron irradiation was used, and hence the size of the defects which might be formed by the post-irradiation aging is considered to be much smaller than the size of defects in the neutron irradiation experiment. As will be discussed later in this section, the formation of small defect clusters is very likely. If it is so, a small defect cluster may be considered to contribute to the plastic deformation in a similar way as the interstitial impurities. Thus the temperature insensitive hardening observed here for the macroscopic flow stress suggests the importance of the inherent lattice hardening. This is strongly supported by the irradiation softening observed at low temperatures

without any annealing treatment.

The apparent activation energy for the aging process determined from Fig. 25 is 0.60 eV for the irradiated case, and 0.45 eV for the unirradiated case. These values are much smaller than the migration energy of carbon or nitrogen in iron, where most of the earlier works have agreed with the values of 0.85 - 0.87 eV for carbon⁽¹²⁵⁻¹²⁷⁾ and 0.70 - 0.79 eV for nitrogen.⁽¹²⁸⁻¹³¹⁾ Fujita and Damask⁽¹³²⁾ analyzed the two distinct steps at -30°C and 50°C in the isochronal annealing after neutron irradiation on 0.01 wt.%C alloy and they obtained 0.50 eV for the first step and 0.86 eV for the second step. According to their interpretation the first step is due to the release of self-interstitials from the carbon trap, and the second step is due to the free migration of carbon. Recently, Glaeser and Wever⁽¹³³⁾ obtained the activation energy of 0.48 - 0.51 eV by the isochronal annealing at 300 - 400°K after quenching into liquid helium with high purity iron. This process was explained by the motion of vacancy carbon pairs. The age hardening without irradiation may be explained by the motion of vacancy carbon pairs from the agreement of the activation energy for the process. The increased activation energy in the irradiated case may be related to the release of self-interstitials trapped around carbon. Although the expected clusters could not be identified at this stage, the recent work, using a high voltage electron microscope,^(124,134) clearly shows the formation of defect clusters in high purity iron.

Theoretically, the interaction between a stronger defect and a dislocation requires an increase in the energy for overcoming the defect as seen in the case of $\epsilon = 0.50$ in Fig. 37. Here, the misfit strength is given by a product of the misfit strain and the volume, as mentioned in the previous section. Thus, the increase in the defect volume may be the cause of the

hardening due to the electron irradiation followed by the aging treatment.

As seen in Fig. 26(a), the effect of electron irradiation on the appearance of the yield drop was not significant. However, the specimen purity was quite sensitive to this phenomenon. As seen in the comparison of Figs. 26(a) and 26(b), the rate of the saturation was much faster for the less pure specimen (specimen F), while the saturation value was nearly the same for both cases. The activation energy for the process was calculated to be 0.40 eV for specimen C and 0.45 eV for specimen F, according to the Cottrell-Bilby relation,⁽¹³⁶⁾

$$N(t) = n_0 3 \left(\frac{\pi}{2} \right)^{1/3} \left(\frac{ADt}{kT} \right)^{2/3} \quad (16)$$

where $N(t)$ is the total number of impurities coming into the dislocation, n_0 is the density of impurities in the matrix, D is the diffusion coefficient, and A is a parameter which depends on the elastic constants, the volume change caused by the interstitial impurities, and the strength of the dislocation. By using the activation energy as determined above and also by using a value of 0.02⁽¹²⁵⁾ for a pre-exponential term in the diffusion coefficient, a mean diffusion distance was found to be $\sim 10^{-5}$ cm for specimen F. These values are much larger than the values given for the higher concentration of carbon⁽¹³⁷⁾ and nitrogen,⁽¹³¹⁾ where the distance was determined to be $10^{-7} \sim 3 \times 10^{-7}$ cm. These facts indicate that for a high concentration of impurity interstitials a small fraction of them is enough to lock mobile dislocations, and that only the impurities very close to the mobile dislocations contribute to the formation of the Cottrell atmosphere. However in a high purity sample a long range migration is required. The separation of the slip bands was measured to be approximately $\sim 10^{-3}$ cm, and this value coincides with the value

of the mean diffusion for specimen C, indicating that the impurities in most of the matrix are required to form the Cottrell atmosphere in a high purity sample.

7. Irradiation hardening in the microyield region

As demonstrated in Figs. 28, 29 and 30, the electron irradiation on a prestrained specimen caused a significant hardening effect in the microyield region. In the tests, dislocations were introduced by prestraining at 24.5°C prior to the irradiation and subsequent tensile tests at low temperatures. The characteristic features which appeared in the present tests are as follows:

- a) The hardening effect appeared only in the microyield region.
- b) The effect was strongly enhanced at lower temperatures.
- c) The effect was also enhanced by post-irradiation aging.

These observations yield strong clarification on the plastic deformation mechanism of b.c.c. metals.

