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Dispersed barrier hardening in irradiated metals

by

Marvin Sidney Beck

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Approved:
Signature was redacted for privacy.
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I. INTRODUCTION

Radiation hardening in metals and alloys is a phenomenon of long-standing interest in the field of nuclear metallurgy. This interest stems from fundamental and practical considerations. From the fundamental point of view, radiation hardening results from the interaction of slip dislocations with radiation-produced defects and defect clusters. A better basic understanding of these interactions is desired, consistent with the background of knowledge of the nature of plastic deformation in the absence of radiation. From the practical point of view, radiation hardening is important because of the need to use materials in radiation environments, particularly in nuclear reactors. Structural metals and alloys are both hardened and embrittled upon exposure to reactor neutrons (1,2). A better appreciation of the nature of radiation hardening and its relationship to radiation embrittlement should help to improve the design, operation and safety of nuclear reactors.

Many mathematical models have been developed to study various types of mechanical hardening (3,4,5) of which some have been applied to interpreting RH (6,7). These models are mainly phenomenological, but they do provide some insight into possible fundamental mechanisms. Once a fundamental explanation for the hardening phenomenon is obtained, parameters which control it may be identified and optimized to
minimize the hardening and embrittlement. Materials more resistant to hardening may also be developed. The following dissertation is a study of the application of some of these models to RH and to a further increase of hardening observed upon post-irradiation annealing, usually referred to as radiation-anneal hardening (RAH).

The agent responsible for RH is generally considered to be clusters of vacancies or interstitial atoms (8). During the irradiation of metals by energetic particles, lattice atoms are displaced from their lattice sites creating a damaged lattice structure. At ambient reactor temperatures much of this damaged structure rapidly recovers, although some intrinsic point defects remain which tend to coalesce and form clusters (9). These clusters are microscopic in size and are considered to interact with dislocations and thus retard slip. Since the yield stress is a measure of the onset of slip, any change of the slip behavior would produce a change in the yield stress. A change in the yield stress between the irradiated and unirradiated states could thus be interpreted as a measure of the change in dislocation mobility resulting from the radiation produced defects. But the yield stress is also controlled by solute impurity atoms, especially interstitial impurities in bcc metals (10). These interstitial impurity atoms appear to act as nucleation sites for defects (11) and the defects appear to operate as sinks for the
interstitial impurities (12). Thus, the impurity atoms tend to form complexes with the intrinsic radiation-produced defects and may be effectively removed from solid solution. The amount of these impurity atoms in solid solution therefore varies as a result of irradiation and in the bcc metals can change the yield stress considerably. This, then, also contributes to the RH in bcc metals which in conjunction with the hardening due to the intrinsic radiation-produced defects creates a fairly complex hardening mechanism.

Another effect associated with RH is that of the radiation-anneal hardening (RAH) (8,12). It is an additional hardening contribution which occurs upon post-irradiation annealing and is of considerable value in studying the mechanisms of RH. The annealing is done in stages, each at a higher temperature than the previous. As the temperature is raised, point defects and solute atoms become more mobile and move under the influence of internal gradients. Eventually the defect clusters also begin to change, some growing and others shrinking until all are eliminated from the material. By observing a physical property at each stage of the annealing some information about the kinetic behavior of the annealing mechanisms may be obtained. To date, though, these kinetics have not been resolved and the mechanisms involved in RAH and even in RH have not been firmly established.

The purpose of this project is to investigate the vari-
ous contributions to RH and RAH in bcc metals and to determine possible explanations of the hardening phenomena. Yield stress and hardness measurements have recently (11) been obtained for irradiated and unirradiated, high-purity vanadium samples doped with three levels of oxygen. The densities and size distributions of defects observable by transmission electron microscopy (TEM) were also obtained for the as-irradiated condition and following post-irradiation annealing. The question arises as to whether a correlation may be found between the measured hardness and the observed defects. An initial objective of this research project is thus to determine whether the variation in the TEM-observed defect density and size distributions obtained after irradiation and after post-irradiation annealing could account for the RH and RAH, respectively. The results of this study are given in section III and, although negative, do provide the basis upon which a subsequent analysis was developed. Less detailed information taken from the literature has also been used to help justify the conclusions reached and to provide a more general evaluation of the resulting hardening model.
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II. BACKGROUND

A. General

The effects of irradiation on the behavior of metals have been studied intensively over the past 25 years. Although extensive insight into the nature of intrinsic and extrinsic defects and their interactions has been gained, little progress has been made in understanding their specific contributions to the mechanical properties. A review of RH by Vineyard (1) in 1962 is about as valid today as when published. More recently, Bement (7) has provided a summary of the status of the effects of neutron irradiation on metals and alloys and he concludes that "while many available theories for the strength of metals and alloys are being extended in an eclectic manner to treat irradiation effects, the kinetic nature of neutron damaging processes and the special effects of internally generated constituents, which would normally be absent from conventional materials, indicate the need for new theoretical development."

One of the major difficulties encountered in modeling RH is the lack of a first principle model for mechanical hardening itself. On the atomic level, hardening is considered due to the pinning of dislocations and an interference of the slip process. However, mechanical hardening as measured by the yield or flow stress change in a tensile test is an average response of a very large number of mobile dislocations
C~10^ cm on a large number of slip planes (≈10^7 cm^-1) which, in polycrystalline materials differ in orientation from grain to grain. The slip planes may be part of various interacting slip systems which in different grains will have different resolved shear stresses and thus different dislocation behaviors. Hardening measurements thus represent some sort of average of many local dislocation maneuvers. The presence of irradiation defects sometimes further complicates this averaging by restricting the number of active slip planes to small groups, which are referred to as dislocation channels (13). Thus not all regions of a crystal are equivalent and this can give rise to an inhomogeneous deformation, the extent of which depends upon the amount of irradiation. The modeling of such a system would thus be a very formidable task as most of the grain interaction and slip system parameters are, quantitatively, not known, nor are the distribution functions available over which the averages could be made.

B. Mechanical Hardening

The basic approach to interpreting mechanical hardening, or the strengthening of metals as it is referred to when it is a desirable quantity, is to consider a somewhat classical dispersed barrier model. This model assumes a single, constant-line-tension dislocation on a single slip plane to be representative of the average dislocation behavior. When this dislocation, gliding under the applied resolved shear stress \( \tau \),
encounters a planar dispersion of barriers it will be pushed against them and restrained from further motion. Under the applied stress $\tau$, the dislocation then bows out between the barriers and after the suggestion by Orowan (14) can bypass the barriers when the stress becomes large enough for the bow-out to become a semicircle. In this configuration, the dislocation becomes unstable and can freely glide beyond the semicircle, provided no barriers lie adjacent to it. By this method the dislocation can bypass barriers by encircling them or groups of them and advance by what is termed the Orowan process. When the dislocation can always attain a semicircular configuration between some pair of barriers and thus move continuously, the applied shear stress is at an upper limit, termed the critical shear stress, $\tau_c$. This critical stress is thus the stress necessary to move the dislocation through the array of dispersed barriers. It represents an increase of the flow stress due to the presence of the barriers and, as will be shown, is inversely proportional to an effective interbarrier spacing, $L$.

Extensive use has been made of this model in the analysis of various types of hardening, such as solution hardening, precipitation hardening, work hardening and other types of hardening where, phenomenologically, dislocations are considered to be restrained by barriers of one kind or another. In the work hardening the glide dislocations on secondary slip
systems intersect the primary slip plane to create the barriers. A review of these applications of the model along with several variations is given by Brown and Ham (15). In assessing the various applications of the model they conclude that "although there are many reports of direct observations of dislocations looping between particles, or of flow-stresses in order-of-magnitude agreement with looping stresses calculated from observed particle spacings, convincing quantitative demonstrations of the Orowan process are few." They do add, however, that this lack of convincing demonstrations may be due more to the inability to perform ideal experiments with which to compare the theory, than to a weakness of the theory itself. A similar, but slightly more recent review by Nabarro (16), is more optimistic. He makes reference to the success of various recent statistical variations of the hardening model (17,18) indicating that statistical techniques are probably the best approach to modeling such complex systems.

One modification of this dispersed barrier hardening model often considered is to assume the dislocation, at some stress level below the critical shear stress, cuts through the barrier. This consideration introduces a barrier strength parameter to the hardening model. It also adds a second degree of freedom (the first degree of freedom being the effective barrier spacing along the dislocation, L - a
quantity very difficult to ascertain), allowing a much wider range of correlations between calculated and measured hardening values. This barrier strength parameter is, however, difficult to interpret and is often more a correlation coefficient than a parameter with physical significance as the barrier strengths are usually not well-defined.

The major difficulty in using the dispersed barrier model is in determining the appropriate effective barrier spacing along the dislocation, \( L \) (19). Much of the early work following Orowan's classical paper was concerned with this problem, usually resulting in some relation between \( L \) and the average interbarrier spacing on the slip plane, \( \bar{\lambda} \). Eventually a solution of this was found by considering a planar array of barriers and using a geometric analysis to statistically determine \( L \). Kocks (20,21) developed a statistical theory using this approach and Foreman and Makin (22,23) used a computer code to study the statistics of such a model. Although a line-tension model of a single dislocation on a single slip plane is still the basis of this hardening model, the dislocation can now encounter a spectrum of barrier sizes with a distribution of barrier strengths. Upon considering a large planar random array of point barriers, for example, a gliding dislocation will be pinned by many barriers as shown in Figure 1(a), giving rise to a distribution of interbarrier spacings along the dislocation, \( s_i \). A unique force will be
Figure 1. A line-tension model of a dislocation in a random array of barriers
produced on each barrier by the dislocation dependent upon the local dislocation configuration, as shown in Figure 1(b), where $T$ is the line tension of the dislocation and $F$ is the force. If the barriers are weak these forces will readily overcome the barrier restraint allowing the dislocation to move through them. When the barriers are strong, however, the dislocation can only move past them by the Orowan process. To do so, the largest interbarrier spacing along the dislocation becomes the "weakest link" and thus the controlling factor as this is where the Orowan process will operate. As the applied stress is increased the dislocation will bow through this opening and move on to a new stable configuration. The next controlling interbarrier spacing will thus be smaller and will ultimately approach the effective interbarrier spacing, $L$, as the stress rises to the critical shear stress or flow stress, $\tau_c$. $L$ is thus determined as the limit of a geometric simulation of the flow process. In the case of the weak barriers, the flow stress is controlled by the barrier strength so that $L$ no longer controls the flow process. $L$ then becomes only a length associated with the dispersed barrier model at some particular barrier strength. It follows, therefore, that this $L$ is a function of the dislocation configuration which is a function of the average planar interbarrier spacing, $\bar{\lambda}$, the barrier strengths, the applied stress and the unique combination of barriers pinning the disloca-
tion. Friedel (24) analytically derived an expression for \( L \) as a function of \( \bar{k} \) and \( \tau \), but Foreman and Makin (22,23) subsequently found it to be valid only for weak barriers.

