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DISLOCATION-SUBSTRUCTURE INTERACTIONS
IN IRRADIATED IRON AND STEEL

June 1969

AEC RESEARCH &
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BNWL-1045

UC-25, Metals, Ceramics,
and Materials

DISLOCATION-SUBSTRUCTURE INTERACTIONS
IN IRRADIATED IRON AND STEEL

By

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June 1969

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RICHLAND, WASHINGTON 99352

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BNWL-1045

Printed in the United States of America
Available from
Clearinghouse for Federal Scientific and Technical Information
National Bureau of Standards, U.S. Department of Commerce
Springfield, Virginia 22151
Price: Printed Copy \$3.00; Microfiche \$0.65

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F. A. Smidt, Jr.

ABSTRACT

The influence of radiation damage upon the mechanical properties of iron and steel are analyzed in terms of the substructure of defect aggregates produced by irradiation, the interaction of dislocations with the substructure, and the implications of these interactions on the macroscopic mechanical properties.

The literature pertinent to damage production and recovery is reviewed to evaluate several models for the defect aggregate. The displacement spike is the most important defect production event in neutron irradiation and leads to the formation of small vacancy aggregates or depleted zones and interstitial aggregates which grow by the migration of free interstitials to eventually reach a size and configuration identified by transmission electron microscopy as a prismatic loop. Impurities are shown to be important in the nucleation and growth of defect aggregates. Irradiation temperature is also shown to strongly influence the final aggregation state of the defects produced by other irradiation.

An analysis of the most probable dislocation-defect interactions leads to the conclusion that the defects, while not influencing the short range forces controlling thermally activated processes in bcc metals, do interact strongly as long range forces affecting the athermal processes. An estimate of the increase in strength from observed defect sizes and densities indicates the increase in strength approaches that predicted by the Orowan theory of precipitation hardening. Transmission electron microscopy observations, however, have shown that the defects are destroyed by moving dislocations.

The implications of the dislocation-substructure interactions observed in irradiated iron and steel are considered in terms of their influence on the technologically important properties of work-hardening, ductility, and embrittlement. Evidence for restricted cross-slip and localized deformation in irradiated and deformed iron are presented. These effects are shown to lead to the decrease in work-hardening and ductility commonly observed. Certain embrittlement effects are also shown to be predictable from the dislocation-defect interactions.

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DISLOCATION-SUBSTRUCTURE INTERACTIONS IN IRRADIATED IRON AND STEEL

F. A. Smidt, Jr.

I. INTRODUCTION

The deleterious effects of neutron bombardment on the mechanical properties of structural materials is a problem prompting expenditure of a great deal of effort in understanding how and why it happens, in estimating how long a component can be used safely, in developing new and better materials, and in resolving a host of other closely related problems. Clearly the key to a major advance in materials technology in the nuclear field is an understanding of the mechanism of radiation damage and its effect on mechanical properties.

Much of the content herein was written from research conducted in preparation for the U.S.-Japan Symposium on Radiation Damage in Iron and Steel, held in San Francisco, California, June 23-25, 1968 under the sponsorship of the National Science Foundation. The author is most appreciative of the stimulus provided by this meeting to review the various papers bearing on dislocation-substructure interactions, and of the opportunity to discuss this subject with his colleagues both from the U.S. and Japan.

II. SUMMARY

Studies of the interaction between dislocations and the defect substructure are of considerable interest because they yield fundamental information about the state of defect aggregation and also provide the basis for extrapolating mechanical properties to ranges of temperature or fluence of technological importance. Substructure as considered in this paper will include existing dislocations and precipitates which may be modified during irradiation at ambient temperatures, as well as the defect aggregates produced by the irradiation.

Pertinent experiments in the literature contributing to the present understanding of dislocation-defect interactions are reviewed. Thermally activated flow experiments on irradiated bcc iron have shown no change in the low temperature deformation mechanism. Consequently, it can be inferred that short range interactions between dislocations and defects with interaction energies less than about 3 eV are not observed. Minor changes in the activation volume at 295 °K, sometimes observed in the initial stages of deformation after neutron irradiation, are shown to be related to the removal of impurity atoms in the Cottrell atmosphere by defect trapping.

The large increase in the athermal component of the flow stress is shown to be the major effect of radiation damage in iron irradiated at 60 °C. Possible hardening mechanisms are reviewed and it is shown that the Orowan mechanism of precipitation hardening or junction reactions with loops are the only mechanisms capable of producing the observed magnitude of hardening.

These observations of dislocation-defect interactions are compared with damage models developed from other techniques such as direct observation by transmission electron microscopy and recovery experiments on low temperature electron and neutron irradiations. It is concluded that (1) the basic damage event is the displacement spike, (2) the most reasonable value for the migration energy for the interstitial point defects is 0.35 eV and for the vacancy about 1.00 eV, (3) impurity trapping of point defects affect the final defect aggregate, and (4) the most important defect aggregate in ambient temperature irradiations is either the small, three-dimensional cluster of interstitial atoms or the interstitial prismatic loop.

Studies of mechanical properties after both cryogenic irradiations and 400 °C irradiations are shown to be consistent with this model of defect aggregate, nucleation, growth, and

dissociation. The observation of saturation and subsequent softening in low carbon iron irradiated to fluences in excess of 10^{20} n/cm² is discussed and a mechanism based on resolution of small precipitates in displacement spikes is suggested as a possible explanation.

III. OVERVIEW OF PROBLEM

The complexity of the problem and the incomplete understanding of the mechanisms of work-hardening and fracture indicate progress will be slow and steady rather than as the result of a major breakthrough. The fact that the mechanical properties of an irradiated metal were determined primarily by the dislocation-defect interactions was recognized at a fairly early stage. Accordingly, this paper is organized around the conceptual theme that such interactions are the common denominator for understanding changes occurring in the mechanical properties. Thus, the problem breaks up logically into (1) the state of aggregation of the defects produced under various environmental conditions, (2) the interaction of dislocations with the defects, and (3) the manifestation of these microscopic events in the macroscopic properties. The first topic is broadened to include modification of the pre-irradiation microstructure by the neutron environment, as well as the production of lattice defects by the irradiation and, as such, is designated substructure. The important initial survey experiments are reviewed on a phenomenological basis to emphasize the predominant features of radiation damage.

The need for an understanding of radiation damage and a recognition of its importance dates back to 1946 when Wigner⁽¹⁾ first suggested the possibility of damage to metals in a nuclear reactor. The pioneering experiments on radiation damage in metals were done on Cu by the Oak Ridge group, and the first paper in a comprehensive series, by Redman, Coltman and Blewitt,⁽²⁾ was published in 1953. The following year,

Kunz and Holden⁽³⁾ reported the first work on iron. They had irradiated iron single crystals to a thermal fluence of 10^{18} n/cm² and observed both the increase in yield strength and decrease in work-hardening coefficient now considered characteristic features of radiation damage. They also had annealed several crystals in the range 225-500 °C and observed that recovery occurred around 300 °C with an activation energy considerably above that expected for the migration of point defects. Also interesting to note was their suggestion of the interstitial cluster as the most likely defect aggregate responsible for damage effects observed.

Bruch, McHugh, and Hockenbury⁽⁴⁾ were the next to study iron as one of several materials in a survey experiment. With the attainment of higher fluences (estimated at $2-7 \times 10^{20}$ n/cm², thermal), they observed the decrease in ductility now considered to be another characteristic of radiation damage. They also observed the development of a drop-in-load yield point, although some of their control specimens showed an upper and lower yield point.

Churchman, Mogford, and Cottrell,⁽⁵⁾ and Hull and Mogford⁽⁶⁾ applied the Petch test to the study of hardening and embrittlement in steel. They noted the shift in ductile to brittle transition temperature, another characteristic feature of radiation damage. They found that grain size had very little effect on the yield or fracture stress and concluded that radiation hardening was caused by "dispersed regions of radiation damage, such as clusters of point defects."

Subsequent experiments have documented these early results and added to our knowledge of the details of the process of radiation damage. The numerous contributions to the field since these early experiments have been adequately reviewed elsewhere.⁽⁷⁻¹²⁾

Problems receiving recent attention have centered around identification of the defect aggregate, or aggregates responsible for radiation damage, studies of the mechanism of their production, migration, and stability, elucidation of possible changes in, or interactions with, the metallurgical parameters of iron and steel, and resolution of specific dislocation-defect interactions. Naturally, the solution of these problems requires a comprehensive utilization of many experimental techniques. If any one technique were to be singled out for special emphasis, it would be transmission electron microscopy. The development of this technique in 1959 has permitted the direct observation of defect aggregates present in the metal after irradiation. The first such observation on iron, made in 1962 by B. L. Eyre,⁽¹³⁾ confirmed the earlier hypothesis that clustered defects were formed during irradiation.

IV. NATURE OF THE SUBSTRUCTURE

The interpretation of radiation effects in iron and steel is dependent upon a knowledge of the nature of the substructure produced by neutron irradiation, and it is in this area that the complexity of the problem originates. Basically, the three general areas to be considered are (1) the basic damage production process as it would occur at 0 °K, (2) the modification of this damage state by dynamic recovery, and (3) the interaction of the products of the damage event with the existing metallurgical substructure to produce the observed damage configuration at a given irradiation temperature.

A. DAMAGE PRODUCTION

Theoretical and experimental studies of damage production have been considered in detail elsewhere.⁽¹⁴⁻²¹⁾ A summary of the principal concepts are given here as a foundation for subsequent discussions of damage recovery. The transfer of kinetic energy from an incident particle such as an electron

to a lattice atom can displace the atom from its lattice site if the energy exceeds the threshold energy for displacement. The vacancy-interstitial pair created by this event does not recombine where separation by a few interatomic spacings occurs. Higher energy knock-ons produced, for instance, by neutrons are capable of producing more severe damage in which many atoms are displaced from a region in the lattice and driven outward. Dynamic rearrangement eliminates many of the displaced atoms and they return to normal lattice sites as the disturbed region collapses. However, a few of the displaced atoms are focused along preferred crystallographic directions and travel far enough from their initial sites to become independent of their point of origin. When the disturbed region collapses, as illustrated in Figure 1, an excess of vacancies is present in the interior of the region and an excess of interstitials surrounds the damaged site. This concept was first suggested by Seeger⁽¹⁴⁾ and termed the depleted zone. Brinkman⁽¹⁵⁾ has contributed to an understanding of the production of such depleted zones with the now generally accepted displacement spike model. Evidence for the existence of displacement spikes has been found in the disorder produced in Cu_3Au by neutrons and electrons, and in the difference in the recovery spectra of electron and neutron bombarded materials.⁽¹⁵⁾ Beeler⁽²¹⁾ has simulated damage production in iron by computer modeling. The reproduction of the displacement spike provides further evidence supporting the validity of the displacement spike concept.