As has been seen in several earlier works,^(96,138-143) the dislocations introduced at higher temperatures are quite different from those at low temperatures; namely, a fraction of the edge components is higher when a pure b.c.c. crystal is deformed at higher temperatures. A number of recent experiments on the microplasticity of b.c.c. crystals have been analyzed by using dislocation dynamics and most of the analyses have agreed that impurities affect the temperature dependence of the microyield stress more than that of the macroscopic flow stress.⁽⁴⁶⁻⁵⁰⁾ The present experimental results are relevant to some of the earlier works in the sense that the results support the same conclusion, that is, the interaction of edge dislocations with dispersed obstacles is responsible in the microyield region.

However, the present study can give more clarified confirmation to the mechanism. First, the specimen history prior to the electron irradiation was identical and hence the comparison among the specimens of different interstitial concentrations made here is more reliable than in the case where a heat treatment had to be given for specimens of different purity. Second, the observation of the softening effect in the macroyield region gives strong confirmation on the point that a different type of dislocation must be operating in the micro- and macrostrain regions. Finally, the enhancement of the hardening due to post-irradiation aging indicates that the interaction of the defects with edge dislocations is responsible for the hardening mechanism, because the condensation of the defects is more expected to edge dislocations than to screw dislocations.

The possible explanation of this temperature dependent hardening can be given by using the results of the present calculation. In the calculation a non-screw type of dislocation was introduced as a sideways motion of kinks. This motion may be considered as a special case of the motion of edge dislocations, because the sideways motion of kinks clearly involves the motion parallel to the Burgers vector. The activation energy required for overcoming the misfit strain center was obtained in the calculation. The result showed that the overcoming energy is stress dependent as shown in Fig. 35; namely the motion of the edge dislocation over the misfit strain center is controlled by a thermally activated process. The temperature dependent hardening due to the electron irradiation clearly supports the above discussion. Thus the present experimental results and the calculation provide the convincing conclusion that the plastic deformation in the microyield region is controlled by the motion of the edge dislocations over the dispersed misfit strain centers. The present discussion

also substantiates that the softening observed in the macroyield region is due to the dispersed misfit strain centers.

8. Comments on the controversy concerning the role of interstitial impurities

There has been a long-standing controversy concerning the role of interstitial impurities on the plastic deformation of b.c.c. metals, as reviewed in Chapter I. The strong temperature dependence of the flow stress in b.c.c. metals has been interpreted in two conflicting ways. One considers the importance of the role of interstitial impurities and the other considers the inherent lattice potential as being responsible.

In most of the works^(1-4,6,8,9) supporting the impurity hardening mechanism the yield stress was taken at a proportional limit. However, as shown in the present experiment (cf. Fig. 15) and also in the literature,^(43,48-50) the determination of the yield stress by taking the proportional limit is strongly dependent on the accuracy of the strain measurement and the amount of prestrain. This is true even for an unprestrained specimen, since there are always residual dislocations, even after annealing at a higher temperature as demonstrated in Fig. 15. The yield stress taken at the proportional limit is most likely controlled by the motion of edge dislocations as discussed in the previous section, where the temperature dependent hardening was noted in the microyield region. This nature was confirmed by direct observation under the high voltage electron microscope by Imura.⁽⁵³⁾ Therefore it seems natural that the effect of the dispersed obstacles on such a stress appears to be strongly temperature dependent as was seen in the present irradiation experiment. The present calculation (cf. Fig. 36) also predicts that the

interaction of the dispersed obstacles with the edge component of a dislocation causes temperature dependent hardening effects, in agreement with experimental observation.

On the other hand, if the stress level is taken at higher strains, most of the edge components are gone, as suggested in the exhaustion hardening theory^(48,50) and also proved by direct observation.⁽⁵³⁾ The stress level at higher strains must then be determined by the motion of the screw dislocations. The results of the calculation revealed that the presence of a small amount of interstitials causes a softening effect for the motion of screw dislocations. The electron irradiation softening observed in the macrostrain region, further, confirmed the softening effect on the motion of the screw dislocations.

The athermal increase of the flow stress by the stronger obstacles can also be explained by the present calculation, as shown by the curve corresponding to $\epsilon = 0.50$ in Fig. 37. This nature is again consistent with the present experimental observation, where the temperature insensitive hardening was observed for the irradiated and aged specimen. For the atomic scale defect as given by Nakada and Keh,⁽¹³⁾ the hardening effect is expected only at higher temperatures, as supported by the results of the calculations (cf. Fig. 37 and 46).