Foreman and Makin used the above geometrical approach but determined the critical shear stress without obtaining an explicit value of \( L \). Their model is thus able to relate the flow stress or critical shear stress to the barrier array and to the barrier strength distributions directly. Although the barrier strengths are not explicitly known, various strength distributions can be postulated, their effect on the flow stress then being a parameter which can be studied. Foreman and Makin have made such studies (in (15),23). In addition, various other authors have since adopted their computerized model for more specific hardening studies (25) and for a more detailed investigation of the thermally activated motion of dislocations (26,27).

C. Dispersed Barrier Hardening Model

The model associated with the dispersed barrier hardening may be determined by considering a segment of a dislocation pressed against a series of barriers by a resolved shear stress \( \tau \). This shear stress produces a force normal to the dislocation causing it to bow between the barriers as shown in Figure 1(a). Ideally, the dislocation possesses an orientation dependence which would create an elliptically shaped bow-out (28,29). However, this dependence is ignored in the
constant-line-tension model and so a circular bow-out of radius $R$ is obtained. If $s$ is the chord length of the bow, then according to Orowan (14), the applied shear stress $\tau$ exerts a force $\tau bs$ on the arc of the bowed dislocation, where $b$ is the magnitude of the Burgers vector of the dislocation. Since the barriers restrain the dislocation, they must balance this force. From Figure 1(b) then, the force that the dislocation exerts on the barriers is given by

$$F = 2\tau \cos \frac{\phi}{2}$$

(1)

where $\tau$ is the line tension of the dislocation and $\phi$ is the angle between the bowed-out segments at the barrier. Considering Orowan's relation from above

$$F = \tau bs$$

(2)

and that the chord length is given by $s = 2R \cos \phi/2$, the radius of the bow may be obtained by eliminating $F$ to yield

$$R = \frac{T}{\tau b} = \frac{Gb}{2\tau}$$

(3)

where the line tension, $T$, has been replaced by an approximate energy per unit length of the dislocation (15,16), $Gb^2/2$, where $G$ is the shear modulus of the material. As the shear stress increases, the radius of curvature of the dislocation decreases until a semicircular configuration is reached. In a regular or square array of spacing, $\bar{\lambda}$, this configuration is a limiting condition such that $\tau$ is at a maximum, $\tau_{\text{max}}$, $\phi = 0$, $R = s/2 = \bar{\lambda}/2$ and the force has a maximum value,
\[ F_{\text{max}} = 2T. \] From Equations 1 and 2 we can write
\[ \tau = \frac{F}{bs} = \frac{T}{bR} = \frac{Gb}{2R} \] (4)
and therefore when \( \tau = \tau_{\text{max}} \)
\[ \tau_{\text{max}} = \frac{F_{\text{max}}}{bs} = \frac{Gb}{\bar{\lambda}} \] (5)
This is a local critical or breaking shear stress and applies to a pair of barriers of spacing \( \bar{\lambda} \) or to a regular square array of average planar interbarrier spacing \( \bar{\lambda} = 1.0/\sqrt{N}, \) where \( N \) is the number of barriers per unit area. This is the classical dispersed barrier hardening model.

Upon considering a random array of barriers, however, as can be seen from Figure 1(c), \( \phi \) can approach zero or some limiting value, \( \phi_c, \) at stresses (bow-outs) less than the critical shear stress, \( \tau_{\text{max}}, \) for the square array. In such cases the dislocation is able to pass a barrier at a lower stress level and thus at a radius of curvature \( R > \bar{\lambda}/2. \) Ultimately the entire array may be traversed at a lower stress we shall call the critical shear stress, \( \tau_c, \) which we can express, analogous to Equation 5, as
\[ \tau_c = \frac{F_{\text{max}}}{BL_o} = \frac{Gb}{L_o} \] (6)
where \( L_o \) is an effective length (\( L_o > \bar{\lambda} \)) characteristic of the larger radius of curvature. When the barriers have strengths less than the maximum, say \( F_c, \) then from Equation 1 the bow-out angle will have a minimum, \( \phi_c, \) which we shall refer to as
the critical cutting angle
\[
\phi_c = 2\cos^{-1}\left(\frac{F_c}{2G}\right) = 2\cos^{-1}\left(\frac{F}{Gb^2}\right)
\]  
(7)

The critical shear stress for a random array of such barriers will, then, from Equation 6, with \(F_{\text{max}}\) replaced with \(F_c\), be
\[
\tau_c = \frac{F_c}{bL_0} = \frac{2T}{bL_0}\cos\frac{\phi_c}{2} = \frac{Gb}{L_0}\cos\frac{\phi_c}{2} = \frac{Gb}{L}
\]  
(8)

where we have written
\[
L = L_0/\cos\frac{\phi_c}{2}
\]  
(9)

and \(L\) is the effective interbarrier spacing for a random array of critical cutting angle \(\phi_c\). This is the same \(L\) as was described above in subsection II-B and which is so difficult to calculate. The last forms of Equations 5 and 8 are often used interchangeably in the literature (19), the \(\lambda\) and \(L\) values being incorrectly equated. To avoid ambiguity we shall write Equation 8 in the form of Equation 5 as
\[
\tau_c = \frac{\lambda Gb}{L\lambda} = K\frac{Gb}{\lambda}
\]  
(10)

where \(K\) is a parameter varying from 0 to 1 which depends upon the randomness of the array, the strength of the barriers and the applied stress. The quantity \(\bar{\lambda}\) is still the average planar interbarrier spacing \(1/\sqrt{N}\), where \(N\) is the number of barriers per unit area. Equation 10 may also be written in the form
\[ K = \frac{\tau_c}{Gb/\lambda} \]

In which \( K \) is considered as a normalized critical shear stress. In section III, \( K \) will be determined in this form with a computer calculation.

The critical shear stress, \( \tau_c \), is the shear stress required to move a dislocation through an array of point barriers. It is assumed to be independent of the existing yield mechanisms without the barriers such that \( \tau_c \) represents an increase of the hardness due to the presence of the barriers. More correctly, then, the critical shear stress should be written as \( \Delta\tau_c \).

In the analysis of mechanical hardening it has been found advantageous to divide the yield stress into two components, a thermal or effective stress and an athermal stress. The athermal component is normally considered to be that stress required to overcome long-range internal stresses and to initiate slip. It is a function of only the elastic parameters and is virtually independent of temperature. The thermal component is the difference between the applied stress and the athermal component and is assumed to be temperature dependent in the bcc lattices. It is also considered to be an effective stress as it is that part of the applied stress which is available to assist the dislocations in surmounting the barriers. In the analysis of the dispersed barrier hardening
it is generally assumed justifiable to simply add the hardening, \( \Delta \tau_c \), to the above components.

D. Temperature Dependence

One of the more controversial aspects of mechanical behavior is that of the temperature sensitivity. The yield stresses, for instance, of fcc and bcc metals exhibit very different temperature dependencies, the yield stress for the bcc lattices showing a larger increase with decreasing test temperature than the yield stress for fcc metals. Possible mechanisms for this behavior have been proposed but a general acceptance of them has not been found. The addition of strengthening or hardening agents to these metals often modifies the temperature dependence of their yield stresses considerably, making identification of the hardening mechanisms extremely difficult.

Techniques to study the temperature effects have been developed around a quasi-thermodynamic, reaction rate theory (30,31,32). Currently, two mechanisms based on this theory are considered, one in which an energy variation between lattice planes, the Peierl's potential, gives rise to a friction-type drag on the moving dislocation (33), and the other is a barrier-type impediment (15,16,24) similar to the dispersed barrier hardening model. In this latter thermal model it is assumed that the dislocation-barrier interactions are of an energy comparable to the thermal energies such that the bar-
riers may be overcome with the assistance of thermal fluctuations of the dislocation. If a dislocation is held against a barrier by an effective shear stress \(\tau\), then the inherent thermal vibrations of the lattice produces a vibration of the dislocation on its glide plane. By the reaction rate theory the dislocation will jump the barrier with a probability \(\exp(-Q(\tau)/kT)\) where \(Q(\tau)\) is the activation energy for the event at the stress \(\tau\) and temperature \(T\) and \(k\) is the Boltzmann constant. \(Q(\tau)\) is a measurable parameter and its stress sensitivity, \(v\), the activation volume, is also measurable, such that

\[
Q(\tau) = Q_o - v\tau \tag{12}
\]

where \(Q_o = Q(\tau=0)\) is the activation energy at zero effective stress. (For a more rigorous interpretation, see Makin (34)).

These activation parameters are used to compare the plastic flow characteristics of different metals or of different states of the same metal. These differing states may include, for example, different impurity contents, the effects of precipitate particles or of irradiation. The variations of these activation parameters show the sensitivity of the plastic flow mechanisms to the lattice imperfections or dislocation barriers. A symposium has been held on this topic, the papers of which give a very comprehensive overview of the subject and the current state-of-the-art of the theory of rate processes (35).
The dispersed barrier hardening model using the Orowan process is an athermal model, giving the same amount of hardening at 0ºK as at room temperature. The barriers are considered to be too large of an energy barrier for the dislocation to cut or surmount by thermal activation.

E. Radiation Hardening

The early approaches to analyzing radiation hardening (RH) centered around various postulated damage structures in the lattice, some theories of which are presented in reference (36). One theory, by Seeger (37), based on a dispersed barrier hardening by "depleted zones" formed from damage spikes, is still often considered valid. With the introduction of transmission electron microscopy (TEM) and the subsequent micrographs of the damage structures, interpretation of RH has tended towards mechanisms based upon the TEM-observable defects. However, as pointed out by Diehl (38) in a review of the hardening of fcc metals by quenching and irradiation, this approach has not been completely satisfactory.