B. DEFECT AGGREGATION AND ANNEALING

The most obvious conclusion to be drawn from a study of defect aggregation and damage recovery is its extreme complexity and the need for additional investigation. The interpretation in regard to the bcc metals, and iron in particular, must be regarded as tentative in view of the large amount of work done on copper and the remaining unresolved questions concerning

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FIGURE 1. Depleted Zone as Visualized by Seeger and Essman, Reference 9, Showing Damage Production Processes

that metal. Specific experiments with a minimum number of variables are necessary to make interpretation of the recovery processes easier. It should prove helpful, for example, to irradiate the material under consideration at low enough temperatures so that point defects are not mobile. Then, upon annealing the sample at progressively higher temperatures, the migration processes by which defects move to sinks and aggregate

can be studied in more detail. To a first approximation, this effort should reveal the processes occurring at a much faster rate during irradiation at ambient reactor temperature. Because the defect concentrations may be radically different, this technique should be employed only to provide approximations. Another technique useful in reducing the complexity of the experiment is to limit the damage production to simple Frenkel pairs by electron irradiation.

The interpretation of damage recovery is also dependent upon the specificity of the property measured. Resistivity, for example, is a measure of electron scattering by imperfections in the lattice and is most sensitive to individual vacancies and interstitials, and relatively insensitive to clustered defects. Stored energy release is especially useful for establishing the occurrence of mutual annihilation because the energy of this process is well defined. It is also useful in studying trapping of point defects.

Internal friction and magnetic after-effect measurements are specific to defects with a symmetry lower than cubic, such as the split interstitial. In internal friction experiments, the change in the distribution of orientations of these defects between the stressed and unstressed condition provides a sensitive indication of their presence. Magnetic after-effect measurements similarly are sensitive to reorientation of a low symmetry defect in the domain wall of a ferromagnetic material after demagnetizing the material. A more detailed discussion of this matter can be found in Reference 22.

Transmission electron microscopy is, of course, extremely useful in studying defects and defect aggregates possessing a strain field rendering them visible under suitable diffraction conditions. This technique is limited to observation of defects greater than about 20 Å in size. While resolution may be too

low to resolve Burger's vectors of loops below 150-200 Å in size, vacancy and interstitial character of loops above this size may be determined, and planar defects can be differentiated from three-dimensional defects in the larger sizes. Field ion microscopy should be a valuable addition to the techniques available for resolving defects of atomic dimensions. No work, however, has yet been published on iron.

Hardness recovery has frequently been used in damage recovery studies, particularly at room temperature and above. Its usefulness, however, is limited by the knowledge of its functional relationship with defect substructure, the subject of this paper. The interpretation of hardness and yield strength measurements and their correlation with physical properties will be additionally discussed in a following section.

Length change-lattice parameter measurements, also useful as a technique in determining the relative concentrations of vacancies and interstitials, have been used very little in studies of iron.

The complexity of the problem requires a careful study of evidence gained from all the previously referenced techniques to build up a model of damage recovery consistent with all evidence available. Perhaps the mistake most frequently made in the field is to place too much reliance on the results of a single experimental technique. For this reason, complimentary techniques presenting a more comprehensive picture of recovery processes are summarized in Table 1.

The first major recovery process in iron after irradiation in the range 4-20 °K occurs around 100 °K. This recovery process, commonly designated Stage I by analogy with the terminology evolved from studies of resistivity recovery in copper, will

TABLE 1. Damage Recovery Processes in Irradiated Iron.

	Temperature of Recovery Peak, °K	Technique*	Probable Mechanism
<u>Stage I (0-130 °K)</u>			
IA	63	ρ , mag.	Close-pair Recomb, Reorient di-i
IB	108	ρ , S.E., mag.	i Migration, Reorient di-i
IC	~120	ρ , mag.	Ni-i Trap, Reorient di-i
<u>Stage II (130-273 °K)</u>			
IIA	152	ρ	Crowdion Recovery
IIB	190	ρ , S.E., mag.	di-i Migration
IIC and IID	207-273	ρ	Impurity Traps
	210	S.E., mag.	{ Migration of Larger i Aggregates
	250	S.E., mag.	
	240	ρ	Dissoc. C-i Complex
<u>Stage III (273-373 °K)</u>			
	293	ρ	{ Impurity Effects - Si, Sb, Al
	323	ρ	
	348	ρ , S.E.	
	323	ρ , I.F., S.E.	C Migration to v Trap
	268		Reorient - v Complexes
	320	mag.	Reorient - v - Impurity Complex
	366		Reorient - v - Impurity Complex (Anneal out 300-410 °K)
<u>Stage IV (373-773 °K)</u>			
	423	ρ	Pct'n ϵ Carbide
	538	ρ	Dissoc. C-v Complex
	573-773	ρ , H, E.M.	Dissoc. i Clusters or Loop Growth

* ρ -resistivity; mag.-magnetic disaccommodation; S.E.-stored energy; I.F.-internal friction; H-hardness; E.M.-transmission electron microscopy.

include, in this report, the 0-130 °K range. Stage I is important to our understanding of radiation damage because it represents the first rearrangement of defects by migration after the damage event. If the damage production process is assumed to produce displacement spikes and Frenkel pairs, then this first recovery stage should provide information about motion of the most mobile species, the interstitial. Correlated motion with vacancies, interaction with traps in the vicinity of the interstitial, and clustering of multi-interstitial aggregates might be expected to result in several closely related recovery processes. Electron irradiations with only pair production should be somewhat simpler.

Lucasson and Walker⁽²³⁾ conducted electron irradiations of iron at 20 °K and studied damage recovery in Stage I. As shown in Figure 2, they noted several substages at 60 °K and

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FIGURE 2. Resistivity Recovery Curves for Several Metals Irradiated with Electrons at 20.4 °K. Samples Subsequently Pulse Annealed for 10 min at Specified Temperatures and Cooled to 20.4 °K for Measurement. (From Reference 23)

70 °K, with the largest recovery occurring between 80 and 120 °K. These stages accounted for about 75% of the recovery in resistivity. The first two substages were attributed to close-pair recombination, and the larger one to interstitial migration.

Some very extensive and comprehensive work on iron performed at Centre d'Etudes Nucleaires at Grenoble since about 1960 has included contributions by many individuals and is described in numerous papers over the intervening years. The work seems to be fairly well summarized in two recent documents representing the present concepts of this group in regard to damage recovery. Only these two papers are referenced inasmuch as the original contributions are adequately referenced in these reviews. Minier-Cassayre⁽²⁴⁾ has done electron irradiations at 20 °K and has studied the resistivity recovery. A recovery stage observed at 63 °K accounted for 11% of the total increase, and a peak between 75 and 139 °K accounted for 75% of the increase. The magnitude of these peaks varied with energy of the incident electrons, the first one decreasing and the second one increasing as a function of electron energy. An activation energy of 0.25 eV was derived from isothermal annealing studies of the peak at 110 °K. Neutron irradiations were also conducted at 30 °K and 80 °K to fast fluences up to 4.5×10^{18} n/cm² ($E > 1$ MeV). The results from the 80 °K irradiation shown in Figure 3 have a shoulder on the upper side of peak IB and indicate a simultaneous recovery process. The lower peak, IA, of course, was not observed after the 80 °K irradiation, but was present after the 30 °K irradiation in diminished size as compared to the electron irradiations. The recovery peaks in iron doped with up to 0.74% Ni and irradiated to a fast fluence of 8×10^{17} n/cm² (Figure 4) can be compared with those in Figure 3 and show a decrease in the height of peak IB, while peak IC is clearly resolvable. Stage IA at 63 °K, with an activation

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FIGURE 3. Derivative of Resistivity Recovery Curves for Iron Irradiated to Fast Fluence of $8 \times 10^{17} \text{ n/cm}^2$ ($E > 1 \text{ MeV}$) at a Temperature of 80°K . (From Reference 24)

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FIGURE 4. Derivative of Resistivity Recovery Curves for Iron Doped with 0.74% Ni and Irradiated to Fast Fluence of $8 \times 10^{17} \text{ n/cm}^2$ ($E > 1 \text{ MeV}$) at 80°K . (From Reference 24)

energy of 0.19 eV, was attributed to close-pair recombination, and Stage IB, centered at about 108 °K, is attributed to migration of free interstitials to vacancies with subsequent annihilation, to impurity traps, and to other interstitials with the formation of di-interstitials. A migration energy of 0.25 eV is associated with this recovery process. Stage IC is attributed to release of interstitials from a Ni impurity trap. The binding energy was estimated to be 0.05 eV.

Iron has also been irradiated at Oak Ridge⁽²⁵⁾ at 14 °K to a fluence of 4×10^{17} n/cm². These results also show a major recovery over the range 60-140 °K, but did not resolve the fine structure observed by the French group. G. Burger et al.⁽²⁶⁾ have also studied resistivity recovery in iron irradiated at 4.5 °K to fluences of 7.2×10^{17} n/cm² ($E > 0.1$ MeV) and have observed the same recovery stages as the French group.

More specific information about the processes occurring during the Stage I recovery is obtained from stored energy release experiments reported by Moser.⁽²⁷⁾ A stored energy release peak at 110 °K in a sample irradiated to a fluence of 10^{18} n/cm² at 77 °K corresponded to annihilation of roughly half the calculated number of interstitials produced. It was suggested that the remainder were eliminated by close-pair recombination or association into di-interstitials. Measurements of magnetic after-effect⁽²⁷⁾ reveal additional details of the recovery process. Moser⁽²⁷⁾ interprets measurements in the region of Stage I to be composed of two processes, free interstitial migration with an activation energy of 0.23 eV, and di-interstitial migration with a migration energy of 0.35 eV. The symmetry of these defects is different and so their reorientation can be distinguished. In addition, the concentration of di-interstitials is observed to increase during migration of the single interstitial. About 75% of the total concentration of di-interstitials arises from the initial damage

production during irradiation and 25% from association during migration of the single interstitial.