Thus the foregoing discussions yield the following conclusion concerning the rate controlling mechanism of plastic deformation in b.c.c. iron: The strength of iron at a low temperature is basically controlled by the inherent nature of the b.c.c. lattice while the low concentration interstitials cause a softening effect at low temperatures and a hardening effect at higher temperatures. On the other hand stronger obstacles, such as small clusters introduced by electron irradiation and aging or small

solute clusters, cause a temperature insensitive hardening effect in a relatively wide temperature range.

9. Comments on the extension of the present work

a) The effect of electron irradiation on other high Peierls potential materials

Observation of the electron irradiation softening provided strong evidence for the presence of high lattice resistance in b.c.c. iron. According to the present model, the observation of the softening must be reproducible in other high Peierls potential materials. To accomplish such an experiment, several requirements must be satisfied. In this paragraph, these requirements are examined for several high Peierls potential materials including a few h.c.p. crystals.

(a) A specimen must be plastically ductile at low temperatures.

(b) Since the softening effect is not expected at absolute zero temperature but only at intermediate temperatures as discussed in section 3, the recovery temperature of the irradiation-induced defects must be reasonably high.

(c) The residual impurity level must be low enough so that the addition of defects by irradiation can cause a softening effect. This critical concentration is expected to be relatively low according to the present experiment, although the critical concentration may be different for the different materials.

Various natures concerning the above-mentioned requirements are listed in Table III for several materials which are supposed to have high Peierls potential. As seen in the table, iron appears to be the most appropriate material. However, there are a few possible materials which

Table III. Examination of the applicability of some b.c.c. and h.c.p. metals for the observation of irradiation effect.

Material	Atomic number	Ductile temp (°K)	Interstitial impurity (ppm)	T _d [*] (eV)	E _{min} ^{**} (MeV)	T _R ^{***} (°K)
Fe	26	4.2 ^(a)	5 ^(a)	24 ^(b)	1.0	80 ^{(a), (b), (c)}
Nb	41	17 ^(d)	$\rho_{300^\circ\text{K}}/\rho_{4.2^\circ\text{K}} = 600^{(d)}$	36 ^(e)	1.6	30 ^{(f), (g)}
Mo	42	20 ^(h)	15 ^(h)	37 ^(b)	1.7	25 ^{(b), (i)}
Ta	73	4.2 ^(j)	10 ^(j)	32 ^(e)	2.1	40 ^(f)
V	23	$\rho_{300^\circ\text{K}}/\rho_{4.2^\circ\text{K}} = 15^{(k)}$				
Ti	22	77(basal) 4.2(non-basal)	115 ^(l)	29 ^(b)	0.9	60 ^(b)
		} ⁽¹⁾				
Mg	12	4.2(basal) 77(non-basal)				4.2 ⁽ⁿ⁾
		} ^(m)				
Zn	30					13 ^(o)

* T_d : Threshold energy

** E_{min} : Electron energy required to give $T_m > 3 \times T_d$

*** T_R : Recovery temperature for $\Delta\rho/\Delta\rho_o = 80\%$

AlphabetReferences

a	present work
b	106
c	117
d	144
e	147
f	148
g	149
h	6
i	150
j	21
k	151
l	145
m	97
n	151
o	152

may be applicable for the study. Tantalum is the most promising material among the b.c.c. metals listed in the table, as the recovery temperature is relatively high, provided that enough electron energy can be generated to transfer a relatively high recoil energy to the tantalum atoms. In the case of h.c.p. metals, titanium may be the most interesting material, where the controversy^(145,146) about the role of interstitial impurities on the plastic deformation in a non-basal slip system has been reported, and the large temperature dependence of the flow stress for the basal slip has not been explained. The main recovery temperature is also relatively high as listed in the table.

b) Detailed study on the irradiation hardening in the microyield region

In the present study, the hardening due to electron irradiation was studied on a total stress strain diagram, but an elastic limit σ_E or microyield stress σ_A was not determined. This is because the measurement of σ_E and σ_A requires more accuracy for the strain measurement than in the present experiment, since a stress cycling test must be employed to resolve these two quantities. When a stress cycling test is employed, the strain produced for each cycle is very small. Therefore the return of the strain upon complete unloading must be reproducible in the strain sensitivity of 10^{-6} to measure the appearance of a hysteresis loop and deviation in the unloaded strain (permanent strain).

Thus by improving the experimental techniques the effect of electron irradiation on σ_E and σ_A can be studied. The results will yield more precise information for the interaction of interstitial atoms with the dislocations.

Furthermore, it will be more meaningful if the experimental results

can be correlated to theoretical studies. In the next paragraph the extension of the present calculation is presented. The formulae derived there will be applicable for the microstrain experiment.

c) Double kink nucleation over two-dimensional Peierls potential

The elastic limit σ_E in the microstrain experiment has generally been explained by the appearance of the motion of geometrical kinks, and σ_A has been attributed to the motion of edge dislocations over dispersed obstacles. The transient from the microyielding defined by σ_A to macroscopic yielding, has then been explained by the exhaustion hardening theory. As discussed in the previous section, the above model is strongly supported by the present experiments. However, no theoretical calculation has been performed on the model.