Extensive RH studies have been carried out in some of the fcc metals, especially copper, and although the TEM-observable defects contribute to the hardening, the temperature sensitivity of the RH requires a more sophisticated interaction
mechanism than that which can be achieved with a dispersed barrier model. Experimentally it is determined that in the irradiated fcc metals, the variation with temperature of the activation energy, $Q_o$, is too large to be explained by current hardening theories (34). This variation is considered to indicate (34,39,40) that the dislocation barriers are composed of a spectrum of strengths rather than an array of constant strength barriers. Several theories based on this barrier spectrum approach have been developed (41,42,43) with a good degree of success. Another approach (44) has continued to use the basic dispersed barrier model, despite the barrier spectrum conclusion, with a good degree of correlation also. However, this last approach requires, according to Diehl (38), rather arbitrary assumptions.

Hardening studies in irradiated bcc metals have not been as exhaustive as with the fcc metals. A review of RH in bcc refractory metals by Wechsler (8) indicates that the hardening exhibits only a slight temperature dependence over and above the large temperature dependence already inherent in the bcc lattices. A detailed study of the rate determining processes in neutron-irradiated vanadium by Boček et al. (45) has shown this to be a valid conclusion at low fluence levels only (where the fluence is the time-integrated neutron flux). At higher fluences (above $5.0 \times 10^{18}$ n/cm$^2$, E>1 MeV), the effective or thermal stress develops a fluence dependence indicating a
change in the rate controlling mechanism (45,46). Arsenault (47) initially concluded that the rate controlling mechanism in bcc metals was not changed by neutron irradiation but later reversed himself (48) in showing that much of the thermal mechanism is associated with the presence of the interstitial impurity atoms. This can also be seen to some extent in the hardening studies of Wechsler et al. (49) on irradiated vanadium where the yield-stress-temperature relations show considerable sensitivity to the oxygen concentration. The analysis of RH in bcc metals is thus strongly associated with the interstitial impurity atom concentration and it is the contribution of these atoms to RH which must be resolved to ascertain the mechanisms of RH and RAH.

The hardening of metals by neutron irradiation often occurs at an ever decreasing rate with increasing fluence such that the measured hardness shows a limiting or saturation value (39). Loomis and Gerber (50) have found, however, that the resulting plateau in the hardening-fluence curve may, depending upon the impurity atom content, be fairly small, the hardening again increasing thereafter. Nevertheless, preliminary analysis tends to indicate that the thermal sensitivity of the effective shear stress begins to develop at fluences approaching this saturation limit. For example, roughly comparing different workers results: in polycrystalline vanadium, Bocek et al. (45) indicate that "the effective
stress is changed to a major extent only at doses above $5 \times 10^{18}$ n/cm$^2$, whereas Shiraishi et al. (51) show a saturation fluence of about $2.5 \times 10^{19}$ n/cm$^2$; in single crystal niobium, Tucker and Wechsler (52) show a saturation fluence of about $9 \times 10^{17}$ n/cm$^2$ whereas Ohr et al. (12) show significant temperature sensitivities at and above a fluence of $2 \times 10^{18}$ n/cm$^2$ (all fluences, E>1 MeV). In high purity iron, however, the hardening appears athermal well past the saturation fluence (53,54). Since vanadium and niobium have been considered for reactor core materials, their end-of-life fluences could approach $10^{23}$ n/cm$^2$. This is certainly beyond the range of athermal hardening in these materials. In low alloy steels, however, which are used typically in light water reactor pressure vessels, the maximum end-of-life fluences are of the order of $1-5 \times 10^{19}$ n/cm$^2$. This fluence is still in the range of athermal hardening (55), so the dispersed barrier hardening model would be suitable for correlating irradiation behavior in pressure vessel steels.

F. Defect Stability

An area of this project which is not very amenable to experimental investigation but which is of extreme relevance is that of the stability of the defect loops during deformation. It is generally considered that the loops are immobile or sessile and that they create rigid barriers to the gliding dislocations. However, various studies have shown this may
not be the case. Brimhall and Mastel (56) working on molybdenum have reported that during low temperature anneals (170°-200°C) some small loops below a hundred angstroms in diameter shift position within the foils and some glide out of the foils along their glide cylinders. Silcox (57) has reported observing by TEM a straight glide dislocation pushing a loop of several hundred angstroms diameter and Makin (58) has shown a micrograph of a glide dislocation "sweeping-up" loops. Makin has done an analytical study of the long-range forces between dislocation loops and glide dislocations and considers loop mobility to be a very real possibility, especially when one considers that dislocation channels are cleared of the TEM-observable defects during deformation. Makin's analysis is for fcc copper so if one considers the larger number of possible slip planes in the bcc lattice, a higher mobility may be possible in the bcc metals. Thus it appears difficult to justify complete defect loop immobility. However, in the fcc systems, some of the loops are considered to possess a stacking fault across the area of the loop (Frank-type loops) (9) which would greatly retard their mobility. In the bcc systems the loops are considered to unfault (59) and thus are purely prismatic. But in the bcc lattices another phenomenon, analogous to strain aging, can occur in which mobile interstitial impurity atoms diffuse to form atmospheres or Cottrell clouds (60) about the loop dislocations and thereby effect,
at least, a partial immobilization. These quasi-sessile loops could give rise to RH, the amount of hardening dependent upon the extent of impurity atom cloud formation. Subsequent post-irradiation annealing could also give rise to impurity atom diffusion to the loops (this has been measured in niobium (61) and vanadium (62)), either creating the clouds or increasing their density so as to further immobilize the loops and thus provide a strengthened barrier. This would then give rise to RAH. These concepts will be considered again in Section IV as a partial explanation of the results of this investigation.
III. HARDENING ANALYSIS

A. Dispersed Barrier Computer Code

The starting point of this study is to interpret the TEM observable radiation produced defects as dislocation loops. These loops are considered to intersect the glide planes and act as dispersed barriers to the glide dislocations. If the density of defects is \( n \text{ cm}^{-3} \) and the average loop diameter is \( \bar{d} \text{ cm} \), then the number of loops which intersect a slip plane per unit area would be \( N = n\bar{d} \text{ cm}^{-2} \). Since the defects are loops, however, they generally intersect the glide plane in two places giving rise to a pair of point barriers. The spacing of these pairs of point barriers varies with the size of the loop and with the distance between the loop center and glide plane. The \( N \) defects per unit area of glide plane thus give rise to \( 2N \) point barriers from which we can obtain an average interbarrier spacing on the glide plane, \( \bar{\lambda} \), as

\[
\bar{\lambda} = \frac{1}{\sqrt{2N}} = \frac{1}{\sqrt{2n\bar{d}}} \tag{13}
\]

In subsection II-C it was shown how a planar array of barriers of average interbarrier spacing, \( \bar{\lambda} \), could give rise to a hardening increment in the form of a critical shear stress, \( \Delta\tau_C \), given by

\[
\Delta\tau_C = K \frac{Gb}{\bar{\lambda}} \tag{14}
\]

To study the effect of the spacing of the barrier pairs on
this critical shear stress, a computer code was written based on the dispersed barrier hardening model. The code determines values of $K$ as a function of the barrier strength and the barrier pair spacing, which can then be used to determine values of $\Delta \tau_c$. A brief description of the code methodology will follow to illustrate the procedure by which the code determines $K$ and to indicate how the barrier pairs are produced in the code. A short flow chart of the code is given in Appendix A.

The code procedure, basically, is to construct a 2-dimensional array of barriers and then determine what shear stress is required to enable the constant-line-tension dislocation to move continuously through the array. Since the shear stress is inversely proportional to the radius of curvature of the dislocation by Equation 3, the problem is a geometric one of determining what radius of curvature and thus what extent of bow-out is required to allow the dislocation to always find an opening and keep moving. At a shear stress below the critical limit, the dislocation will be pinned at many points giving rise to what we shall refer to as a stable or stationary configuration. Along this configuration there will be many interbarrier spacings or chords and a bow-out angle $\phi$ at each barrier, as shown in Figures 1(a) and 1(b). One of these chords will be a maximum and one of the $\phi$'s will be a minimum such that as the radius of curvature decreases
with increasing stress, either the bow-out at the maximum chord will become a semicircle or the minimum \( \phi \) will decrease to below \( \phi_c \). In the first case the semicircle will be an unstable configuration and will bow out like a Frank-Read source thus freeing the configuration to move beyond the pinning barriers. In the second case the barrier with \( \phi < \phi_c \) would be cut or surmounted, also freeing the configuration to move beyond the pinning barriers. In either case, the change of curvature necessary to make either of these a local critical value can be calculated. Whichever change is smaller is used to calculate an increment of shear stress which, when applied, allows the dislocation to cut through or by-pass this "weakest link" and continue to move. Movement of the dislocation is effected by scanning from this "weakest link" barrier to find the next closest barrier and then to assume the dislocation to be pinned there. If all chords are shorter than the limiting value and all \( \phi \)'s are greater than their respective limits (\( \phi_c \)'s), then a stationary configuration is achieved. Otherwise the dislocation can surmount some barrier and move progressively, by scanning, to the next barrier. Once a stationary configuration is achieved, then, as above, the shear stress is incremented to the level just necessary to free the configuration and the scan cycle starts again. When the dislocation continues to move through the array, i.e. no configuration of barriers can pin it, then the shear
stress is considered to be at the critical limit. The statistics of this geometric model arise as a size effect. The larger the array, the more barrier points lie along a stationary configuration of the dislocation, such that, on the average, a more precise critical shear stress will be determined. It was found that for a random array of point barriers a variation of less than 5% in $K$ was possible with 1000 points. For the barrier pairs it was found that a planar array of 5000 points was required for this range of standard deviation on $K$. The output of the code is in terms of $\Delta \tau_c$ normalized to $Gb/\lambda$ which is equal to $K$ as shown in Equation 11. With $\tau$ replaced by $\Delta \tau$ in Equation 3, comparison of Equation 3 with Equation 11 shows also that $K = \lambda/2R$, i.e. $K$ is one-half the reciprocal of the normalized radius. This relation is used in the code, the radius being gradually reduced until the dislocation can move continuously through the array. $R$ is then at the critical limit and gives rise to the value of $K$. Since the output is normalized, the dimensions used within the code are somewhat arbitrary, limitations being imposed from the partial use of integer variables. A dislocation in a typical array of barrier pairs is shown in Figure 2, the critical cutting angle, $\phi_c = 0.0$ for all barriers and the normalized critical shear stress, $K = 0.633$. All the barriers in each array were considered to have the same barrier strengths which were varied in terms of $\phi_c$ from 0.0 to 3.0
Figure 2. Typical random array of barrier pairs with the critical cutting angle, $\phi_c = 0$ and a normalized critical shear stress $K = 0.63$
radians in increments of 0.5 radians. A K value was obtained with the code for each array at these barrier strengths and for several different random arrays in order to obtain an average K at each $\phi_c$. It was observed that the standard deviation on K varied with the barrier strengths, being largest around $\phi_c = 0.5$ radians for the single barrier arrays. This largest standard deviation shifted to $\phi_c = 1.0$ radians for the paired barrier arrays. The explanation for this will be given below.