The migration of di-interstitials actually lies above the region designated Stage I in terms of resistivity recovery, although reorientations of the di-interstitial occur in Stage I and apparently contribute to the resistivity recovery peaks at 63 °K (IA) and 110 °K (IB). The reorientation of the di-interstitial occurs in three substages, with peaks at 60 °K, 90 °K, and 123 °K.

Balthesen et al.⁽²⁸⁾ have also studied magnetic after-effects in iron irradiated at 30 °K. They report more fine structure with reorientation peaks at 15, 57.5, 69, 85, 100, 101, 121, 126 and 143.5 °K, most of which are grouped around the three peaks described by Moser. They did not attribute any of their magnetic after-effect to migration of defects as did Moser. The analysis of magnetic after-effect data is still in the developmental stage, and additional work seems necessary to clarify the points of disagreement between various investigators.

Perhaps more distressing is the lack of agreement between magnetic after-effect measurements and computer calculations of defect symmetries and migration energies. A migration energy of 0.33 eV calculated by R. A. Johnson⁽²⁹⁾ for the interstitial is only in fair agreement with the Grenoble results of 0.25 eV, while the symmetry of the defect from calculation and experiment do not agree. Furthermore, Johnson calculated a lower migration energy for the di-interstitial than for the single interstitial, 0.18 eV, while the experiments show a higher energy 0.35 eV and, again, the symmetries do not agree. However, in spite of these discrepancies, it is quite clear that long-range migration of interstitials occurs in Stage I around 110 °K with a migration energy of about 0.3 eV.

Following the nomenclature of Minier-Cassayre, Stage II recovery extends over the range from 130 °K to about 273 °K (Figure 3). Four peaks identified in the recovery of electrical resistivity are IIA (152 °K), IIB (190 °K), and IIC and IID (207-273 °K).⁽²⁴⁾ Burger et al.⁽²⁶⁾ resolved a peak at about 160 °K and 190 °K. The Oak Ridge⁽²⁵⁾ results also show recovery in this range, but do not resolve the peaks.

Stage IIA, not detectable by magnetic after-effect measurements, must therefore be either symmetrical or possess a high reorientation energy. This stage represents 16% of the increase in resistivity after neutron bombardment, 6% after bombardment with 2.9 MeV electrons, and is absent after bombardment with 1.2 MeV electrons. Impurities do not seem to significantly affect the peak height. Minier-Cassayre attributes this stage to recovery of crowdions and assigns to it an energy of 0.38 eV.

Stage IIB, which covers the range 170-210 °K, is observed in both electron and neutron irradiations. The magnitude of Stage IIB is greater in neutron irradiations, increases with fluence, and decreases with impurity content. The energy of the recovery process is about 0.5 eV and the kinetics are complex. A stored energy release is observed in this region as well as a magnetic after-effect band extending from 150-200 °K. Moser⁽²⁷⁾ interprets this band as due to migration of di-interstitials with a migration energy of 0.35 eV. The concentration of this species is higher in neutron bombardments and about 25% of the total forms during Stage IB. The experimental evidence seems consistent but, as previously noted, does not agree with the symmetry and migration energies calculated by Johnson.

Stages IIC and IID occurring over the range 207-273 °K, although poorly defined in pure iron, were greatly enhanced in iron doped with Ni and Co. The peak temperature seems to be

dependent on the specific impurity. One of these peaks probably corresponds to the resistivity recovery stage observed by Damask⁽³⁰⁾ at 243 °K and has been attributed to the dissociation of a carbon-interstitial complex.

Several additional recovery processes observed in the Stage II recovery range apparently do not influence the resistivity. Moser⁽²⁷⁾ reports that a magnetic after-effect band in this region liberating stored energy in two broad peaks at around 210 °K and 250 °K could not be produced by quenching. These processes were highly complex and were attributed to reorientations of aggregates of interstitials at around 183 and 233 °K, and with reorientation energies of 0.55 and 0.7 eV, respectively. They annealed out over the range 235-400 °K. In view of the stored energy release peaks, this stage rather surprisingly was not reflected in the resistivity recovery curves, although details of the process might have been obscured by Stage IIB.

The obvious complexity of the recovery processes in iron making designation of distinct stages rather arbitrary is especially true of Stages III and IV. For the sake of discussion, Stage III will be considered to extend over the range 273-373 °K, and Stage IV will cover the 373-773 °K range.

Minier-Cassayre⁽²⁴⁾ detected several poorly characterized recovery stages in the 273-473 °K range which were strongly dependent upon impurity content. A peak was present in pure iron at about 75 °C (325 °K) after neutron irradiation at low temperature. Doping with silicon not only produced another more prominent peak at 20 °C but increased and shifted the 75 °C peak to about 62 °C. Antimony and aluminum doped material showed a peak at 50 °C, while Ni, Co, and Pt had little effect on the recovery spectra in this temperature range.

Damask and his co-workers⁽³⁰⁻³⁴⁾ very carefully examined this region by internal friction, stored energy, resistivity, and electron microscopy. The data from a resistivity recovery curve are shown in Figure 5. They observed that precipitation of carbon during an anneal of a quenched sample occurred in two stages. The first, at 170 °C, was attributed to precipitation of metastable ϵ -carbide and the second, at 250 °C, corresponded to the formation of Fe_3C . After irradiation, peaks were observed at -30 °C (as previously mentioned), at 50, 150, 170, 265, and 300 °C. The 50 °C peak corresponding to the decay of the Snoek peak had a migration energy of 0.87 eV, and was

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FIGURE 5. Resistivity Recovery Stages in Iron Specimens Containing Carbon in Solution, Before and After Neutron Irradiation at 175 °K. (From Reference 30)

accompanied by a stored energy release. It was attributed to trapping of a vacancy by a moving carbon atom.

The 150-170 °C stage was assigned to precipitation of carbon in excess of that trapped by the vacancies. The 265 °C step was attributed to dissociation of the carbon-vacancy trap with precipitation of Fe_3C . Stored energy measurements showing 0.41 eV as the binding energy to the vacancy compares with 0.27 eV per carbon atom in ϵ -carbide, and with 0.52 to 0.89 eV for Fe_3C . The higher binding energy in the vacancy complex should suppress precipitation of ϵ -carbide, a fact confirmed by electron microscopy. The stage at 300 °C was tentatively attributed to vacancy migration on the basis of the observation of rapid growth of precipitates. This rapid growth could result from strain relief due to vacancy migration to the precipitates.

Moser⁽²⁷⁾ reported a small release of stored energy at around 75 °C, and also noted several magnetic after-effect bands in this range. One band with reorientation effects at 268 °K, 320 °K (47 °C), and 366 °K (93 °C) disappeared in the range 300-410 °K after low temperature irradiations and also after fast quenches, but not after irradiation at 20 °C. The peaks at 320 and 370 °K were affected by impurity content, while the one at 268 °K appeared to be an intrinsic effect. The latter peak had the same characteristics as one appearing after a 30 min anneal in the range 600-1000 °K, following a low temperature irradiation. Although similar to reorientation effects from carbon, the 268° peak occurred at a lower temperature, was not observed in unirradiated iron, increased in amplitude with exposure and, in contrast to carbon which should disappear after an anneal at 300 °C, the amplitude of the peak increased after the anneal. Moser attributed the 268 °K peak to reorientation of 2, 3, and 4 vacancy complexes, and the higher peaks to reorientations of vacancy-impurity complexes.

These effects annealed out when the complex became larger than quadri-vacancy.

Moser's observation of vacancy aggregates reorienting and growing in size by vacancy-vacancy reactions is difficult to reconcile with other knowledge of vacancy motion in bcc materials. Schultz⁽³⁵⁾ found the migration energy for vacancies in tungsten to be 1.93 ± 0.3 eV by quenching experiments, compared to a self-diffusion energy of 5.23 eV, or $E_m^V = 0.37 E_{s.d.}$. Meakin, Lawley, and Koo,⁽³⁶⁾ in quenching experiments on molybdenum, found the formation energy to be $E_f^V = 2.5$ eV and the migration energy by difference to be $E_m^V = 1.5$ eV. An estimate of the migration energy of vacancies in iron can be made from the ratio of $E_m^V/E_{s.d.}$ in molybdenum which, for the preceding data, is 0.37. For a self-diffusion value of 2.6 eV for iron,⁽³⁷⁾ an E_m^V in iron might be expected to be in the range of about 0.91 to 1.04 eV.

R. A. Johnson's⁽²⁹⁾ computer calculations of vacancy migration in iron gave a value of 0.68 eV and indicated that di-vacancies and tri-vacancies should have about the same migration energy. Beeler⁽³⁸⁾ has suggested a value of around 0.8 eV or higher as more realistic. This value is also consistent with Damask's experiments in which carbon was observed to disappear from solution at 50 °C with an activation energy of 0.86 eV, thus indicating the vacancy migration energy must be 0.86 eV or higher.

At first glance, Moser's interpretation of the observed reorientation effects as attributable to vacancy aggregates is difficult to accept because the quenched vacancies are destroyed as the metal cools through the phase transformations. It is difficult to conceive of an impurity which would go into solution upon quenching or irradiation, increase in solubility at 300 °C instead of precipitating, and appear after an anneal of

irradiated specimens in the range 325 to 725 °C. To reconcile the evidence, it must be concluded that if the observed magnetic after-effects were due to vacancy aggregates, they were formed during irradiation or during the quench and the observed effects were due only to reorientations and not migration. Long-range migration of vacancies, di-vacancies, and tri-vacancies in excess of the trap concentration would be expected in the range between 50 and 300 °C with an activation energy of about 1.0 eV.

Eyre and Bartlett,⁽³⁹⁾ Bryner,⁽⁴⁰⁾ and Smidt⁽⁴¹⁾ have studied the radiation damage produced in iron irradiated at ambient reactor temperatures by transmission electron microscopy. The damage observed for irradiation at temperatures between 40 and 80 °C might be expected to be the product of long-range interstitial migration or vacancy clusters formed in the displacement spike.

Eyre and Bartlett⁽³⁹⁾ observed defect clusters in iron irradiated to a fluence of 2.5×10^{20} n/cm² (fission), estimated to be $\sim 8 \times 10^{19}$ n/cm² ($E > 1$ MeV) at 60 °C. Black dots from 10-100 Å in diameter were observed to change contrast upon tilting the microscope stage. Annealing at 300 and 400 °C caused a slight coarsening and permitted resolution as dislocation loops.