In this paragraph, one method, which is simply an extension of the present calculation, is suggested in order to study the motion of the edge dislocations. Suppose a dislocation loop of which two parallel screw components lie in the Peierls potential valley along the x-axis. The energy for the motion of the edge dislocation can then be given by the integration along the y-axis instead of the integration along the x-axis used in the present study [cf. Eq. (4) in IV-1]. The excess energy referred to $x = x_0$ is then written by

$$U = \int_{y_1}^{y_2} [\Gamma(x, y) \sqrt{1+x'^2} - \Gamma(x_0, y) - \int_{x_0}^x F(x, y) dx - \tau b(x - x_0)] dy \quad (17)$$

where $\Gamma(x, y)$ represents the two-dimensional Peierls potential, and the meanings of the other terms are identical to Eq. (4) in Chapter IV. The Euler's condition [cf. Eq. (6) in Chapter IV] can be given by

$$\Gamma(x,y)(1+y'^2)^{-3/2} \frac{dx'}{dy} - \frac{\partial \Gamma}{\partial x}(1+y'^2)^{-1/2} + \frac{\partial \Gamma}{\partial y}(1+y'^2)^{-1/2} x' - F(x,y) - \tau b = 0 \quad (18)$$

Therefore if an analytic formula for $\Gamma(x,y)$ is given, the above equation can be solved in the same way as for the motion of a screw dislocation. In the above equation, if $F(x,y)$ is taken as zero then the solution for Eq. (18) will explain the motion of the edge dislocations over the Peierls barrier along the x-axis. If $F(x,y)$ is not zero then Eq. (18) will give the motion of edge dislocations over the combined stress field of Peierls potential and misfit strain centers.

To study the motion of geometrical kinks, however, the dislocation configuration has to be taken in a way similar to that shown in Fig. 34(b). By applying the same procedure as used there, the energetics for the sideways motion of kinks over the Peierls potential along the x-axis must be obtained. The equations corresponding to Eqs. (4) and (6) in Chapter IV are given by

$$U = \int_{\ell_1}^{\ell_2} [\Gamma(x,y) \sqrt{1+y'^2} - \Gamma(x,y_0) - \int_{y_0}^y F(x,y) dy - \tau b(y-y_0)] dx \quad (19)$$

and

$$\Gamma(x,y)(1+y'^2)^{-3/2} \frac{dy'}{dx} - \frac{\partial \Gamma}{\partial y}(1+y'^2)^{-1/2} + \frac{\partial \Gamma}{\partial x}(1+y'^2)^{-1/2} y' - F(x,y) - \tau b = 0 \quad (20)$$

If $\frac{\partial \Gamma}{\partial x} = 0$, namely, Γ is not a function of x , the above equation becomes identical to Eq. (6) in Chapter IV.

VI. APPENDIX

A. Purification by ZrH_2 system

A thermodynamic analysis given by Stein et al.⁽¹⁾ is reviewed in this section. In their analysis it was assumed that carbon was transferred as methane from a specimen to ZrH_2 by means of a circulating hydrogen atmosphere, nitrogen was transferred as ammonia gas, and oxygen as water vapor. The additional assumption made there was that the activity of zirconium was approximately unity because of the low stability of the ZrH_2 compound at high temperature.

On the basis of the above-mentioned assumptions, theoretical residual amounts of carbon were estimated as follows:

From Eq. (3) the mass action law yields $K = P_{H_2}^2 / P_{CH_4}$. Then the thermal equilibrium condition can be written by

$$\Delta F = -RT \ln \left(\frac{P_{H_2}}{P_{CH_4}} \right) \quad (4)$$

Rearranging the terms one can rewrite this equation by

$$P_{CH_4} = P_{H_2}^2 e^{\Delta F/RT} \quad (5)$$

By substituting the values for ΔF from Eq. (3), and taking $P_{H_2} \approx 1$ atm., the equilibrium partial pressure for CH_4 was calculated to be $P_{CH_4} = 5.88 \times 10^{-11}$ atm.

On the other hand, a theoretical partial pressure of CH_4 under equilibrium in the specimen furnace at 850°C was calculated to be $P_{\text{CH}_4} \approx 0.03 \text{ atm}$. When these two partial pressures are compared, carbon atoms in the specimen must be pulled out and transferred to the ZrH_2 , to attain the thermal equilibrium condition. Thus the theoretical residual carbon concentration was estimated to be $\text{wt.\%C} = 5.28 \times 10^{-7} \text{ ppm}$ by following the relation $\text{wt.\%C} = 0.9(P_{\text{CH}_4}/P_{\text{H}_2}^2)$ given by R. P. Smith.