The analysis was initiated by considering a random array of single point barriers. Values of $K = K(\phi_c)$ were obtained with the code and compared favorably with the results of Foreman and Makin (22, 23) for a similar array as shown by the square symbols in Figure 3. To consider the barrier pairs, they were generated in the code by constructing a random array of single points and then locating a pair of barriers symmetrically about each random point, the orientation of each pair being in either of two orthogonal positions. This orientation dependence will be discussed later. Since the code operates with arbitrary dimensions, the spacings of the barrier pairs were considered as fractions of the average interbarrier spacing in the code, $\bar{x}_a$. From Bajaj (11), typical defect cluster loop sizes were of the order of 10% to 20% of their respective $\bar{x}$'s (see Equation 13) so that typical maximum barrier pair spacings would be of the order of 10% to
Figure 3. Normalized critical shear stress, $K(\phi_c)$ as obtained by the computer code for various critical cutting angles, $\phi_c$, and different barrier pair spacings, $\delta_a$. The square symbols denote the results of Foreman and Makin (22,23).
20% of \( \bar{\lambda} \) and, thus, of \( \bar{\lambda}_a \). That is, if \( \delta \) is the barrier pair spacing on the slip plane and \( \delta_a \) is the barrier pair spacing in the code, then \( \delta/\bar{\lambda} = \delta_a/\bar{\lambda}_a = 0.1 \) to 0.2.

The next step of the study was to consider the effect of the constant spacing barrier pairs on the critical shear stress. Figure 3 shows the resulting \( K(\phi_c) \) values for constant pair spacings of 10% and 30% of \( \bar{\lambda} \). Also shown in Figure 3 are the \( K(\phi_c) \) values for the single point barrier array. The resulting \( K(\phi_c) \) curve for this latter array is drawn twice, the lower curve differing from the upper by a factor of \( \sqrt{2} \). If the upper curve is representative of an array of 2N point barriers, then, the lower curve represents the change in \( K(\phi_c) \) when the density of barriers, 2N, is halved, i.e. reduced to N barriers. Also, the lower curve represents the reduction of \( \Delta \tau_c \) and thus the softening obtained from halving the barrier density. The lower curve represents the case when the barrier pairs have been reduced to a spacing of zero width and thus appear as single point barriers. Hence it is a lower limit for \( K(\phi_c) \). The upper curve represents the \( K(\phi_c) \) values when the 2N point barriers are totally random, i.e. the barrier pair spacings have become so large that the relationship of the pairs is lost in the array. This curve thus represents an upper limit of the \( K(\phi_c) \) values such that the two curves together form an envelope inside of which the \( K(\phi_c) \) values for the paired barrier arrays must lie.
The results of the constant spacing barrier pairs lie well within this envelope and as shown in Figure 3, asymptotically approach the upper curve at decreasing barrier strengths, i.e. increasing \( \phi_c \)'s. An interpretation of this behavior is that the hardening produced by the strong barrier pairs is proportional to the pair spacing but the hardening produced by relatively weak barrier pairs is virtually independent of the pair spacing. A more mechanistic interpretation is that with the strong barriers the interbarrier spacing along the dislocation controls the yield or flow stress, not the barrier strength. Thus, due to the barrier pairs, the Orowan mechanism operates at barrier strengths considerably less than the maximum. This gives rise to the somewhat flat \( K(\phi_c) \) at high barrier strengths. Between critical cutting angles, \( \phi_c \), of about 1.0 to 1.5 radians there appears to be a transition from the Orowan mechanism to the barrier cutting mechanism and it is during this transition region that the variance of \( K \) becomes significantly larger. In the single point barrier arrays this transition occurs as soon as the barrier strength begins to decrease so the wider variance of \( K \) in this case is observed mainly at \( \phi_c = 0.5 \) radians, as indicated previously. The results in Figure 3 thus show the effect of constant spacing barrier pairs on the normalized hardening coefficient \( K \) and its subsequent variation with the strength of the barriers.

The barrier pair spacing distributions were obtained from
the dislocation loop size distributions. The defect cluster-
dislocation loop size distributions were obtained from Bajaj
(Figures 14, 16 and 18 in reference 11, or see (63)) and are
shown in Figures 4, 5 and 6. Each of these figures is for a
different oxygen level in vanadium, these being 95, 300 and
500 wt ppm, with each level representing different controlled
amounts of interstitial impurity atoms. Since all samples
were irradiated under identical conditions, at a temperature
of 95°C to a fluence of $1.4 \times 10^{19}$ n/cm$^2$, $E>1$ MeV, the varia-
tion in the loop density and size distributions are a result
of only the differing impurity concentrations. The total dis-
location loop densities of the samples, $n$, and their average
diameters, $\bar{d}$, are shown in Table 1 (from Table 4 in reference
11) as are the average interbarrier spacings, $\bar{\lambda}$, calculated
with Equation 13 and the ratio $\bar{d}/\bar{\lambda}$. (Bajaj, in his Table 4,
also shows an "average interbarrier distance," but relative
to this work, it is more rigorously an average planar inter-
defect distance and is $\sqrt{2}$ larger than our $\bar{\lambda}$). Also shown in
Figures 4, 5 and 6 are the loop size distributions following
1 hour, post-irradiation anneals at the indicated tempera-
tures. The corresponding changes in the loop density, aver-
age loop diameter and average interbarrier spacing are shown
in Table 1. The loop distribution data points used were taken
from the points plotted in the figures. However, the ordi-
nates are in the form of differential densities and had to be
Figure 4. Defect cluster density size-distribution function $n'$ vs defect cluster size in as-irradiated and post-irradiation annealed (to $400^\circ$C) vanadium - 95 ppm oxygen. Refs. (11,63)
Figure 5. Defect cluster density size-distribution function $n'$ vs defect cluster size in as-irradiated and post-irradiation annealed (to 400°C) vanadium - 300 wt ppm oxygen. Refs. (11,63)
Figure 6. Defect cluster density size-distribution function \( n' \) vs defect cluster size in as-irradiated and post-irradiation annealed (to 400°C) vanadium - 500 wt ppm oxygen. Refs. (11,63)
Table 1. Defect cluster parameters from TEM micrographs of as-irradiated and post-irradiation annealed single crystal vanadium

<table>
<thead>
<tr>
<th>Oxygen conc. (wt ppm)</th>
<th>T (°C)</th>
<th>$n^a$ (10^{15} cm^{-3})</th>
<th>$d^a$ (Å)</th>
<th>$\bar{d}$ (Å)</th>
<th>$\bar{d}/\bar{\bar{d}}$</th>
</tr>
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<td>136</td>
<td>730</td>
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<td>778</td>
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<td></td>
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<td>1421</td>
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</tr>
<tr>
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<td>77</td>
<td>608</td>
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<td></td>
<td>0.70</td>
<td>271</td>
<td>1626</td>
<td>0.167</td>
</tr>
</tbody>
</table>

\(a\) From reference 11.
converted to a histogram-type representation. Since the plotted points in Figures 4-6 are virtually equally spaced along the abscissa, this spacing was used for the loop diameter group widths such that when multiplied by the differential densities gave a loop density, \( n_i \), at an average diameter, \( d_i \), over the \( i \)th diameter group width. This information was entered into the code in terms of the planar density, \( N_i (= n_i d_i) \), and \( d_i \), along with suitable scaling parameters, such as \( \bar{\lambda}/\bar{\lambda}_a \), and was used to produce the barrier pair spacings. These spacings were determined by using a random number between 0 and \( d_i/2 \) to represent the distance of the center of each loop from the glide plane and then calculating the spacing, \( \delta_a \), where the loop of diameter \( d_{i,a} (= d_i \bar{\lambda}_a/\bar{\lambda}) \) intersected the plane. The resulting \( K(\phi_c) \) values are plotted in Figures 7, 8, 9. Also included in these figures are curves representing the constant spacing barrier pairs, 10% and 30% of \( \bar{\lambda} \), from Figure 3. As can be seen from the figures, the effect of the barrier pair spacing distributions and thus of the loop size distributions on \( K(\phi_c) \) is minimal. The results are virtually the same as those which would be obtained by considering the barrier pair spacings constant with a spacing equal to the average loop diameter of each distribution. In effect, the code statistically determined the average barrier pair spacing for each distribution in terms of \( K(\phi_c, \bar{d}) \). Since the loop size distributions effect very little change of
Figure 7. Normalized critical shear stress, $K(\phi_C)$, as obtained by the computer code for various critical cutting angles, $\phi_C$, and a distribution of barrier pair spacings obtained from the defect loop size distributions for the 95 wt ppm oxygen samples.
Figure 8. Normalized critical shear stress, \( K(\phi_c) \), as obtained by the computer code for various critical cutting angles, \( \phi_c \), and a distribution of barrier pair spacings obtained from the defect loop size distributions for the 500 wt ppm samples.
Figure 9. Normalized critical shear stress, $K(\phi_c)$, as obtained by the computer code for various critical cutting angles, $\phi_c$, and a distribution of barrier pair spacings obtained from the defect loop size distributions for the 500 wt ppm oxygen samples.
$K(\phi_c)$, the mechanical hardening will be a function mainly of $\bar{\lambda}$ and slightly of $\bar{\alpha}$.

B. Hardness Measurements

The material used by Bajaj to obtain the TEM micrograph-defect size measurements was single crystal vanadium. Small cylinders of this material, 0.5 mm thick and 3 mm in diameter, were tested with a microhardness tester containing a diamond pyramid indenter, the results of which (from Figure 12 in reference 11) are shown in Figure 10. The majority of Bajaj's hardness measurements, however, were made in terms of room temperature tensile yield stresses on polycrystalline vanadium samples. Again, three levels of oxygen were considered, 60, 205 and 640 wt ppm, which varied somewhat from the single crystal samples, as did the irradiation conditions of 105°C and $1.2 \times 10^{19}$ n/cm$^2$, E>1 MeV. For the tensile tests, Bajaj used a deformation rate of $1.67 \times 10^{-4}$ sec$^{-1}$ and he reported the lower yield stress with the 0.2% offset strain being used when the yield drop was absent. The results of Bajaj's yield stress measurements (from Figure 9 in reference 11) are shown in Figure 11 and the impurity atom concentrations of both the single crystal and the polycrystalline samples are tabulated in Appendix B.