Several large loops analyzed after the anneal at 400 °C were found to be interstitial in nature, but several smaller loops appeared to be of the vacancy type. The authors, while concluding the majority of loops to be interstitial, could not exclude the possibility of a few vacancy loops. The density of clusters decreased upon annealing above 300 °C, and all visible damage had disappeared at 500 °C.

Bryner⁽⁴⁰⁾ examined a number of iron specimens irradiated at 60-90 °C at fluences of 2×10^{18} to 2×10^{20} n/cm²

($E > 1.45$ MeV). After irradiation, only the 2×10^{20} n/cm² specimen showed any evidence of radiation damage, but annealing for 2 hr at 300 °C or higher produced spots in the 1×10^{19} n/cm² specimens and increased the size and density in the higher exposure specimens. Continued annealing caused growth of the clusters and many were resolved as loops. No analysis of the sense of the loops was made. A 4 hr anneal at 600 °C removed all traces of loops.

Smidt⁽⁴¹⁾ reported the observation of a low density of defect clusters in Johnson-Matthey iron foils irradiated to a fluence of 9×10^{19} n/cm² ($E > 1$ MeV) at an irradiation temperature of 60 °C. A 2 hr anneal at 400 °C (Figure 6) caused growth of the clusters. While some were resolvable as loops, they were too small to use in determining the sense of Burger's vector.

Transmission electron microscopy studies thus show that aggregates of interstitial atoms form by long range migration in iron irradiated to fluences of 10^{19} to 10^{20} n/cm² ($E > 1$ MeV). Vacancy aggregates may also exist, although this is not unequivocally established. In short, microscopy studies still leave several questions unanswered because of the small defect sizes in iron.

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93,600 X

FIGURE 6. Transmission Electron Micrograph of Defect Aggregates in Johnson-Matthey Iron Irradiated to Fast Fluence of 8×10^{19} n/cm² ($E > 1$ MeV) at a Temperature of 60 °C and Subsequently Annealed for 2 hr at 400 °C. (Reference marks are one micron apart.)

V. DISLOCATION--SUBSTRUCTURE INTERACTIONS

An analysis of the damage produced by neutron irradiation and the damage recovery spectra indicate that resulting damage will be strongly dependent upon irradiation temperature because the state of aggregation of the defects is dependent upon their migration characteristics and the thermal stability of the aggregate. Thus, specimens irradiated and tested below 77 °K might be hardened by point defect interactions or small vacancy clusters. Irradiations and testing at around 273 °K should reflect the formation of interstitial clusters or loops by long range migrations, and irradiation and testing in the range 300-400 °C should reflect the dissociation of clusters or loops.

A. REVIEW OF INTERACTION MECHANISMS

The estimate of the strength of a material from the dislocation-substructure interactions involves an estimation of the increase in energy or work done in the interaction process, a calculation of the work done by the dislocation in traversing the slip plane, and a calculation of obstacle separation on the slip plane.⁽⁴²⁻⁴⁴⁾ More recent refinements attempt to take into account the flexibility of the dislocation line.^(43,44)

The calculation of the increase in energy for a single interaction event might include elastic interactions or the creation of new surface area by the dislocation passing near or through a defect aggregate. Elastic calculations, in addition to being laborious and complex, are valid only as far as the assumptions based on linear elasticity theory are valid. Many of the more common cases have been worked out. The work done in a single interaction event is then equated to the work done for a dislocation traversing unit area of the slip plane

$$W/\text{unit area} = F/\text{unit length} = \tau b.$$

The unit length over which this interaction takes place can be roughly estimated from the volume density of obstacles as $\bar{x} \approx N_V^{-1/3}$, but a more appropriate estimate is based upon the areal density of obstacles on the slip plane. If interaction is assumed to occur for all obstacles within a distance $r = \ell/2d =$ radius of particle from the slip plane, then a unit volume ℓ^3 is divided into ℓ/d slices of thickness d , to yield an areal density $N_A = \frac{N_V}{(\ell/d)} = N_V d$ (for unit volume). The average separation on the slip plane is then $\bar{x} = N_A^{-1/2} = (N_V d)^{-1/2}$. More refined calculations should take into account the fact that, for strong obstacles, the dislocation line will bow out and thus contact more obstacles. Foreman and Makin's⁽⁴⁵⁾ computer calculations show Friedel's⁽⁴⁴⁾ approximation to be quite good.

$$D \approx \left(\frac{\mu b \ell^2}{2\tau} \right)^{1/3} \approx \left(\frac{\mu b}{2\tau N_V d} \right)^{1/3}$$

where D is the separation between pinning points, μ is the shear modulus, τ is the shear stress, and ℓ is the distance between obstacles along the dislocation line.

The interaction of point defects with dislocations is still under active discussion, due in large part to the inability to properly treat the dislocation core. Linear elasticity theory predicts a size interaction in which a vacancy would be attracted to the compressional side of an edge dislocation and an interstitial to the tension side. There is also an interaction due to the inhomogeneity in the elastic medium. An additional interaction arising from the tetragonality of the point defect has been treated by Fleischer.⁽⁴⁶⁾ A point defect can always add onto an existing jog to lower its energy, but the addition to a straight segment of dislocation requires the formation of a double jog or creation of $2b$ length of dislocation line. This occurrence is unlikely except in high supersaturations of defects.

The possible interactions of a dislocation with point defects are determined by the temperature. At very low temperatures, the dislocation must move to the defects, while at higher temperatures the defects can migrate to the dislocation line and perhaps form jogs. A jog on a screw dislocation is, of course, a segment of edge dislocation and can only move non-conservatively with the creation of point defects.

Kimura and Maddin⁽⁴⁷⁾ have shown the interaction between a moving dislocation and dispersed point defects (case of no migration at low temperature) to be about 0.2 kg/mm^2 maximum for quenched vacancies in fcc metals and, being strongly temperature dependent, to decrease to zero interaction at 200°K .

A stronger interaction results from the Fleischer tetragonal distortion interaction which might arise from an interstitial atom or a vacancy, or from an interstitial platelet. Fleischer has shown that for carbon in iron the interaction amounts to $\tau = \frac{\mu C^{1/2}}{10}$ for an interstitial atom with [100] symmetry, where C is the concentration.⁽⁴³⁾ This interaction is thermally activated and varies from the maximum stress, τ_0 , with the temperature dependence $\tau = \tau_0 [1 - (\frac{T}{T_0})^{2/3}]$, where T_0 is an experimental constant.

The motion of a screw dislocation with jogs is controlled by the process of creating point defects. The estimated energy for creating vacancies is the energy $E_F^V \approx \frac{1}{5} \mu b^3$, and for interstitials is $E_F^i \approx 3\mu b^3$.⁽⁴⁸⁾ This estimate would require stresses of the order of $\frac{1}{5} \mu \frac{b}{\ell}$ and $\frac{3\mu b}{\ell}$, where ℓ is the separation between jogs. Such an interaction could amount to a sizeable increase in strength, especially for the interstitial for which the theoretical shear strength would be approached for $\ell = 90b$. A drag force of this magnitude would probably immobilize the dislocation, and deformation would proceed by multiplication.

A small cluster of vacancies or interstitials could be treated in the same manner as hardening by precipitates. If the obstacles are weak, they will be sheared by the moving dislocation, thus breaking bonds or creating new surface areas. The energy to create two surfaces of new area A_0 will be $E = 2A_0\Delta F_s$, where ΔF_s is the free surface energy.⁽⁴⁹⁾ For an area bd created on particles spaced at a distance ℓ along the dislocation line, the stress will be given by $\tau = \frac{\Delta F_s d}{b\ell}$. The free surface energy per unit area, ΔF_s , can be estimated from the force to separate the atoms, $\Delta F_s \approx \frac{\mu b^2}{16}$, or from the binding energy per defect, $\Delta F_s \approx \frac{n}{b} E_b N$, where n is the number of bonds broken per atom, E_b is the binding energy per atom, and N is the number of atoms per cm^2 .⁽⁵⁰⁾ In the case of strong obstacles, the limiting process is the bowing of a line segment to form new dislocations as envisioned by Orowan⁽⁵¹⁾ for precipitation hardening: $\tau = \frac{\mu b}{2\ell}$.

The crossover point for the latter process can be seen to occur at about $d = 8b$ for precipitates with a shear modulus close to that of the matrix. The obstacles will also be sheared by the Orowan mechanism after the stress concentration from the pile-up of several encircling loops builds up sufficiently to shear the obstacle. An alternative to the pile-up in materials with unextended dislocations is the cross-slip of screw dislocations around strong obstacles.

Kroupa and Hirsch⁽⁵²⁾ have analyzed the interaction between the dislocation and a small cavity of radius, R_c , from elastic considerations and find $E = \frac{\mu b^2 R_c}{5}$. The resulting contribution to the flow stress was estimated to be $\tau = \frac{\mu b R_c N_v^{2/3}}{10}$ using the Friedel⁽⁴⁴⁾ approximation for spacing of cavities along a line and an average force of $\mu b^2/10$. Kroupa and Hirsch calculated the energy of this process to be about 1.5 eV, a value in the proper range for thermal activation.

The final interaction to be considered is that with a dislocation loop. Since only unfaulted loops have been observed in bcc metals, the only case considered will be for the prismatic loops. Kroupa and Hirsch⁽⁵²⁾ have considered the elastic interaction between a prismatic loop and a dislocation. They found the short range elastic interactions to be substantial in favorable orientations. Averaging overall orientations, an average force of $F \approx \frac{\mu b^0 b^\ell}{4}$ was found, where b^0 represents the Burger's vector of the loop, and b^ℓ that for the line. The stress to move the dislocation was given as $\tau = \frac{\mu b^0}{8 \ell^2}$, where ℓ^2 is the average separation of interacting loops. This latter quantity was shown to depend on the interaction energy, and τ became $\tau = \frac{1}{8} \mu b^0 R N^{2/3}$, where N is the volume density of loops. This interaction energy also yields $\tau = \frac{1}{4} \mu b (N_V d)^{1/2}$ when Friedel's estimate is not used.

Saada and Washburn⁽⁵³⁾ have considered contact interactions or junction reactions between dislocations and prismatic loops in fcc structures. Depending on the relative orientations of the loop and moving dislocation, no interaction, intersection, or a junction reaction may result. The junction reaction, the most significant, produces an increase in line length of $2R$, an energy increase of $\mu b^2 R$, or a stress to move the dislocation of $\tau = \frac{\mu b R}{\ell^2} \approx \frac{\mu b N_V^{2/3} R}{\beta}$, where β is about 4, following Friedel's⁽⁴⁴⁾ analysis of attractive junctions.