The amount of nitrogen remaining in the specimen was calculated as follows:

Then the partial pressure of NH_3 was calculated to be $P_{\text{NH}_3} = P_{\text{H}_2}^{3/2} e^{\Delta F/RT} = 1.7 \times 10^{-24}$. By taking account of the low solubility of nitrogen in iron at high temperature and hence by applying the Henry's law the remaining nitrogen was calculated to be $\text{wt.\%N}_2 = 2.3 \times 10^{-11} \text{ ppm}$.

For estimating the amount of oxygen the following method was used in their report:

For the formation of a layer of FeO on Fe the equilibrium value of $P_{\text{H}_2\text{O}}/P_{\text{H}_2} = 0.2$, and for iron in equilibrium with FeO (saturated with oxygen) the oxygen in iron is less than $0.001 \text{ wt.\%}$ at 1073°K . Therefore the

maximum wt.% of oxygen in the system is estimated to be wt.% $O_2 = 1.6 \times 10^{-15}$.

Thus, the estimate given for these three nonmetallic impurities resulted in showing such low concentrations. Therefore, as far as these interstitial impurities are mobile in the crystal, the impurity concentration should approach the theoretically expected equilibrium concentration.

They also measured the residual carbon concentration by using a radioactive tracer technique, and it was shown that carbon was reduced from 50 ppm to less than 0.005 ppm. This fact clearly verified that iron can be purified by the ZrH_2 treatment.

B. Derivation of the stress field due to tetragonal misfit strain centers

By employing the equation given by Eshelby,⁽¹⁵⁴⁾ the displacement due to any shape of misfit defect can be derived in the matrix away from the defect. This equation is written by

$$U_i^C(\bar{X}) = V \epsilon_{jk}^T g_{ijk} / 8\pi(1-\nu)r^2 \quad (1)$$

which has been already referred to in Chapter IV. The meaning of the symbols are

- U^C - actual displacement
- V - volume of misfit strain center
- ϵ_{jk}^T - stress-free strain
- r - Distance from the strain center
- $\bar{X}$ - point of observation
- ν - Poisson's ratio

$$g_{ijk} = (1 - 2\nu)(\delta_{ij}l_k + \delta_{ik}l_j - \delta_{jk}l_i) + 3l_i l_j l_k \quad (2)$$

where l_i , l_j and l_k is the direction cosine and δ_{jk} is the Kronecker delta. Since the material is assumed to be isotropic, ϵ_{11}^T , ϵ_{22}^T and ϵ_{33}^T can be chosen as the only non-zero terms if they are taken along principal strain axes. If a misfit strain center is placed at the origin of the coordinate system for the observation, the direction cosine can be given by

$$l_1 = \frac{x}{r} = x / \sqrt{x^2 + y^2 + z^2} \quad (3-a)$$

$$l_2 = \frac{y}{r} = y / \sqrt{x^2 + y^2 + z^2} \quad (3-b)$$

$$l_3 = \frac{z}{r} = z / \sqrt{x^2 + y^2 + z^2} \quad (3-c)$$

As an example of the simplest case, the solution for a spherical misfit case is given as follows: since $\epsilon_{11}^T = \epsilon_{22}^T = \epsilon_{33}^T = \epsilon^S$ for a spherical misfit strain center, the actual displacement can be given by

$$\begin{aligned} U_1^c &= v \epsilon_{ijk}^S g_{1jk} / 8\pi(1 - \nu) r^2 \\ &= v \epsilon^S (g_{111} + g_{122} + g_{133}) / 8\pi(1 - \nu) r^2 \end{aligned} \quad (4)$$

Substituting for g_{111} , g_{122} and g_{133} from Eqs. (2) and (3), Eq. (4) can be written by

$$U_1^c = \frac{v \epsilon^S (1 + \nu)}{4\pi(1 - \nu)} \cdot \frac{x}{(x^2 + y^2 + z^2)^{3/2}} \quad (5-a)$$

Similarly,

$$U_2^c = \frac{v \epsilon^S (1 + \nu)}{4\pi(1 - \nu)} \cdot \frac{y}{(x^2 + y^2 + z^2)^{3/2}} \quad (5-b)$$

$$U_3^c = \frac{v \epsilon^S (1 + \nu)}{4\pi(1 - \nu)} \cdot \frac{z}{(x^2 + y^2 + z^2)^{3/2}} \quad (5-c)$$

By using a standard transformation from a displacement function to a stress function, $\sigma_{ij} = \mu \left(\frac{\partial U_j}{\partial x_i} + \frac{\partial U_i}{\partial x_j} \right)$, one of the stress components at a point (x, y, z) is given by

$$\sigma_{zx} = \frac{6V\epsilon^S}{4\pi(1-\nu)} z x \mu (x^2 + y^2 + z^2)^{-5/2} \quad (6)$$