Since the hardening determined by the dispersed barrier model is in terms of a shear stress, it is necessary to con-
Figure 10. Diamond pyramid hardness vs annealing temperature for unirradiated and irradiated single crystal samples. Irradiation dose $1.4 \times 10^{19}$ n/cm$^2$ (E>1 MeV) at 95°C. Ref. (11)
Figure 11. Lower yield stress vs annealing temperature for unirradiated and irradiated polycrystalline samples. Irradiation dose and temperature: $1.2 \times 10^{19}$ n/cm$^2$ ($E>$1 MeV) at 106°C. Test temperature 300°K, annealing time, 1 hr. Ref. (11)
vert the uniaxial tensile yield stress to shear stresses. Also, since the material is polycrystalline there will be a multiplicity of active slip systems, so that some sort of average slip behavior is required to correlate the yield stress with an average critical resolved shear stress. Taylor (64) was the first to recognize this problem and recent expositions are given by Backofen (65) and Chin (66). Ideally, homogeneous plastic deformation occurs on a plane 45° to the tensile axis. The shear stress is a maximum on this plane, and is given by

$$\tau_c = \frac{1}{2} \sigma_y$$

where $\tau_c$ is the critical resolved shear stress (CRSS) and $\sigma_y$ is the uniaxial tensile yield stress. In a single crystal, a geometric factor, the Schmid factor, is used to make this correlation which varies from $1.0/3.674$ to $1.0/2.0$, i.e. from 0.272 to 0.5, depending upon the slip direction on the slip plane. In a polycrystalline specimen where the grains are randomly oriented there will be a random distribution of Schmid factors required to make each grain slip. Since the standard stereographic triangle encompasses all possible orientations, an average of the Schmid factor over this triangle leads to an average factor of $1.0/2.24$ (65, page 73), such that

$$\bar{\tau}_c = \frac{1}{2.24} \sigma_y = 0.446 \sigma_y$$

(15)
where $\bar{\tau}_c$ is an average critical resolved shear stress. But this average factor is at most an upper limit as the deformation of a polycrystalline material introduces an additional constraint in that the grains must adhere to one another during deformation. This adherence requires a strain compatibility between grains which can only be realized by the simultaneous operation of many slip systems. This behavior is contained in another geometrical factor, termed the Taylor factor, which is analogous to the inverse Schmid factor and equates to it in some cases. Averaged over the standard stereographic triangle for mixed <111> slip on the \{110\}, \{112\} and \{123\} planes of the bcc lattice, the Taylor factor has a value of 2.754 (65), such that

$$\bar{\tau}_c = \frac{1}{2.754} \sigma_y = 0.363 \sigma_y$$ (16)

The average factor for each of the planes individually is slightly higher. Thus, to correlate the tensile uniaxial yield stress with the calculated critical shear stress, $\tau_c$, we assume that $\tau_c$ is equal to the average CRSS, $\bar{\tau}_c$. There is, therefore, effectively a range of possible correlation coefficients varying from 0.363 to 0.446, depending upon the sample size and degree of intergranular restraint. For round samples with the diameter large relative to the grain diameter, the axisymmetric deformation mode of the uniaxial tensile test requires that the lower limit, Equation 16, be imposed. This form shall be used in the following correlations.
Another adjustment which must be made to effect the hardness correlations is that concerning the differing oxygen concentrations of the samples. The hardness calculations based on the TEM-observed defect structures were for oxygen levels of 95, 300 and 500 wt ppm whereas the yield stress hardness measurements were for oxygen levels of 60, 205 and 640 wt ppm. Since the hardness values are fairly strong functions of the impurity atom concentrations, one of these sets of hardness values must be converted to the oxygen level of the other. By converting the measured hardening to the oxygen levels of the calculated values, a better conversion is achieved as interpolation can be used. The measured hardening values were taken from Figure 11 as the difference between the irradiated and unirradiated yield stresses at temperatures corresponding to the TEM-foil annealing temperatures, i.e. in 100°C increments from 100°C which is the as-irradiated condition. These hardening values are listed in Table 2 with the corresponding shear stress increments as calculated with Equation 16. The shear stress increments are plotted as functions of the oxygen concentration in Figure 12 from which representative measured hardness values at 95, 300 and 500 wt ppm oxygen were obtained. These data are tabulated in Table 3 along with the corresponding $\hat{l}$ from Table 1 and the calculated hardening, $\Delta \tau_c / K$. 
Table 2. Measured yield stress increments and their shear equivalences (MPa)\textsuperscript{a}

<table>
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<tr>
<th>T (°C)</th>
<th>Oxygen concentration (wt ppm)</th>
<th>( \Delta \sigma_y )</th>
<th>( \Delta \tau_C )</th>
<th>( \Delta \sigma_y )</th>
<th>( \Delta \tau_C )</th>
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<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

\textsuperscript{a}\( \Delta \tau_C \) calculated from Equation 16 as \( \Delta \tau_C = 0.363 \Delta \sigma_y \).
Figure 12. Measured hardening values as shear stress increments and as a function of oxygen concentration; data points from Table 2
Table 3. Summary of hardening analysis

<table>
<thead>
<tr>
<th></th>
<th>$\frac{g}{\varepsilon}$ (Å)</th>
<th>$\Delta \tau_c/K^a$ (MPa)</th>
<th>$\Delta \tau_c^b$ (MPa)</th>
<th>K</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>95 wt ppm O</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>As-irrad.</td>
<td>740</td>
<td>89.1</td>
<td>36.5</td>
<td>0.410</td>
</tr>
<tr>
<td>200°C anneal</td>
<td>730</td>
<td>90.3</td>
<td>44.1</td>
<td>0.488</td>
</tr>
<tr>
<td>300 °C anneal</td>
<td>778</td>
<td>84.8</td>
<td>47.5</td>
<td>0.560</td>
</tr>
<tr>
<td>400 °C anneal</td>
<td>862</td>
<td>76.5</td>
<td>48.1</td>
<td>0.629</td>
</tr>
<tr>
<td>500 °C anneal</td>
<td>1421</td>
<td>46.4</td>
<td>44.1</td>
<td>0.951</td>
</tr>
<tr>
<td>600 °C anneal</td>
<td>n.a.</td>
<td>14.9</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td><strong>300 wt ppm O</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>As-irrad.</td>
<td>608</td>
<td>108.5</td>
<td>44.8</td>
<td>0.413</td>
</tr>
<tr>
<td>200°C anneal</td>
<td>614</td>
<td>107.4</td>
<td>57.5</td>
<td>0.535</td>
</tr>
<tr>
<td>300 °C anneal</td>
<td>615</td>
<td>107.3</td>
<td>62.3</td>
<td>0.581</td>
</tr>
<tr>
<td>400 °C anneal</td>
<td>645</td>
<td>102.2</td>
<td>62.5</td>
<td>0.612</td>
</tr>
<tr>
<td>500 °C anneal</td>
<td>1039</td>
<td>63.5</td>
<td>54.6</td>
<td>0.860</td>
</tr>
<tr>
<td>600 °C anneal</td>
<td>2157</td>
<td>30.6</td>
<td>19.0</td>
<td>0.620</td>
</tr>
<tr>
<td><strong>500 wt ppm O</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>As-irrad.</td>
<td>537</td>
<td>122.8</td>
<td>50.3</td>
<td>0.410</td>
</tr>
<tr>
<td>200°C anneal</td>
<td>550</td>
<td>119.9</td>
<td>63.7</td>
<td>0.531</td>
</tr>
<tr>
<td>300 °C anneal</td>
<td>523</td>
<td>126.1</td>
<td>69.8</td>
<td>0.553</td>
</tr>
<tr>
<td>400 °C anneal</td>
<td>568</td>
<td>116.1</td>
<td>69.4</td>
<td>0.597</td>
</tr>
<tr>
<td>500 °C anneal</td>
<td>962</td>
<td>68.5</td>
<td>56.0</td>
<td>0.817</td>
</tr>
<tr>
<td>600 °C anneal</td>
<td>1626</td>
<td>40.5</td>
<td>18.5</td>
<td>0.456</td>
</tr>
</tbody>
</table>

\(^a\)Calculated from Equation 17.

\(^b\)Interpolated from measured values in Figure 12.

\(^c\)n.a. - not available.
C. Correlations

One final assumption required to make the hardness correlations is with regards to the density of effective barriers. It is generally accepted that the dislocation loops in bcc metals are prismatic with Burgers vector $\mathbf{b} = a_0/2[111] \ (67)$ where $a_0$ is the lattice parameter and that they lie on the $\{111\}$ planes (50,51). Assuming slip in bcc metals to occur primarily on the $\{110\}$ planes, the loops will intersect the slip planes such that half are normal and half are inclined at an angle of 35.26°. Since the loops inclined to the slip plane would produce jogs in the glide dislocation, we shall assume for now that only these are active barriers, the loops normal to the plane being a much weaker barrier. With this assumption, then, and the inclination of the loops, the number of active loops intersecting the slip plane is reduced to $N/(2\sqrt{3})$, where $N$ is the planar defect density. For the problem under consideration the calculated dispersed barrier hardening will be, from Equation 14, given by

$$\Delta \tau_c = \frac{1}{\sqrt{2\sqrt{3}}} K \frac{G b}{\bar{\lambda}} = 0.537 K \frac{G b}{\bar{\lambda}} \tag{17}$$

where $\bar{\lambda}$ is still defined by Equation 13. For vanadium, $Gb = 1.23 \times 10^5 \ \text{MPa Å} \ (1.78 \times 10^4 \ \text{kpsi Å})$ and the values of $\Delta \tau_c$ are tabulated in Table 3. It is realized that a range of averaging coefficients could have been used in Equations 16 and 17, but those chosen will, as shown later, lead to a possible
interpretation of the hardening phenomena.