Fleischer⁽⁴⁶⁾ has also applied his analysis of hardening by tetragonal distortions to the case of vacancy and interstitial loops and found $\tau = \frac{\mu c^{1/2}}{3.3} \left(\frac{b}{d}\right)^{1/2}$. Converting c to volume density of loops gives the result $\tau = \frac{1}{3.7} \mu b (N_V d)^{1/2}$, which is very close to the value obtained by Hirsch.

Valuable insight into the nature of the dislocation-defect interaction can be gained from the study of thermally activated deformation. If the obstacle to slip is small and the interaction

forces are short range, the deformation can be described in terms of an Arrhenius equation

$$\dot{\gamma} = N A b v \exp\left(-\frac{G}{kT}\right)$$

where $\dot{\gamma}$ is the shear strain rate, and $N = \frac{\rho}{\ell}$ is the number of interaction points for a dislocation density, ρ , and active segment of dislocation, ℓ . A is the area swept out per activation event $\approx \ell^2$, v is the vibration frequency of the dislocation $= \frac{v_D b}{\ell}$, and G is the free energy of activation. At 0 °K, the energy to surmount the obstacle is supplied entirely by the applied stress. As the temperature increases, however, thermal fluctuations assist the process, while the applied stress required to maintain motion at strain rate $\dot{\gamma}$ decreases. Eventually, the thermal fluctuations become so large that the obstacles no longer control the rate, although an applied stress is still required to move the dislocation against the long range forces.

The applied stress can thus be separated into two components as shown in Figure 7, where $\tau_a = \tau^* + \tau_i$. τ^* is defined as the effective stress for thermally activated flow, and τ_i is the stress to overcome the long range stress fields. The activation energy, G , is then the difference between the total energy to overcome the obstacle and that done by the mechanical work, namely $G = G_0 - \tau^* V^*$, where V^* , the activation volume, is approximately b times the area swept per successful fluctuation (the exact form of V^* depends upon the shape of the force-displacement curve). Processes with an activation energy up to perhaps 2-3 eV are thermally activated at room temperature, while processes with a greater activation energy will appear to be athermal.

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FIGURE 7. Variation of Flow Stress with Temperature in Iron Showing Partition of Flow-Stress into Athermal and Thermally Activated Components

B. ANALYSIS OF EXPERIMENTAL RESULTS

1. Thermally Activated Flow

The nature of the damage production processes and the observation of small aggregates of defects by transmission electron microscopy would lead one to suspect that these atomic-sized defects should influence the thermally activated flow properties of irradiated iron. A program to examine this possibility was initiated at Battelle-Northwest in 1963, and since

that time a considerable amount of data has been obtained on iron of two purity levels, (1) Ferrovac E, containing 70 ppm carbon, and (2) BMI zone refined iron, containing 3 ppm carbon. These materials have been irradiated at 60 °C to fast fluences of 1.5×10^{18} n/cm² to 2×10^{20} n/cm² ($E > 1$ MeV) and examined by strain rate change tests and temperature change tests. The most definitive test employed was the temperature change test, illustrated schematically in Figure 8, in which the change in flow stress was determined for 10-12 °C decrements in temperature, i.e., the derivative of the flow stress-temperature curve was determined experimentally.⁽⁵⁴⁾

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FIGURE 8. Test Sequence in Temperature Change Test Used to Obtain $(\Delta\tau/\Delta T)_\gamma$ as Function of Test Temperature

A difference in the slope of $\frac{\Delta\tau}{\Delta T}$ versus T curves between the irradiated samples and the unirradiated controls would be indicative of a new rate controlling process, presumably due to the defects introduced by irradiation. The results, shown in Figures 9 and 10 for Ferrovac E and BMI iron, show the slopes to be essentially unchanged by irradiation.

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FIGURE 9. Comparison of $(\Delta\tau/\Delta T)$ versus T Data for Irradiated and Unirradiated Ferrovac E Iron. (Decrease in slope and relatively minor change indicate defects introduced by irradiation do not change rate controlling mechanism of thermally activated flow in iron.)

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FIGURE 10. Comparison of $(\Delta\tau/\Delta T)\dot{\gamma}$ versus T Data for Irradiated and Unirradiated BMI Zone Refined Iron. (As in Figure 9, data indicate defects introduced by irradiation do not change the rate controlling mechanism of thermally activated flow in iron.)

Strain rate change tests to determine the activation volume confirm this result.⁽⁵⁴⁾ The results of V^* measurements as a function of strain at 295 °K are shown in Figures 11 and 12 for Ferrovac E and BMI iron controls, for specimens irradiated to 1.4×10^{18} n/cm² and 8.3×10^{19} n/cm², and for

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FIGURE 11. Activation Volumes for Ferrovac E Iron Samples Measured at 295 °K as Function of Strain. [Sample conditions examined include a furnace cooled control sample, irradiations to fast fluence of 1.4×10^{18} and $8.3 \times 10^{19} \text{ n/cm}^2$ ($E > 1 \text{ MeV}$), and a postirradiation anneal for 2 hr at 350 °C.]

specimens irradiated to $8.3 \times 10^{19} \text{ n/cm}^2$, and subsequently given a postirradiation anneal. The effective stress at 295 °K is quite small and the activation volume is an extremely sensitive function of τ^* in this temperature range, as shown in Figure 13. Thus, a small change in τ^* for this deformation temperature will result in large changes in V^* . The changes in V^* produced by the irradiation and recoverable after a 2 hr anneal at 350 °C are substantial enough to indicate a slight

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FIGURE 12. Activation Volumes for Zone Refined Iron Samples Measured at 295 °K as Function of Strain. [Sample conditions examined include a furnace cooled control sample, irradiations to fast fluences of 1.4×10^{18} and $8.3 \times 10^{19} \text{ n/cm}^2$ ($E > 1 \text{ MeV}$), and a postirradiation anneal for 2 hr at 350 °C.]

change in mechanism, but not enough to conclude that defects introduced by the irradiation have become rate controlling. It should also be noted in Figures 11 and 12 that the change in V^* is not dependent on fluence. Activation volume changes at lower temperatures, where V^* is not changing as rapidly with τ^* , show little change with irradiation. This problem will be considered in more detail in a subsequent section where it will be shown that the observed effects can be explained by the drag of a carbon atmosphere.

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FIGURE 13. Variation of Activation Volume with Effective Stress for Ferrovac E Iron. (At temperatures below 200 °K, activation volume is relatively insensitive to stress, but above 200 °K large changes in V^* result from minor changes in the thermally activated process and corresponding changes in τ^* .)

Ohr et al.⁽⁵⁵⁾ studied thermally activated stress relaxation in Ferrovac E irradiated with electrons ($4.2 \times 10^{18} \text{ e/cm}^2$) and with neutrons ($1.3 \times 10^{18} \text{ n/cm}^2$) at 65 and 95 °C, respectively. In this type of test, the flow stress for a given

temperature and strain-rate is established, the machine stopped, and the decrease in flow stress recorded as a function of time. When the machine is stopped, the plastic strain rate decreases from the imposed rate to a very low value at long times. The dislocation velocity which is related to the strain rate, $\dot{\gamma} = \rho b v$, can be expressed as a power law, $v = \mu(\sigma - \sigma_i)^m$, where μ and m are constants, σ is the applied stress, and σ_i the internal stress. Ohr determined the coefficient, m , of the stress dependence of the dislocation velocity and found no change in m for either electron or neutron irradiation. Ohr⁽⁵⁶⁾ has also determined at several test temperatures down to 77 °K the tensile strength of Ferrovac E iron, irradiated to a fast fluence of 1.2×10^{16} n/cm² ($E > 1$ MeV) and, within experimental error, noted no change in temperature dependence. He also measured the activation volumes at several test temperatures between 77 and 300 °K and noted no significant change in V^* , as might be expected at these low exposures.

McRickard⁽⁵⁷⁾ has also studied thermally activated flow in Ferrovac E iron doped with carbon and nitrogen and irradiated to a fluence of 2×10^{18} n/cm² ($E > 1$ MeV). He used a stress relaxation technique with a functional dependence somewhat different than Ohr's and performed his experiments in the Lüder's band. Although one might expect spurious results to arise from the inhomogeneous nature of the deformation in the Lüder's band, McRickard found no change in the activation volume of the irradiated iron determined by this technique.

One of the more perplexing contradictions stemming from studies of thermally activated flow in irradiated iron was the observation that no major change in mechanism and, therefore, no change in the temperature dependence of the flow stress is indicated from temperature change tests despite a rather large change in activation volume observed at room temperature. The observation of a change in activation volume following

irradiation and the trend for this activation volume to approach the unirradiated value as deformation progressed, coupled with the development of "channels" cleared of defects formed, led Laidler and Smidt⁽⁵⁸⁾ to postulate a change in deformation mechanism. Subsequent experiments on the temperature dependence of the flow stress, while showing no major change in mechanism,⁽⁴⁴⁾ disclosed an apparent contradiction.

This contradiction has been rationalized in a number of ways. Smidt,⁽⁴¹⁾ in identifying the activation volume as an extremely sensitive function of stress in this temperature range, has pointed out that a change in the density of mobile dislocations might influence the activation volume. Smidt also pointed out that changes due to irradiation were larger than the range normally observed between quenched and aged specimens from the same lot, and that the activation volume recovered more rapidly than the hardening, both of which indicated an interaction with a defect produced by the radiation. Arsenault⁽⁵⁹⁾ examined the possibility of observing athermally activated deformation controlled by the Fleischer mechanism in a material where the Peierls mechanism was ordinarily controlling, and concluded it would be observed only near T_0 (the temperature where τ^* approaches zero). Since T_0 in iron occurs around room temperature, it seemed possible that a new rate controlling mechanism might be observed at room temperature but not as a change in temperature dependence at lower temperatures. However, Smidt pointed out that the change in activation volume was not a function of fluence as might be expected if the Fleischer mechanism were actually being observed. Arsenault⁽⁵⁹⁾ has also suggested that the changes of V^* with strain may be a consequence of the highly localized deformation in irradiated materials similar to the changes in true strain rate observed by Schwink and Neuhauser⁽⁶⁰⁾ in irradiated Cu. This should not influence the results, however, since the activation volume is determined

from a ratio of strain rates and the results should be valid so long as this ratio is proportional to the ratio of cross-head velocities.