This result is consistent with the one referred to in the text [Chapter IV, Eq. (7)]. The difference in the constant term between this equation and the one that appeared in the preceding chapter [IV-1, Eq. (7)], is due to the difference in the choice of misfit strength unit; namely, the strain ϵ was taken as the resultant strain after the relaxation of misfit defect into matrix in Chapter IV. On the other hand, in the above equation ϵ^S is taken as the stress-free strain which would undergo in the absence of matrix. These two parameters are mutually related by

$$\epsilon = \frac{\epsilon^S}{3} \cdot \frac{1+\nu}{1-\nu} \quad (7)$$

For the orthogonal defect, where the values of ϵ_{11}^T , ϵ_{22}^T and ϵ_{33}^T are not the same, Eq. (1) is written by the following expressions

$$U_1^C = \frac{V}{8\pi(1-\nu)r^2} \begin{bmatrix} \epsilon_{11}^T \{ (1-2\nu)\ell_1 + 3\ell_1^3 \} \\ \epsilon_{22}^T \{ (1-2\nu)(-\ell_2) + 3\ell_1\ell_2^2 \} \\ \epsilon_{33}^T \{ (1-2\nu)(-\ell_3) + 3\ell_1\ell_3^2 \} \end{bmatrix} \quad (8-a)$$

$$U_2^C = \frac{V}{8\pi(1-\nu)r^2} \begin{bmatrix} \epsilon_{11}^T \{ (1-2\nu)(-\ell_1) + 3\ell_1^2\ell_2 \} \\ \epsilon_{22}^T \{ (1-2\nu)\ell_2 + 3\ell_2^3 \} \\ \epsilon_{33}^T \{ (1-2\nu)(-\ell_3) + 3\ell_3^2\ell_2 \} \end{bmatrix} \quad (8-b)$$

$$U_3^c = \frac{V}{8\pi(1-\nu)r^2} \begin{bmatrix} \epsilon_{11}^T \{(1-2\nu)(-\ell_1) + 3\ell_3\ell_1^2\} \\ \epsilon_{22}^T \{(1-2\nu)(-\ell_2) + 3\ell_3\ell_2^2\} \\ \epsilon_{33}^T \{(1-2\nu)\ell_3 + 3\ell_3^3\} \end{bmatrix} \quad (8-c)$$

The simplest case of the so-called tetragonal defect is where one of ϵ_{ii}^T is not zero and the other two are zero. Let us consider the case, $\epsilon_{11} \neq 0$ and $\epsilon_{22} = \epsilon_{33} = 0$; Eq. (8) can then be reduced to:

$$U_1^c = \frac{\epsilon_{11}^T V}{8\pi(1-\nu)r^2} \{(1-2\nu)\ell_1 + 3\ell_1^3\} \quad (9-a)$$

$$U_2^c = \frac{\epsilon_{11}^T V}{8\pi(1-\nu)r^2} \{-(1-2\nu)\ell_2 + 3\ell_2\ell_1^2\} \quad (9-b)$$

$$U_3^c = \frac{\epsilon_{11}^T V}{8\pi(1-\nu)r^2} \{-(1-2\nu)\ell_3 + 3\ell_3\ell_1^2\} \quad (9-c)$$

One of the stress components at point (x, y, z) is given by a relation $\sigma_{zx} = \mu \left(\frac{\partial U_3}{\partial x} + \frac{\partial U_1}{\partial z} \right)$, therefore σ_{zx} can be calculated by using Eq. (9) and the result is given by

$$\sigma_{zx} = \frac{V\epsilon_{11}^T \mu}{8\pi(1-\nu)} 6zx \{(1-2\nu)(x^2 + y^2 + z^2) - 5x^2\} (x^2 + y^2 + z^2)^{-7/2} \quad (10)$$

Similarly σ_{zx} for ϵ_{22}^T and ϵ_{33}^T type of tetragonal defect is given by

$$\sigma_{zx} = \frac{V\epsilon_{22}^T \mu}{8\pi(1-\nu)} 6zx \{(1-2\nu)(x^2 + y^2 + z^2) - 5y^2\} (x^2 + y^2 + z^2)^{-7/2} \quad (11)$$

and

$$\sigma_{zx} = \frac{V\epsilon_{33}^T \mu}{8\pi(1-\nu)} 6zx \{(1-2\nu)(x^2 + y^2 + z^2) - 5z^2\} (x^2 + y^2 + z^2)^{-7/2} \quad (12)$$

respectively. As we will see in the following formulation, only the stress component σ_{zx} is required for our interest. Therefore the solution for the other stress component is not given here.