As indicated above, the dislocation loops are considered to lie on \{111\} planes. Those planes which intersect the \{110\} glide planes at an angle do so in parallel lines of intersection on the \{110\} planes. Thus the active loop barrier pairs will all lie parallel as shown schematically in Figure 13. As a result, the paired barrier array will exhibit an orientation dependence with regards to an orientation independent glide dislocation (i.e. the constant-line-tension model). Since the Burgers vectors of the glide dislocations lie at 35.26° to either side of the parallelism, the screw and edge components of the real glide dislocation would require some sort of average behavior to interact with the loop barriers. Hence, in order to produce an average behavior, characteristic of the orientation independent, constant-line-tension model, the barrier pairs were located in the array in both parallel and perpendicular orientations. This type of array showed no orientation dependence and was considered a good average representation of the glide behavior. The normalized critical shear stress values, $K$, for the two orientations shown in Figure 13 and for the mixed arrays were all within the 5% standard deviation indicated previously.

Considering the normalized shear stress, $K$ as a hardening correlation coefficient, the value required to correlate the measured and the calculated hardening increments were
Figure 13. Schematic illustration of dislocation loops intersecting a slip plane. Direction A - parallel to loop barrier pairs, direction B - perpendicular to loop barrier pairs.
obtained by equating the interpolated $\Delta^*_c$ from Figure 12 and the corresponding $\Delta T_c$ in Equation 17 using $\lambda$ values from Table 3. The $K$ values obtained are tabulated also in Table 3 and are plotted in Figure 14 as a function of the annealing temperatures. From Figure 14 it can be seen that in the as-irradiated condition, shown as the 100°C anneal stage, the correlation of the hardening in the three oxygen levels is very consistent, the $K$ values being essentially the same at 0.41. Once annealing begins, however, the goodness of this correlation varies somewhat, especially above the 400°C annealing stage. It appears, therefore, that based on the TEM-observed defect structure in the as-irradiated and low temperature annealed specimens, the dispersed barrier model has provided a good correlation of the hardening. Above the 400°C annealing stage, though, this does not appear to be the case.

The interpretation usually given to the increasing value of $K$ with annealing temperature in Figure 14 is that during annealing the barriers are getting stronger (68). This barrier strengthening mechanism is generally attributed to the diffusion of interstitial impurity atoms to the defect clusters (8,68). Upon comparing $K$ with Figures 7, 8 and 9, however, it can be seen that the maximum $K(\phi_c)$ values are about 0.61-0.62. Thus the initial increase of $K$ with annealing temperature is within the range of the dispersed barrier model,
Figure 14. Hardening correlation coefficient, K, at the various annealing stages. (100°C anneal = as-irradiated condition)
but the hardening correlation at 500°C indicates that the hardening is in excess of that which can be produced by the dispersed barrier model. If the as-irradiated hardening (i.e. RH) were considered due to some other mechanism, then, in this case, at least, the total RAH could be explained as a strengthening of some barrier. It may be possible that some non-TEM-observable defects could be responsible for the RH, their existence being in a quasi-equilibrium with and therefore proportional to the observable defects. Since the RH has been observed in metals with no TEM-observable defect structures, for example in Al (35, 69), Ni (70), V (51) and Fe (71), this possibility cannot be ruled out. However, the good correlation obtained here of the RH with the observed defect structure would tend to indicate that the defect loops could be responsible for the RH. Other investigators have also found various correlations between the RH and the TEM-observable defects, as summarized by Moteff et al. (72) so we shall pursue this approach in the following discussion.
IV. DISCUSSION

If we accept as a premise that the RH is due to dispersed barrier-type hardening by the TEM-observable defect loops, then based on the previous analysis some other mechanism must contribute to at least some of the RAH. In the following discussion we shall look at several possible models for RAH and propose an explanation for the phenomenon. In addition, we shall look at the RAH reported by other investigators in an attempt to provide a more complete analysis.

The conclusions reached in the preceding correlations are based on the following assumptions: (1) that the constant-energy line tension of the dislocation, \((1/2)Gb^2\), is a realistic value; (2) that only the loops inclined to the slip planes produce significant barriers; and (3) that the average Taylor factor gives a reasonable relation between the uniaxial tensile yield stress and an average critical resolved shear stress. The latter two assumptions are believed valid whereas the first may be somewhat less reliable. The constant of 1/2 in the line tension energy term, while being a value typically reported in the literature, depends, generally, upon the type of barriers and the self-interaction energy of the bowed dislocation about the barrier.
A. Hardening Due to Defect Loop Immobilization

In order to study the defect loop immobilization we shall look at the diffusion of IIA to the loops. Bullough et al. (61) have investigated the diffusion of IIA in the loop strain field and the subsequent attraction of the IIA to the loop. If \( N(t) \) is the number of impurities that arrive at the loop in time \( t \), and \( F(t) \) is the fraction if IIA that arrive at the loop in time \( t \), then from Bullough et al. we can write

\[
N(t) = B C_0 t^{3/5} \quad (18)
\]

and

\[
F(t) = n N(t)/C_0 = n B t^{3/5} \quad (19)
\]

where

\[
B = (3.68 \times 10^{-4} \ °K \ cm \ \epsilon \ Dr^2/T)^{3/5}, \quad (20)
\]

\( C_0 \) is the initial IIA concentration (cm\(^{-3}\)), \( n \) is the volume density of defect loops (cm\(^{-3}\)), \( \epsilon \) is the spherical dilatation strain of the IIA, which we shall assume constant with \( \epsilon = 0.87 \) (from (61)), \( D \) is the diffusion coefficient (cm\(^2\)/sec) at temperature \( T(°K) \) and \( r \) is the mean radius (cm) of the defect loops. The constant in \( B \) differs from that which would follow from Equation 22 in Bullough et al., viz 0.913, and was obtained by recalculating their Equation 22. This corrected form will be used in the following analysis.

The Equations 18-20 assume no matrix depletion of IIA and therefore apply only to the early stages of the anneal, i.e. when \( F(t) < 1 \). They will be used to compare the IIA behavior
during the anneals of materials from several investigators. A normalized form of $N(t)$ is included to compare the number of IIA per Burgers vector of loop length. This is considered a pinning strength and is obtained by multiplying $N(t)$ by the magnitude of the Burgers vector, $b$, and dividing by the circumference of the loop $2\pi r$.

Some RAH observations in niobium by Ohr et al. [12] are shown in Figure 15. The first peak shows a hardening increment of about $1.74 \text{ kg/mm}^2$ (2.47 kpsi, 17.06 MPa) and the second about $3.61 \text{ kg/mm}^2$ (5.14 kpsi, 35.43 MPa). Ohr et al. attribute these peaks to the diffusion of oxygen and carbon, respectively, to the defect loops. These IIA are then considered to strengthen the defects giving rise to the RAH peaks. Bullough et al. [61] have shown with their theoretical model that, in similar niobium samples, oxygen may be going out of solution during annealing at temperatures up to 200°C. However, if we consider the diffusion behavior of the IIA in terms of $N(t)$, the strengthening of the defect loops by the carbon becomes questionable, at least within the range of the dispersed barrier hardening model. Table 4 shows the diffusion behavior of the oxygen and carbon during the annealing stages of this irradiated niobium. The annealing time of the niobium work was 2 hrs and, based upon similar material studied by Ohr et al. [73], a defect density $n = 5 \times 10^{15} \text{ cm}^{-3}$ and a loop radius $= 40 \text{ Å}$ was used. Since the
Figure 15. Radiation-anneal hardening in polycrystalline niobium. Test temperature 24°C. Refs. (12, 73)
Table 4. Diffusion behavior of oxygen and carbon in niobium during various annealing stages (annealing time = 2 hrs)

<table>
<thead>
<tr>
<th>T(°C)</th>
<th>D(^a)(cm(^2)/s)</th>
<th>(\sqrt{6Dt}) (Å)</th>
<th>N(t)</th>
<th>F(t)</th>
<th>N(t)b/2πR</th>
</tr>
</thead>
<tbody>
<tr>
<td>125</td>
<td>3.538E-17</td>
<td>123</td>
<td>1.7</td>
<td>0.001</td>
<td>0.019</td>
</tr>
<tr>
<td>150</td>
<td>2.641E-16</td>
<td>338</td>
<td>5.4</td>
<td>0.002</td>
<td>0.061</td>
</tr>
<tr>
<td>175</td>
<td>1.576E-15</td>
<td>825</td>
<td>15.2</td>
<td>0.006</td>
<td>0.172</td>
</tr>
<tr>
<td>200</td>
<td>7.783E-15</td>
<td>1834</td>
<td>38.3</td>
<td>0.015</td>
<td>0.435</td>
</tr>
</tbody>
</table>

41 wt ppm oxygen \((C_o = 1.30 \times 10^{19} \text{ cm}^{-3})\)

<table>
<thead>
<tr>
<th>T(°C)</th>
<th>D(^a)(cm(^2)/s)</th>
<th>(\sqrt{6Dt}) (Å)</th>
<th>N(t)</th>
<th>F(t)</th>
<th>N(t)b/2πR</th>
</tr>
</thead>
<tbody>
<tr>
<td>250</td>
<td>6.332E-17</td>
<td>165</td>
<td>2.3</td>
<td>0.001</td>
<td>0.026</td>
</tr>
<tr>
<td>300</td>
<td>1.013E-15</td>
<td>661</td>
<td>11.5</td>
<td>0.004</td>
<td>0.130</td>
</tr>
</tbody>
</table>

35 wt ppm carbon \((C_o = 1.48 \times 10^{19} \text{ cm}^{-3})\)

\(^a\)From reference 74, \(D(\text{oxygen}) = 0.0212 \exp\{\frac{-1.167}{kT}\}\text{cm}^2/\text{s}\).

\(D(\text{carbon}) = 0.004 \exp\{\frac{-1.432}{kT}\}\text{cm}^2/\text{s}\).