Recent tests of the temperature dependence of the flow stress of Ferrovac E iron in the range 200 °C to -70 °C show that a drag stress produced by carbon (and/or nitrogen) impurities influences the flow stress around room temperature and provides a more satisfying explanation of the mechanism by which irradiation changes the activation volume. Quantitative results were difficult to obtain from a temperature change test because dynamic strain aging causes serrated flow above 60 °C and, from 50 °C to 10 °C, the time between tests influences the magnitude of $\Delta\tau/\Delta T$. However, it was clear that $\Delta\tau/\Delta T$ reached a maximum at about 35 °C and the drag stress from this source became inconsequential at about 0 °C.

This effect was not previously considered because Cottrell's theory⁽⁶¹⁾ of atmosphere drag predicts a condensed atmosphere in this temperature range. Arsenault⁽⁶²⁾ had previously studied stress-induced ordering in creep tests of low carbon iron and concluded the effect could be observed at creep rates of $2.5 \times 10^{-5} \text{ min}^{-1}$ at 274 °K but would not be observed in tensile tests at common strain rates. Estimates of the plausibility of the drag mechanism using Cottrell's equations for an uncondensed atmosphere at 50 °C were made with the following equation:⁽⁶³⁾

$$v_D = \left(\frac{D}{kT} \right) F = \frac{D}{kT} \frac{A}{r^2} = \frac{\dot{\gamma}}{\rho b} .$$

A relation between ρ and r was determined from the other experimental parameters and Cottrell's value of $A = 1.5 \times 10^{-20} \text{ dynes/cm}^2$ was used. An atmosphere radius of $r = \frac{A}{2kT}$ was consistent with a reasonable dislocation density of $2 \times 10^{11} \text{ cm/cm}^3$. The fraction of carbon atoms condensed on the dislocation line,

C_0 , was estimated and the maximum drag stress calculated from $\sigma_c = \frac{17A}{b} C_0$. This estimate of 2.7 kg/mm^2 is of the same magnitude as the serrations, 1.4 kg/mm^2 . The problem of atmosphere drag has recently been studied in detail by Keh⁽⁶⁴⁾ who concluded that such effects do indeed influence the flow stress of iron-carbon and iron-nitrogen alloys.

Returning now to the application of this information to the interpretation of the changes in activation volume observed in radiation damage experiments discloses a more consistent picture. First, it should be noted that Ferrovac E iron (Figure 11), with more carbon, has a higher activation volume than zone-refined iron (Figure 12), and that quenching both samples increases the activation volume slightly while aging lowers the activation volume. Irradiation to fluences of 1.4×10^{18} , 8.3×10^{19} , and $2 \times 10^{20} \text{ n/cm}^2$ causes about the same decrease in activation volume for all fluences, thus indicating that the decrease in activation volume observed at low strains is due to removal of carbon from solution. Carbon is released by the interaction of dislocations with defects in which the carbon is trapped, and the activation volume approaches that of the unirradiated sample as the deformation proceeds. Postirradiation annealing also causes dissociation of the defects and release of carbon so that the activation volume recovers.

It is also interesting to note that the activation volume in BMI iron after irradiation and deformation or annealing is larger than the activation volume in unirradiated samples. This result would be consistent with small carbide precipitates going into solution during the irradiation. Thus, it can be concluded that the activation volume measured at 295°K is influenced by Cottrell's atmosphere drag, and that changes produced by irradiation are a consequence of the trapping of interstitial carbon and/or nitrogen in the atmosphere by the

defects produced during the irradiation. It can further be concluded that the interaction of moving dislocations with the impurity-defect complex destroys the complex and releases the interstitial impurity atoms which again form an atmosphere.

2. Athermal Stress

The observation that neutron irradiation has little influence on the thermally activated flow process means that any changes in the athermal stress will merely raise or lower the stress-temperature curve without changing the shape. This permits us to draw inferences about the athermal stress from conventional tensile tests, provided the data are taken at the same strain rate. Tests run under different strain rates can still be compared if the data were taken above the critical temperature at which the process ceases to be controlled by a thermally activated mechanism. In iron, for strain rates in the range $0.002 - 0.02 \text{ min}^{-1}$, this critical temperature is about room temperature.

Figure 14 shows the three primary characteristics of the athermal hardening to be (1) fluence dependence, (2) saturation at high exposures, and (3) dependence upon interstitial impurity content. In the figure, the increase in lower yield stress is shown for Ferrovac E and zone-refined iron at fluences of 1.4×10^{18} , 8.3×10^{19} , and $2 \times 10^{20} \text{ n/cm}^2$ ($E > 1 \text{ MeV}$). The hardening, seen to be substantial at the higher exposures, amounts to roughly a doubling of the yield strength in Ferrovac E at $8 \times 10^{19} \text{ n/cm}^2$.

The influence of interstitial impurities and saturation effects will be discussed in more detail in a subsequent section. For the moment, the possible mechanisms of athermal hardening consistent with knowledge of the substructure will be considered while qualitatively noting that the hardening is enhanced in materials with a high impurity content. The defect

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FIGURE 14. Increase in Athermal Hardening (shear stress, $\tau = 1/2\sigma$) as Function of Fast Fluence Observed in McRickard's Experiments⁽⁸⁴⁾ and in Smidt's Experiments.⁽⁶³⁾ (Note increase in hardening with fluence, effect of carbon content, and softening at high fluences.)

substructure has previously been shown to be dependent upon irradiation temperature and annealing temperature, so it is logical to anticipate that the athermal hardening will also be dependent upon irradiation temperature.

Low temperature irradiations of iron have been done at Oak Ridge and Brookhaven. McRickard and Chow⁽⁶⁶⁾ irradiated Ferrovac iron to a fluence of 2×10^{18} n/cm^2 ($E > 1.45$ MeV) at an irradiation temperature of 183-203 °K. About half the hardening was observed to occur from this low temperature irradiation, as compared with an equivalent exposure at

343 °K (70 °C). There may also have been an increase in the temperature dependence of the flow stress, although this interpretation is dependent on one point (Figure 15). Hinkle and Smith⁽⁶⁷⁾ have shown that Ferrovac E iron irradiated to a fluence of 7.8×10^{16} n/cm² at 143 °K shows little or no increase in the lower yield stress when tested at 0 °C, but the yield stress increases about 2.3 kg/mm² (shear stress) after a 10 min anneal at 100-150 °C. An equivalent exposure at 90 °C produced an increase of 3.9 kg/mm². It would thus

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FIGURE 15. Tensile Test Results of Hinkle and Smith⁽⁶⁷⁾ Showing Lack of Hardening in Iron After Irradiation at -130 °C and Increase in Hardening After 10 min Anneals Above 50 °C as Compared with Samples Irradiated at 90 °C.

appear that hardening by the defects produced during a low temperature irradiation is substantially less than that produced by irradiation at 60-90 °C.

Recovery of damage after irradiation at 60-90 °C and during elevated temperature irradiations is better characterized than in the low temperature range. McRickard and Chow⁽⁶⁶⁾ report a very slight increase in yield stress after a 2×10^{18} n/cm² ($E > 1.45$ MeV) irradiation at 250 °C. Smidt⁽⁶⁸⁾ has observed an increase of 0.5 kg/mm² after an irradiation to $\sim 8 \times 10^{19}$ n/cm² at 400 °C. Harries and Eyre⁽⁶⁹⁾ (Figure 16) have studied the influence of irradiation temperature on hardening in mild steel from 100 °C to 350 °C. They found

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FIGURE 16. Relative Irradiation Hardening in Arbitrary Units for Several Steels Irradiated to Fission Fluence of 2.5×10^{17} n/cm² at Irradiation Temperatures from 50 °C to 300 °C. (From the work of Harries and Eyre, Reference 69)

maximum hardening to occur at 100 °C, with a steady decrease in hardening as the irradiation temperature increased. On a relative scale, a mild steel containing about 0.10-0.13 wt% carbon had a hardening of 8, a silicon-killed steel had 7, and an aluminum-killed steel had 5 after irradiation at 100 °C to a fission fluence of 2.5×10^{17} n/cm², and all three had a value of about 1 after irradiation to the same fluence at 350 °C.

Hardness recovery experiments on iron, after ambient temperature irradiations, have been reported by several investigators. Eyre and Bartlett⁽³⁹⁾ report microhardness recovery in the range 250-500 °C. Smidt⁽⁷⁰⁾ observed recovery of hardness in the range of 255 to 500 °C (Figure 17). This recovery stage correlates rather well with observations by Eyre and Bartlett⁽³⁹⁾ of cluster growth after 1 hr anneals between 300 and 400 °C. The density of clusters decreased and some were resolvable as loops after anneals in the 350 °C to 500 °C range. All traces of damage were removed after anneals at 500 °C. Bryner⁽⁴⁰⁾ observed growth of spots and an increase in density after anneals at 300 °C. They also observed loops resolved at 350 °C, as well as continued loop growth, although with a decrease in density up to 500 °C. Eyre and Bartlett as well as Bryner concluded that the total number of defects contained in the loops reached a maximum at about 350 °C and decreased thereafter so that, above 350 °C, loop growth was by agglomeration rather than migration of point defects to the loops. This finding agrees with evidence obtained by Brimhall⁽⁷¹⁾ that loop growth in Mo in the range 400-600 °C results from agglomeration of spot defects into loops by a combination of self-climb and prismatic glide. This occurrence in iron at equivalent homologous temperatures would be in the 140 to 265 °C range.

The presence of hardening in samples possessing no visible defects, and the coincidence of hardness recovery and loop

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FIGURE 17. Recovery of DPH Hardness in Cold-Worked and Irradiated Iron Samples of Two Purities. [Irradiation to a fast fluence ($E > 1$ MeV) of 2×10^{20} n/cm² at an irradiation temperature of 60 °C. Annealing time was two hours at the indicated temperature.]

growth provide conclusive evidence that defect aggregates of less than 30 Å in diameter produce greater hardening than larger aggregates. The observation that low temperature irradiations produce little hardening indicates point defect dispersions to be less effective barriers to dislocation motion than clustered defects. It would also appear that a radiation temperature of about 0-100 °C results in the greatest hardening. The observation of interstitial loops by electron microscopy and the low mobility of vacancies in this temperature range, as well as the observation that interstitial impurities trap single

vacancies, would indicate aggregates of interstitials less than 30 Å in diameter as the main source of hardening. While the growth mechanism of these aggregates is still open to question, Moser's studies of magnetic after-effect indicate that migration of tri- or quadri-interstitials or higher might occur around 0 °C. Another possibility is the release of single or di-interstitials from impurity traps in this temperature range with subsequent growth of existing clusters.