The force exerted on a dislocation can be given by the Peach-Koehler⁽¹⁵⁵⁾ equation,

$$\vec{F} = \begin{vmatrix} \vec{i} & \vec{j} & \vec{k} \\ t_x & t_y & t_z \\ G_x & G_y & G_z \end{vmatrix} \quad (13)$$

where t_x , t_y and t_z represent the three components of the unit vector tangent to the dislocation. G_x , G_y and G_z are given by the following relations,

$$\begin{aligned} G_x &= \sigma_{xx} b_x + \sigma_{xy} b_y + \sigma_{xz} b_z \\ G_y &= \sigma_{yx} b_x + \sigma_{yy} b_y + \sigma_{yz} b_z \\ G_z &= \sigma_{zx} b_x + \sigma_{zy} b_y + \sigma_{zz} b_z \end{aligned} \quad (14)$$

where b_x , b_y and b_z are the three components of the Burgers vector. In the preceding calculations t_z is taken to be zero and the Burgers vector is taken along the x-axis. Equation (13) reduces to

$$\vec{F} = \begin{vmatrix} \vec{i} & \vec{j} & \vec{k} \\ t_x & t_y & 0 \\ G_x & G_y & G_z \end{vmatrix} = t_y G_z \vec{i} - t_x G_z \vec{j} = \sigma_{zx} b_x (t_y \vec{i} - t_x \vec{j}) \quad (15)$$

Thus the stress component σ_{zx} is the only term that appeared in the expression.

C. Scattering of electrons by a window and correction for the electron dose

The distribution of the electrons which have undergone multiple scattering has been found to be written by a Gaussian⁽¹⁵⁶⁾ relation,

$$f(\Theta) = \exp[- \Theta^2 / \Theta_w^2] \quad (1)$$

where Θ_w is the angle at which the distribution has a value $1/e$ of its maximum value. The best fit to the experimental data has been found⁽¹⁴⁵⁾ with

$$\Theta_w^2 = 0.157 \frac{x}{(pc\beta)^2} \frac{z(z+1)}{A} (B - 1.2) \quad (2)$$

by using the Moliere theory. Here Θ_w is in radians, x is the sample thickness in g/cm^2 . The other symbols are:

p = momentum

c = velocity of light

β = velocity of electron/velocity of light

z = atomic number

A = atomic mass number

B is defined by

$$\frac{e}{B} = \frac{7800(z+1)z^{1/3}}{A(1 + 3.33\alpha^2/\beta^2)} \frac{x}{\beta^2} \quad (3)$$

with $\alpha = z/137$. From Eqs. (2) and (3), Θ_w was calculated to be $\sim 20^\circ$ for 1 mil thick iron and $\sim 2^\circ$ for 10 mil thick beryllium at the energy of 1.3 MeV.

The loss of electrons due to the above-mentioned scattering was then calculated by approximating the distribution function as $f(r) = \exp(-r^2/r_o^2)$ where the scattering angle Θ is replaced by a distance r which gives the

deviation from the incident beam direction as shown in Fig. 47. Since the angle concerned here is so small, the above approximation can be safely applied to calculating the electron density in the target.

The ratio of the total electron density coming into the target to that of the collimated beam density, is used here for representing the effect of electron scattering. Since both the collimator size and that of a specimen were elongated to one direction the ratio defined above is expected to be close to unity, and the scattering effect was not concerned along this direction. In order to obtain the ratio the electron density distribution along a specimen width direction (along the x-axis) was computed by using the above-mentioned distribution function, $\exp(-r^2/r_o^2)$. The peak height, h , for electrons coming through a differential area, $dx'dy'$, in a window can be given by

$$N_o dx'dy' = \int_0^\infty h 2\pi r f(r) dr \quad (4)$$

where N_o is the density of the collimated beam. Equation (4) gives

$$h = \frac{N_o}{\pi r_o} dx'dy' \quad (5)$$

where

$$r_o = d \cdot \tan \theta_w \quad (6)$$

Here, d is the distance between a window and a target, and θ_w is the scattering parameter described in the beginning of this section. The density $F(x)$ is then given by

$$F(x) = \int_{-D/2}^{D/2} \int_{-C/2}^{C/2} \frac{N_o}{\pi r_o} e^{-r^2/r_o^2} dx'dy' \quad (7)$$

where r is given by

$$r^2 = (x' - x)^2 + y'^2 \quad (8)$$

Then the ratio, R, can be given by

$$R = \frac{\int_{-A/2}^{A/2} F(x) dx}{\int_{-A/2}^{A/2} N_0 dx} \quad (9)$$

By substituting Eq. (7) into Eq. (9), and also substituting for r from Eq. (8),

$$R = \frac{1}{\pi r_0 A} \int_{-A/2}^{A/2} \int_{-D/2}^{D/2} \int_{-C/2}^{C/2} e^{-\{(x'-x)^2 + y'^2\}} dx' dy' dx \quad (10)$$