\(k = 8.616 \times 10^{-5} \text{ eV/°K}\).
anneal at 150°C produces a large hardening peak, one could conclude from \(N(t)b/80\pi\) that, on this relative scale, \(=0.06\) IIA per b of loop length immobilized the loops as the next annealing stage at 175°C produces very little additional hardening, although a considerably larger amount of oxygen has diffused to the loop, \(=0.17\) IIA per b of loop length. Thus the loops may be considered as being saturated. Ignoring the diffusion model, it could be argued that the matrix was virtually depleted of the oxygen atoms during the 150°C anneal so the higher temperature anneals contribute no more oxygen. A numerical evaluation of the hardening in this material, developed in Appendix C, supports this argument by showing that, based on the dispersed barrier hardening model, complete defect immobilization would produce a hardening slightly in excess of the oxygen peak. From this analysis, there appears to be insufficient oxygen to completely immobilize the loops. The analysis based on the diffusion model, however, indicated otherwise, i.e. not all the oxygen gets to the loops during the 150°C anneal. From Table 4, the volume fraction, \(F(t)\), at 175°C is still much less than unity \((= 0.006)\) so matrix depletion has not yet occurred and the diffusion model should still be valid. In either case, the loops appear to be virtually immobilized by the oxygen atoms so that diffusion to them by the carbon atoms would contribute little additional hardening. It follows, therefore, that
the carbon hardening peak in Figure 15 cannot be due solely to loop immobilization and it is postulated that either the resolution of carbon atoms produces the hardening or carbide precipitates are formed during the anneal. The resolution of oxygen atoms from the loops may also contribute to this second peak, the carbon atoms being the immobilizing agents.

An analysis similar to the above has been applied to the RAH data of Bajaj. Table 5 shows the diffusion behavior of oxygen in vanadium. The loop size and density were obtained from Table 1 to calculate $B$ and the depletion fraction $F(t)$. The annealing time for this vanadium was 1 hr. As can be seen from Table 5, the volume of IIA available to each loop is depleted very rapidly with increasing annealing temperature, as indicated by the fraction $F(t) \to 1$. This indicates a rapid agglomeration of the impurity oxygen atoms about the loops which, in numbers relative to the niobium analysis, indicate a rather rapid immobilization of the loops with increasing annealing temperature. This result correlates very well with a reinterpretation of the hardening correlation coefficient of Figure 14, as shown in Figure 16. The hardening correlation coefficients, $K$, are being interpreted as asymptotically approaching their maximum values. This, then, corresponds to a complete immobilization of the defect loops by the diffusing oxygen atoms and is considered here to be the primary RAH mechanism in the vanadium of Bajaj. This approach, however,
Table 5. Diffusion behavior of oxygen in vanadium during the various annealing stages (anneal time = 1 hr)

<table>
<thead>
<tr>
<th>T(°C)</th>
<th>D(^a)(cm(^2)/s)</th>
<th>(\sqrt{\delta}D_t^\circ) (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>1.308 E-19</td>
<td>5</td>
</tr>
<tr>
<td>200</td>
<td>5.118 E-16</td>
<td>332</td>
</tr>
<tr>
<td>300</td>
<td>1.117 E-13</td>
<td>4911</td>
</tr>
<tr>
<td>400</td>
<td>4.918 E-12</td>
<td>3.3E4</td>
</tr>
<tr>
<td>500</td>
<td>8.138 E-11</td>
<td>1.3E5</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>95 wt ppm O</th>
<th>T(°C)</th>
<th>N(t)</th>
<th>F(t)</th>
<th>N(t)b/2πR(^b)</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td>16</td>
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<td>0.1</td>
<td></td>
</tr>
<tr>
<td>300</td>
<td>378</td>
<td>0.100</td>
<td>2.4</td>
<td></td>
</tr>
<tr>
<td>400</td>
<td>4848</td>
<td>0.685</td>
<td>22.3</td>
<td></td>
</tr>
<tr>
<td>500</td>
<td>41400</td>
<td>1.0</td>
<td>73.5</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>300 wt ppm O</th>
<th>T(°C)</th>
<th>N(t)</th>
<th>F(t)</th>
<th>N(t)b/2πR(^b)</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td>24</td>
<td>0.006</td>
<td>0.3</td>
<td></td>
</tr>
<tr>
<td>300</td>
<td>590</td>
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<td>6.7</td>
<td></td>
</tr>
<tr>
<td>400</td>
<td>1016</td>
<td>1.0</td>
<td>47.8</td>
<td></td>
</tr>
<tr>
<td>500</td>
<td>73100</td>
<td>1.0</td>
<td>125.0</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>500 wt ppm O</th>
<th>T(°C)</th>
<th>N(t)</th>
<th>F(t)</th>
<th>N(t)b/2πR(^b)</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td>38</td>
<td>0.008</td>
<td>0.5</td>
<td></td>
</tr>
<tr>
<td>300</td>
<td>969</td>
<td>0.192</td>
<td>11.1</td>
<td></td>
</tr>
<tr>
<td>400</td>
<td>8136</td>
<td>1.0</td>
<td>61.7</td>
<td></td>
</tr>
<tr>
<td>500</td>
<td>96300</td>
<td>1.0</td>
<td>177.0</td>
<td></td>
</tr>
</tbody>
</table>

\(^a\)From reference (74): \(D(\text{oxygen})=0.013 \exp \{-1.358/kT\}\) cm\(^2\)/s.
\(D(\text{carbon})=0.0045 \exp \{-1.183/kT\}\) cm\(^2\)/s.

\(^b\)When \(F(t)=1\), \(N(t)\) is taken here as the total IIA concentration in the average volume per defect, i.e. conc. IIA/n, n=defect density.
Figure 16. Hardening correlation coefficient, $K$, at the various annealing stages (100°C anneal = as-irradiated condition)
leaves peaks in the hardening correlation at 500°C. It is believed that these peaks are also observable in the yield stress data in Figure 11. The defect structure data indicates a loss of defect loop hardening at and above 500°C due to the annealing out of the TEM-observable defects. This is evident from the calculated hardening in Table 3. Since the measured hardening does not show this recovery, due perhaps to the growth of some other dislocation barrier, the large $K$ values result. As in the niobium case, these secondary hardening peaks are considered to be due to carbon. In vanadium, though, the diffusion coefficient for carbon is slightly larger than that for oxygen. It is therefore required that the carbon remain bound in complexes and to be released at higher temperatures. The possibility of this phenomenon will be explored below. This carbon, then, when released from the complexes, could produce resolution hardening or precipitate to form some type of precipitation hardening. A numerical evaluation of this 500°C peak as possibly being due to resolution hardening by the carbon is given in Appendix C. However, from (11), it is apparent that the accuracy of the defect structure measurements decreases rapidly above 400°C so the magnitudes associated with this peak are not well-defined. Justification for this peak being carbon dependent appears in the work of Morozumi et al. (75) where a variation of the carbon content in vanadium creates varying peak heights at the
500°C anneal stage. This interpretation of the peak at 500°C also explains why such peaks do not occur in some of the indentation hardness measurements of Bajaj in Figure 10. The medium and high oxygen samples in this figure have about half the carbon content of the low sample and of the polycrystalline samples in Figure 11.

In subsection III-C it was indicated that only half of the defect loops are being considered as effective barriers. If all the defects were barriers then the constant in Equation 17 would be increased from 0.537 to 0.888 and the correlation coefficients, K, in Table 3 would be reduced by 40%. This would enable the hardening correlations to be made completely by the defect loops, as was concluded by Bajaj (11) for the vanadium and by Ohr et al. (73, see also 52) for the niobium. However, as indicated previously, the loops normal to the glide plane are considered not to be effective barriers. Foreman (76), in analyzing RH in fcc metals also disregarded the loops normal to the glide plane, considering only those glide dislocation-loop interactions which form stable, low energy junctions as being of significance. In the bcc systems, this amounts to only 1/4 of the loops, which is an insufficient number of barriers for any reasonable correlation to be made. Thus, both the loops inclined to the glide plane were considered as barriers. However, the impurity atom-Cottrell cloud formation about the normal loops may, in
some way, produce an additional barrier. This would then de­crease the hardening requirements of some secondary hardening mechanism. It may also be that the inclined loops are hard barriers which give rise to the RH so that the immobilization of the normal loops gives rise to the asymptotic hardening in Bajaj's data.

It is thus being proposed that in addition to the loop immobilization which occurs during annealing, a second hardening mechanism must become operative to increase the density of dislocation barriers. Two such mechanisms are the resolu­tion hardening and the formation of coherent precipitates or precipitation hardening.

B. Impurity Atom Complexes and Resolution Hardening

If the IIA become bound to vacancies or vacancy clusters during irradiation such that their tetragonal distortions in the matrix are minimized or removed, then the solution hardening by the IIA would be eliminated. It has been shown that during irradiation the concentration of IIA in solution decreases (62,77,78) and it has been demonstrated by Damask (79) with carbon in α-iron that carbon-vacancy complexes are formed. But whether or not these complexes would affect the glide dislocation is not known. Upon annealing, then, the IIA could return to solution producing a resolution hardening. In the analysis of the niobium of Ohr et al., the resolution
hardening could occur to produce most of the second hardening peak in Figure 15 as shown in Appendix C. The mean diffusion length of oxygen in niobium at 150°C is \( \sqrt{6Dt} = 338 \text{ Å} \) for a 2 hr anneal. This same length would be obtained for the carbon in niobium at 275°C. Thus the carbon peak occurs in the same range of IIA mobility as the oxygen peak, indicating that if the IIA were initially in complexes, their binding energies would be similar. In the case of vanadium, however, the conclusion does not hold. The delayed occurrence of the carbon indicates a fairly large binding energy of carbon to the complexes for the diffusivities of oxygen and carbon in vanadium are very similar (\( \sqrt{6Dt} = 470 \text{ Å} \) and 694 Å for oxygen and carbon, respectively for 2 hrs at 200°C). Damask (79) has observed a fairly large binding energy for carbon to vacancies in α-iron. Although the carbon is fairly mobile in iron at 40°C (\( \sqrt{6Dt} = 265 \text{ Å} \) for 2 hrs, \( D = 0.0039 \exp(-0.831/kT) \text{ cm}^2/\text{sec} \)) (80), Damask found that the carbon was not released from the complexes until around 250°C where, it being in supersaturation, immediately precipitated. Since carbon has a considerably higher solubility in vanadium than in iron, the possibility of a high binding energy in vanadium may be questionable.

A further observation with regards to the oxygen complexes is that in the above niobium work of Ohr et al. and in some vanadium work of Eto et al. (77) their samples were irra-
diated at about 50 to 60°C. Both resulted in fairly rapid RAH peaks with annealing temperature. Bajaj's vanadium was irradiated about 100°C and exhibits a more gradual RAH with temperature, as does the results of Shiraishi et al. (81) where vanadium was irradiated around 200°C. It is speculated that in both of these latter cases, the oxygen is not bound in complexes after irradiation and is thus free to migrate, either during irradiation or during the subsequent annealing stages.