An estimate of the increase in strength obtainable from various interaction mechanisms was made on the assumptions of a 20 Å diameter particle with a density of $1.2 \times 10^{16} \text{ cm}^{-3}$ [Bryner⁽⁴⁰⁾ for Ferrovac iron]. The strongest interaction considered was the limiting case of an impenetrable particle requiring loop bowing as illustrated in Figure 18 (the Orowan mechanism).⁽⁵¹⁾ Taking $b = 2.5 \text{ Å}$ and $\mu = 11.4 \times 10^3 \text{ kg/mm}^2$ for iron, an increase in yield strength of 6.8 kg/mm^2 was calculated from $\tau = 1/2 \mu b (N_v d)^{1/2}$. This result compares favorably with the observed increase in yield strength at 10^{19} n/cm^2 ($E > 1.45 \text{ MeV}$) of 8.8 kg/mm^2 , since an increase in the observed cluster density from 1.2 to 2×10^{16} would give the observed value. Foreman and Makin⁽⁴⁵⁾ have shown for a random array of impenetrable objects that a more appropriate value is $\tau = 0.4 \mu b (N_v d)^{1/2}$. The estimated result, therefore, should be a bit lower. It is clear, however, that a very strong interaction is required to explain the hardening by the defects observed by electron microscopy. Ohr et al.⁽⁷²⁾ have obtained good correlations between the hardening and cluster density in Nb by using Kroupa and Hirsch's estimate of the attractive interaction with dislocation loops, and assuming a strong interaction to give $\tau = 1/4 \mu b (N_v d)^{1/2}$. As pointed out by Ohr, this estimate is very close to Fleischer's mechanism of hardening by tetragonal distortions for loops,

$$\tau b = \frac{F}{l} = \frac{E}{A} = \frac{1}{2} \frac{\mu b^2}{l}$$

$$l = (N_v d)^{-1/2}, \quad \mu = 11.4 \times 10^3 \text{ kg/mm}^2, \quad b = 2.5A$$

$$\tau = \frac{1}{2} \mu b (N_v d)^{1/2}$$

$$\text{FOR } d = 20A, \quad N_v = 2 \times 10^{16} \text{ cm}^{-3}$$

$$\tau = 9 \text{ kg/mm}^2$$

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FIGURE 18. Orowan Mechanism of Precipitation Hardening and Sample Calculation of Hardening for Typical Values of Defect Density and Size in Iron Irradiated to Fast Fluence of About $9 \times 10^{19} \text{ n/cm}^2$.

$\tau = \frac{\mu b}{3.7} (N_v d)^{1/2}$. Both give the same order of magnitude increase in yield stress but are half the value for the Orowan mechanism.

C. ROLE OF IMPURITIES

The influence of impurities, especially interstitial impurities, is well established. Chow, McRickard, and Gurinsky⁽⁷³⁾ showed that the addition of strong carbide and nitride formers to iron resulted in decreased hardening.

Harries and Eyre⁽⁶⁹⁾ have likewise demonstrated this effect. Smidt and Bement⁽⁵⁴⁾ have shown carbon content to influence both the magnitude and rate of hardening under identical irradiation conditions for Ferrovac E and zone refined iron. A further comparison with ingot iron specimens tested by Bement⁽⁷⁴⁾ shows an even greater increase in yield strength for equivalent exposures. Minier-Cassayre⁽²⁴⁾ has also demonstrated that substitutional impurities produce noticeable modifications in the resistivity recovery spectrum of iron.

At present, two general mechanisms for explaining the influence of interstitial carbon and nitrogen impurities on the hardening from radiation damage are under consideration. One model postulates that carbon (and nitrogen) serve as nucleation sites for the growth of clusters by trapping migrating interstitial atoms which otherwise would be lost by recombination with vacancies or migration to other sinks. The other model postulates that the enhanced hardening is due to the precipitation of carbon and nitrogen at defect aggregates.

Evidence favoring the nucleation of clusters at carbon atoms includes the analogous heterogeneous nucleation of vacancy clusters in quenching experiments of the fcc metals,⁽⁷⁵⁾ a calculated binding energy of 0.5 eV between the interstitial and a carbon atom,⁽⁷⁶⁾ and a favorable relationship between the annihilation distance for interstitials and the separation of carbon atoms.⁽⁴¹⁾ In regard to the latter point, Smidt showed that the annihilation distance for an interstitial migrating with $E_m^i = 0.33$ eV to a vacancy $E_M^V = 0.68$ eV at an irradiation temperature of 60 °C in a neutron flux of 3×10^{13} n/cm²/sec should be about 4900 Å. This distance compares to a separation of 40 Å for carbon atoms in iron containing 50 ppm in solution. The calculation was based on Wechsler's⁽⁷⁷⁾ estimate of pair production in iron and,

although oversimplified, clearly showed that the interstitial would encounter many carbon atoms before annihilation at a vacancy.

Damask and Dienes,⁽⁷⁸⁾ in considering the influence of heterogeneous nucleation sites on the density of interstitial clusters for several sink concentrations, have shown that an increase in impurity content will increase the density of clusters.

The main objection to this model and the primary support for a carbon precipitation model is the observation that carbon (and/or N_2) is removed from solid solution at about the same temperature as maximum hardening is observed as a function of irradiation temperature or postirradiation annealing temperature. Damask⁽³⁰⁾ has shown that the disappearance of carbon from solution occurs at 50 °C by trapping a radiation-produced defect which is most probably the single vacancy. An enhanced nucleation rate was also observed and attributed to an increase in the number of nucleation sites. The latter effect saturated at a fluence of about 10^{15} n/cm², however, and thus cannot be correlated with the irradiation hardening which saturates at exposures around 10^{20} n/cm². Stanley⁽⁷⁹⁾ found a similarly enhanced nucleation rate in iron-nitrogen alloys after neutron irradiation, but not after electron irradiation. He concluded that the nucleation site must be a defect aggregate rather than a point defect. The presence of carbon or N_2 in solution following irradiation indicates that not all the carbon was trapped by interstitials.

These objections to the model for nucleation of clusters at carbon atoms can be circumvented by assuming growth of only a small number of the clusters to a stable size during the irradiation. Single interstitial-carbon complexes might dissociate at -30 °C, as suggested by Damask,⁽³⁰⁾ while complexes

grown by trapping additional carbon atoms could be stable and continue to grow as more interstitials are released from traps. A critical distinction between the two models should be possible from a comparison of cluster density and carbon concentration. If the number of clusters varies with carbon concentration, then the cluster nucleation model would be preferred. However, if the number of clusters remains constant but varies in size, then the carbon precipitation model would be substantiated. Tentative support for the first case can be drawn from a comparison of cluster densities observed by Eyre and Bartlett in hydrogen-purified Ferrovac iron ($2 \times 10^{16} \text{ cm}^{-3}$),⁽³⁹⁾ and by Smidt⁽⁴¹⁾ in Johnson-Matthey iron ($2 \times 10^{15} \text{ cm}^{-3}$). A rigorous experiment under controlled conditions would be helpful in resolving this question.

In fully aged alloys where carbon is not in solution, some resolutioning must occur for the carbon-defect interactions to take place. Damask⁽⁸⁰⁾ has reviewed some of the experiments where resolutioning in displacement spikes has been studied, the most informative of which was the work of Piercy⁽⁸¹⁾ on Cu-2% Co. This experiment showed that only particles with a radius less than 12.4 Å and totally enclosed in the spike were resolutioned. This observation of a size effect would indicate that preirradiation heat treatment could influence the amount of carbon resolutioned by irradiation. It is known, for example, that aging at temperatures below 100 °C produces a very fine dispersion of ϵ carbide, while aging above 250 °C produces larger carbide particles of Fe_3C .

D. SATURATION EFFECTS IN IRON

Several observations have shown saturation of radiation damage at high exposures and, intuitively, one would expect this to occur as the volume of atoms perturbed by displacement spikes approaches the volume of the lattice. Among the

relationships suggested to describe the fluence dependence of radiation hardening are $(\phi t)^{1/3}$, observed experimentally at low exposures,⁽⁸²⁾ $(\phi t)^{1/2}$ predicted by Seeger from his theory of hardening by depleted zones,⁽¹⁴⁾ and $A[1-\exp(-B\phi t)]^{1/2}$ suggested by Makin and Minter.⁽⁸³⁾ The latter expression was derived by assuming the formation of new clusters only in an undamaged volume of the crystal, and by using Seeger's estimate to relate number of clusters formed to the increase in yield strength. The constant A is the reciprocal of depleted zone volume^{1/2}.

McRickard⁽⁸⁴⁾ (Figure 14) has recently shown that the saturation of radiation hardening in Ferrovac E iron occurs at 2×10^{19} n/cm² ($E > 1.45$ MeV) with a maximum increase in yield stress of 8.8 kg/mm² and fits Makin and Minter's expression for a damage volume of 4.4×10^{-19} cm³ ($d = 94$ Å).

Smidt⁽⁶³⁾ has also studied Ferrovac E iron in the fluence range from 1.4×10^{18} n/cm² to 2×10^{20} n/cm² ($E > 1$ MeV) and has found the maximum increase in strength to be 8.9 kg/mm² at a somewhat higher fluence of 8×10^{19} n/cm² (Figure 14). The difference in the saturation fluence is probably due to the lower threshold value for neutron damage (1 MeV versus 1.45 MeV).

The greatest significance in these two investigations, however, is the observation that oversaturation or softening occurs at fluences beyond saturation. This observation indicates that not only does the efficiency of damage production decrease at high exposures but dynamic recovery can also occur. Among the mechanisms by which this recovery might take place are loop interactions to form networks, and growth of loops or dissolution of precipitates. The interaction of loops to form dislocation networks has been observed in Mo⁽⁸⁵⁾ irradiated to fluences of 5×10^{19} n/cm² at 60 °C. The influence of network formation on the yield strength is not well characterized because of the onset of embrittlement at these exposures.