The integration of the above equation was carried out by using a computer and the results are shown as a function of distance, d, by solid lines in Fig. 48 for several scattering angles. The constants were taken from the experimental set-up used here and given by

$$A = 1.3 \text{ mm}$$

$$C = 3.0 \text{ mm}$$

$$D = 11.7 \text{ mm}$$

If the distribution function is approximated by a linear function instead of a Gaussian, and the same procedure as above is followed, R is given by the following equation,

$$R = \frac{3}{\pi r_m A} \int_{-A/2}^{A/2} \int_{-D/2}^{D/2} \int_{-C/2}^{C/2} \left(r_m - \sqrt{(x'-x)^2 + y'^2} \right) dx' dy' dx \quad (11)$$

where r_m is given by $\frac{e}{e-1} r_0$, and the integrand is zero for $r \geq r_m$. The results of the above integration are also shown by broken lines in Fig. 48.

As seen in this figure the choice of distribution function did not affect the value of R significantly, indicating that the slight modification of the Gaussian function should give a reliable estimate. Thus, the correction factor for the electron dose was determined to be 0.7 for a 1 mil thick window for $d = 7 \text{ mm}$ which was the practical distance for all the tensile test experiments.

Fig. 48 Effective fraction of electrons hitting a target is plotted against the distance between a target and a window. Solid lines for a Gaussian distribution and broken lines for a corn shaped distribution. The parameter θ_w was determined by the Moliere theory, (145) and is a function of the atomic weight, the window thickness, and the electron energy as given in the text [cf. Eq. (2)].

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XIII. SUMMARY AND CONCLUSION

The effect of electron irradiation on the strength of b.c.c. iron was investigated at temperatures between 4.2°K and 313°K. The strength of a specimen was tested in both the micro- and macrostrain regions by means of tensile deformation. During the course of the present investigation special care was taken to minimize the complication due to residual impurities, dislocations and sub-boundaries. Clean single crystals of an approximately 10 mil thickness were prepared from the ultrapure iron supplied by the Battelle Memorial Institute and the specimens were subsequently purified in the ZrH_2 system under vibrational stress. These single crystal specimens demonstrated remarkable plasticity at low temperatures (down to 4.2°K with prestrain at higher temperature and down to 30°K without prestrain). Single crystals of less purity were also used to examine the impurity effect in this investigation.

The incident electron energy of 1.3 MeV was used for the irradiation. The total electron dosage used in this investigation ranged from 5×10^{16} to 2×10^{18} e/cm². The irradiation temperature was controlled below 60°K to prevent the main recovery of the radiation effect which occurred above 80°K.

The important findings in the present experiments are summarized as follows:

- 1) Electron irradiation caused a softening effect for the macroscopic flow stress at 60°K to the electron dose as high as 2×10^{18} e/cm². No effect was observed when the tensile test was performed at 4.2°K.

- 2) The irradiation with a high electron dose appeared to promote twinning. A similar effect was also caused by the presence of impurities.
- 3) Irradiation followed by aging at higher temperatures caused a hardening effect. The hardening appeared to be athermal in the temperature range of $60^{\circ}\text{K} \sim 313^{\circ}\text{K}$. The appearance of twinning and ductile fracture was promoted when the specimen of this treatment was tested at 60°K .
- 4) In the microyield region, a hardening effect was observed in the as-irradiated specimen at low temperatures (4.2°K and 60°K), in contrast to the softening effect for the macroscopic flow.
- 5) The hardening in the microyield region was significantly enhanced by lowering the test temperature as well as by aging at a higher temperature.
- 6) Besides the above observations, remarkable improvement in the ductility of the ultrapure iron single crystals at low temperatures was achieved by the present purification treatment.

In order to examine the possible effects of interstitials on the dislocation motion in a relatively high Peierls potential field, a calculation was carried out, assuming a model which included the effect of the Peierls potential and the elastic stress field of misfit strain centers which represented the impurities. The results of this calculation successfully explained all the experimental observations: the introduction of dispersed interstitials caused the reduction in the double kink nucleation energy; at the same time, it produced a resistance to the motion of kinks. The combination of these two effects explained the softening effect at relatively

low temperatures and the hardening effect at higher temperatures. This model also explained the athermal hardening for stronger obstacles. The temperature dependent hardening effect observed in the microyield region was explained by taking into account the interaction of edge dislocations with the dispersed obstacles.

The present experimental results and the calculations clarify the importance of the inherent lattice resistance in pure iron and perhaps other b.c.c. metals. The findings in the present investigation can be inferred to a solid solution case; the presence of dilute impurities can give a softening effect and the impurity hardening can be expected above a critical concentration and/or at high temperatures.

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