The dissolution of small carbide precipitates might also result in a decrease in strength. These precipitates presumably strengthen the material by the Orowan mechanism of precipitation hardening in the same manner postulated for defect clusters. It is a well known fact that precipitation hardening is influenced by precipitate spacing and that maximum hardness results from aging under conditions where many small closely spaced precipitate particles form. This possibility is supported by the observation that oversaturation did not occur in an Fe-0.1% C alloy,⁽⁸⁴⁾ and that the increase in yield strength in this alloy was less than for the lower carbon Ferrovac alloy.

Perhaps the most informative experiments to date have been Beeler's⁽²¹⁾ damage simulation by computer experiments. Although absolute values of the parameters are sensitive to the potential functions used in the calculations, relative results should be valid and reveal the general features of the process of radiation damage. These models confirm the displacement spike-depleted zone model of the primary damage event by showing that most interstitials are produced as single interstitials, while small vacancy clusters frequently survive the damage.

Interstitial clusters were observed to form and collapse into loops, but unfortunately the lattice model was not large enough to follow their growth in great enough detail to estimate the density of loops in a saturated damage state. The size of the vacancy clusters was observed to increase as subsequent displacement spikes occurred in the previously disrupted region. In addition, interstitials and vacancies were created in close proximity to those formed by earlier damage events, and the number eliminated by annihilation increased as saturation was approached. Beeler estimated that saturation would occur at about 2.6×10^{19} n/cm² (E > 1 MeV) if 50% of the

defects annealed out at room temperature. Even though interstitial clusters were not considered, their growth in size can also be assumed as saturation is approached, a result indicated by transmission electron microscopy experiments. A softening effect associated with saturation (and larger loops) would require a different dislocation-interaction mechanism for small clusters or loops than for larger loops in the unlikely event that total loop density is not reduced.

At the moment, there is no conclusive evidence to establish the mechanism by which oversaturation occurs. The fact that it does occur indicates radiation damage to be a more dynamic process than envisioned in the fixed damage volume model in which no additional changes can take place. This conclusion was confirmed by Beeler's computer modeling experiments. Of the three mechanisms considered, network formation is possible but might lead simultaneously to embrittlement, previously unobserved in low carbon iron. Dissolution of precipitates could occur, but the additional carbon in solution might nucleate more clusters which would increase the hardening. The growth of loops into larger sizes could result in a change in hardening mechanism with subsequent softening. The author prefers the precipitate dissolution hypothesis at the present time because high carbon alloys have not been observed to show saturation at exposures thus far examined.

VI. APPLICATIONS

In this section, the general treatment of the application of our knowledge of dislocation-substructure interactions to work hardening and embrittlement will stress acquired insights rather than a comprehensive review of the literature. The conclusions reflect our present dependence on empirical knowledge in contrast to more precise formulations from fundamental principles. This situation reflects the status of the fields

of work hardening and fracture, and it is safe to assume that major advances in either of these fields will be rapidly assimilated in the field of radiation damage.

A. WORK HARDENING

Among the more prominent features of irradiation damage is the decrease in work hardening coefficient and loss in ductility observed at exposures greater than about 10^{18} n/cm². Closely related to these phenomena is the observation that slip bands in iron irradiated to exposures greater than 5×10^{18} n/cm² are much better defined and deeper than the diffuse slip bands in unirradiated iron.⁽⁸⁶⁾ It is interesting to note that Cottrell suggested dislocation-defect interactions could lead to coarsening of slip traces and a stage of "runaway slip" as early as 1958.⁽⁸⁷⁾

As has been shown in the preceding section, small clusters or loops of aggregated point defects serve as barriers to the motion of dislocations and harden the lattice. Mastel et al.⁽⁸⁸⁾ have shown conclusively that the defects observed in irradiated Mo are removed from the slip plane by the passage of dislocations. Eyre and Bartlett⁽³⁹⁾ also observed this removal of defects from the slip plane in irradiated and deformed iron.

Keh and his co-workers at U.S. Steel⁽⁸⁹⁻⁹¹⁾ have extensively investigated the formation of substructure in deformed iron under a variety of deformation conditions. In general, a cell structure develops during the first 3% strain and then remains relatively constant in size and in dislocation density in the cell walls during the remainder of the deformation. Higher strain rates, lower deformation temperature, and higher purity all produce a more uniform distribution of dislocations. Work hardening appears to be correlated with the density and distribution of dislocations on secondary slip planes.

These observations on iron agree quite well with the Hirsch-Mitchell⁽⁹²⁾ work hardening theory. According to this theory, dislocations emitted by primary sources pile up against barriers, and internal stresses build up until sources on secondary planes become operative. Dislocation density increases on the secondary planes will stop the primary sources, the stress increases further, and additional secondary dislocations are generated in a continuing process. This sequence continues until the primary sources are surrounded by ribbons of high dislocation density on the secondary planes. The thickness of this barrier depends on the back stress from primary pile-ups, the density of secondary dislocations in the region of the pile-up, and upon any other factors influencing the operation of secondary sources. As deformation proceeds into Stage III, cross-slip around the barriers becomes the predominant process, and the dislocation density in the ribbons (or cell walls) remains relatively constant.

The precise mechanism by which impurities modify the deformation process is not clear, although microscope observations⁽⁸⁹⁾ of substructure formation in high purity iron indicate less frequent occurrence of cross-slip than in iron of higher impurity. This result might be attributed to the absence of precipitates, but is more probably due to the absence of grown-in dislocations strongly pinned by impurities. Such locked dislocations would serve as nuclei for the development of tangles and cell walls.

Figure 19A shows the substructure typical of an iron specimen deformed 5% at room temperature. Cell walls are beginning to form in several areas while, in other areas, they are still rather poorly defined as a mass of dislocation tangles. An identical specimen of this Johnson-Matthey iron foil was irradiated to a fast fluence of 8×10^{19} n/cm² (E > 1 MeV) and then deformed 5%. Figure 19B shows the

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FIGURE 19.

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A. Transmission Electron Micrograph of Dislocation Substructure in Unirradiated Iron Foil Deformed 5% with Typical Cell Wall Formation.

B. Transmission Electron Micrograph of Dislocation Substructure in Iron Irradiated to Fast Fluence of 8×10^{19} n/cm² (E > 1 MeV) and Deformed 5%. (Densely packed slip planes show localized nature of deformation in irradiated material.)

(Reference markers are 1 micron apart.)

striking contrast in the deformation substructure. In place of the dense cell walls and dislocation tangles, there are several well defined slip planes with closely spaced dislocation. These have been indexed and shown to be $\{110\}$ and $\{112\}$ type planes inclined at an angle to the plane of the foil which is $\{111\}$.⁽⁹³⁾

It is quite obvious that cross-slip is severely restricted in the irradiated material. Dislocation barriers which normally would cause tangle formation and ultimately contribute to the operation of secondary sources and cell formation do not form. Once slip has been initiated on a particular plane, the defects present are removed from the plane and it becomes the weakest spot in the lattice. Subsequent dislocations from the source move over the same plane and highly localized deformation results. Since the cell structure does not develop in the normal manner, the material does not work-harden and, at high exposures, may deform to failure on a few planes with little reduction in area.

B. EMBRITTLEMENT

Chow and McRickard⁽⁹⁴⁾ have studied the shift in ductile to brittle transition temperature (Figure 20) resulting from neutron irradiation in a number of iron-carbon alloys using reduction in area as the criteria for embrittlement. They found that embrittlement, most significant in alloys containing greater than 30 ppm carbon, was dependent upon carbon content. It appeared further that the carbon free to go into solution was the important variable because alloys with strong carbide formers showed less embrittlement. The increase in transition temperature appears to saturate in the 10^{19} n/cm² range, the same exposure at which the increase in yield strength saturates. It was further noted that the embrittlement begins to recover after anneals at 250 °C, very close to the temperature at which the athermal hardening begins to recover. This evidence clearly

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FIGURE 20. Influence of Neutron Irradiation on Ductile to Brittle Transition Temperature as Measured by Reduction in Area. (From the work of Chow and McRickard, Reference 66)

indicates that the embrittlement produced by an ambient temperature irradiation and measured by a reduction in area criteria in a tensile test is produced by the same mechanism responsible for the athermal hardening.

On the basis of this evidence, it would appear that the fracture stress of the material is unaffected by the irradiation, and that the shift in transition temperature is due to the increase in athermal hardening. As shown schematically in Figure 21, the fracture stress exceeds the yield stress above about 130 °K in a low carbon alloy, and deformation occurs by plastic flow above the ductile to brittle transition temperature.

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FIGURE 21. *How Increase in Athermal Hardening Shifts Ductile to Brittle Transition Temperature to Higher Temperatures.*

Below 130 °K, the fracture stress is the lower value, and deformation results in fracture. An increase in the athermal hardening raises the whole yield stress-temperature curve but leaves the fracture stress unchanged. As a consequence, the intersection point of the fracture stress and yield stress curves occurs at a higher temperature, i.e., the ductile to brittle transition temperature is raised. The shift in ductile to brittle transition temperature resulting from irradiation can be minimized by alloy additions which, by tying up the

carbon and nitrogen and removing them from solution, reduces the number of nucleation sites at which interstitial clusters can be formed.

In the author's opinion, this interpretation of the shift in ductile-brittle transition temperature also adequately explains the results of low temperature irradiation experiments described by McRickard and Chow,⁽⁶⁶⁾ although it differs from their interpretation. In these experiments, samples were irradiated at 203 °K (-70 °C), 523 °K (250 °C), and 343 °K (70 °C). As shown in Figure 22, the samples irradiated at 203 and 523 °K showed no embrittlement and only a small increase in hardness compared to samples irradiated at 343 °K. Postirradiation annealing of the samples irradiated at 343 °K produced recovery in the hardness and embrittlement at the same temperatures. McRickard and Chow state that this recovery was not simultaneous and interpret the data as an indication that two different defect species are responsible for the hardening and embrittlement. They suggest Frenkel defects or simple combinations of these with carbon are responsible for the hardening, while the carbon trapped by vacancies is responsible for embrittlement. As previously shown in discussions of damage production and annealing, interstitials or simple associations of them are not likely to survive the dynamic annealing at an irradiation temperature of 70 °C. Even those surviving would not produce hardening of the magnitude observed.

In view of the simplicity of the transition temperature concept of embrittlement and the assumption of a fracture stress unchanged by irradiation, it is not too surprising that the hardness and shift in transition temperature do not occur simultaneously. An examination of the data under consideration also suggests sufficient experimental scatter in the results to make it difficult to determine the exact conditions under which hardness and embrittlement begin to recover. It is therefore

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