C. Precipitation Hardening

The RAH of a 300 wt ppm nitrogen doped α-iron sample is shown in Figure 17 and is also taken from Ohr et al. (12). This high nitrogen sample also exhibits fairly rapid anneal hardening and it is postulated that this is partly due to the formation of nitride precipitates. Keh and Wriedt (82) made some quench-aging studies of nitrogen in α-iron and using TEM micrograph analysis found that with 220 wt ppm nitrogen the mode of precipitation depended very much upon the aging temperature. Aging for 1 hour at and below 100°C, for example, produced a "uniform nucleation of nitride particles inside the iron lattice," being, according to them, probably $\text{Fe}_{16}\text{N}_2$ with $\{100\}$ habit planes. Aging at 200°C produced no precipitates in the lattice, the precipitation tending to be concentrated on dislocations. A 60 wt ppm sample also tended to show only precipitation on the dislocations and then only with
Figure 17. Radiation-anneal hardening in polycrystalline iron containing <2, 20 and 300 wt ppm nitrogen irradiated below -50°C to 4x10^17 n/cm^2 (E>1 MeV). Annealing time 15 min and test temperature -36°C. Ref. (12)
longer aging times (>1 hr). (Note: for N diffusion in Fe, 
D = 0.005 exp{-0.796/kT} cm²/sec (80).) Stanley (83) has re-
ported that n-irradiation greatly enhances the precipitation 
of nitrides in aged α-iron, concluding that "the irradiation 
produced nucleation sites are clusters of vacancies or inter-
stitials." Thus, based on the observations of Keh and Wriedt, 
it could be concluded that at 200°C and above, the hardening 
in Figure 17 is due to precipitates having formed on the de-
fect loops, anchoring them and giving very similar hardening 
to the two nitrogen doped samples. Below 200°C, the hardening 
of the high nitrogen samples could, in addition to the loop 
hardening, be attributed to the formation of precipitates in 
the matrix, forming possibly on vacancy clusters or other 
impurity atoms. Thus where the solubility of the IIA in the 
host matrix is very low, there appears to be a good possibil-
ity of precipitate formation. These precipitates increase 
the dislocation barrier density and therefore effect further 
anneal hardening.

The solution hardening at very low concentrations (<200 
wt ppm IIA) of carbon in niobium and vanadium appears not to 
be well defined (84,85,86,87). The hardening increases 
rapidly from below 100 wt ppm IIA to their approximate solu-
ibility limits which lie in the range of 150-300 wt ppm at room 
temperature. But it is also known that in the bcc metals, 
the IIA tend to cluster (88) so whether the rapid hardening
observed is an actual solution hardening or an ordering effect is difficult to ascertain. These ordered clusters are also observed with oxygen in niobium at concentrations (≈200 wt ppm) well below their solubility limits (89) and in vanadium (90). They appear to be bct zones which would produce strain fields and interact with the glide dislocations. Thus the hardening observed during the annealing of irradiated bcc metals may be due in part to some type of IIA clustering or ordering phenomena rather than the more conventional solution or precipitation hardening.
V. CONCLUSION

The results of the computer simulation of radiation hardening and radiation-anneal hardening were compared with the experimentally measured hardening in irradiated vanadium and niobium. For this comparison, the densities and size distributions of the defect clusters observed by transmission electron microscopy and the measured yield stresses were obtained from previous work. The critical resolved shear stresses deduced from the experiments were compared with the computed critical shear stresses.

For the case of vanadium, where defect cluster size distributions were available, the results of the computer analysis indicate virtually the same amount of radiation hardening and radiation-anneal hardening whether the size distribution or the average size is used to determine the barrier pair spacing. For as-irradiated vanadium, and niobium, comparison of the experimental and calculated critical shear stresses implies that the barriers are not of maximum strength and that the slip dislocation breaks through the barriers instead of bowing around them (Orowan hardening). As concerns radiation-anneal hardening, the change in the observed density and size distribution of defect clusters was found to be inadequate to explain the measured hardening. Possible additional mechanisms for the radiation-anneal hardening were suggested as follows: (1) strengthening and/or immobilizing the in-
clined barrier loops due to the trapping of interstitial impurity atoms, particularly oxygen; (2) the immobilization of the loops normal to the slip plane due to interstitial impurity atom trapping at the loops; and (3) the formation of other hardening agents such as impurity atoms returning to solid solution, precipitates or impurity atom clusters.
VI. REFERENCES


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40. T. J. Koppenaal, Phil. Mag., 8, 1313 (1963).


76. A. J. E. Foreman, Phil. Mag., 17, 353 (1968).
VII. ACKNOWLEDGEMENTS

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Deep appreciation is extended to Miss Verna J. Thompson for the meticulous editing and typing of this manuscript under the pressures of time and circumstances.
VIII. APPENDIX A. A SHORT FLOW CHART OF THE DISPERSED BARRIER MODEL COMPUTER CODE

MAIN

INIT - reads input data, generates array, stores on file 9 or retrieves array from file 9

PLOT - plots array on line

BASE(1) - finds basic configuration along lower edge of array, \( R = \min(100\lambda_a, 23170, \frac{\text{SIZE}X}{2} - 1.0) \)

\( i = i + 1 \) increments on \( \tau_i \) up to \( \tau_{\text{max}} \)

\( R = \frac{\lambda_a}{2\tau_i} \) if \( \tau_i = \tau_{\text{max}} \), calculates \( R \) from "weakest link" (angle or length)

BASE(0) - checks LH boundary for points inside new bow-out (uses mirror boundary conditions)

starts at LHS and incrementally moves to point \( j \) along dislocation configuration

\( j = j + 1 \)

SRCH(IO,II,II) - IO = point at \( j - 1 \), \( P_{j-1} \)

II = point at \( j \), \( P_j \)

I2 = point at \( j + 1 \), \( P_{j+1} \)

START(IO,II,II) - calculates lengths \( L_{01}, L_{12}, L_{02} \)

- calculates angles \( \theta_{01}, \theta_{12}, \phi_{01}, \phi_{12} \)
L< \rightarrow

L_{12} < 2R

BOW(I0,I1,I2) - calculate $\alpha$
test $\alpha > \phi_c$

assume Frank-Read-type bow-out
search for new points
if bows off top of array, run
terminates on return to MAIN
if $l_1 < 0$ on return from SRCH, then dislocation has closed on itself, leaving a group of encircled obstacles. Configuration is then shortened to where dislocation crosses.
if not at RH boundary, goes up and increments $j$

BASE(I0,I1,I2) - checks RH boundary for mirror boundary conditions

AREA - determines area under configuration and mean height

if no termination signal from SRCH (bowing out top of array), goes up and increments $\tau$.

(for definitions, see last page of this Appendix)
Figure 18. Diagramatic explanations of variables used in the computer code

\[ \alpha = \theta_{o1} - (\theta_{i2} + \phi_{i2} + \phi_{o1}) \]

\[ \text{IF } (\theta_{o1} < \theta_{i2}) \quad \alpha = \alpha + 2\pi \]
### IX. APPENDIX B. CHEMICAL ANALYSIS OF SAMPLES

<table>
<thead>
<tr>
<th>Element</th>
<th>Low oxygen</th>
<th>Medium oxygen</th>
<th>High oxygen</th>
</tr>
</thead>
<tbody>
<tr>
<td>Interstitial impurities</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>O</td>
<td>60</td>
<td>205</td>
<td>640</td>
</tr>
<tr>
<td>C</td>
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</tr>
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<td>N</td>
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<td>2-6</td>
<td>2-4</td>
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<tr>
<td>H</td>
<td>2-8</td>
<td>10-12</td>
<td>15-18</td>
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<tr>
<td>Substitutional impurities</td>
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</tr>
<tr>
<td>Fe</td>
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<td>15-20</td>
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<tr>
<td>Ni</td>
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<tr>
<td>Si</td>
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</tr>
<tr>
<td>Average grain size in mm</td>
<td>0.024</td>
<td>0.142</td>
<td>0.146</td>
</tr>
</tbody>
</table>

Single crystal TEM and compression samples in weight parts per million

<table>
<thead>
<tr>
<th>Element</th>
<th>Low oxygen</th>
<th>Medium oxygen</th>
<th>High oxygen</th>
</tr>
</thead>
<tbody>
<tr>
<td>Interstitial impurities</td>
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</tr>
<tr>
<td>O</td>
<td>95</td>
<td>290-315</td>
<td>480-520</td>
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<tr>
<td>C</td>
<td>37</td>
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<td>30</td>
</tr>
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<td>N</td>
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<td>5</td>
<td>6</td>
</tr>
<tr>
<td>H</td>
<td>3</td>
<td>2</td>
<td>3</td>
</tr>
<tr>
<td>Substitutional impurities</td>
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<tr>
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<tr>
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<td>&lt;20</td>
</tr>
<tr>
<td>Si</td>
<td>&lt;40</td>
<td>&lt;40</td>
<td>&lt;40</td>
</tr>
</tbody>
</table>

1 From reference 11.

2 Total of other substitutional impurities, <100 ppm.
X. APPENDIX C. NUMERICAL EVALUATION OF HARDENING

This appendix is a numerical interpretation of hardening in the niobium of Figure 15 and the vanadium of Figure 11. A possible contribution of the various hardening models has been determined to explain the RH and RAH in the two cases.

Let us assume for now that both the oxygen and carbon atoms are, after irradiation, bound in vacancy or vacancy cluster complexes which removes their tetragonality and thus hardening capability from the matrix. Upon annealing these bound atoms are then released such that they return to solution and produce a solution hardening. The amount of hardening measured divided by the impurity atom concentration should give a solution hardening coefficient, \( \alpha \). From Figure 15 the hardening increments of each peak are 2.11 kg/mm\(^2\) (3.0 kpsi, 20.69 MPa) and 3.61 kg/mm\(^2\) (5.136 kpsi, 35.40 MPa) which can be associated with the oxygen and carbon atoms, respectively. From reference 73 this material contained 41 wt ppm oxygen and 35 wt ppm carbon. The resulting possible solution hardening coefficients are thus

\[
\alpha'_O = \frac{3}{41} = 0.073 \text{ kpsi/wt ppm O}
\]

\[
\alpha'_C = \frac{5.136}{35} = 0.147 \text{ kpsi/wt ppm C}
\]

A second sample in reference 73 similar to the sample of Figure 15, but with a carbon content of 60 wt ppm showed a slightly larger possible solution hardening coefficient for
the carbon, \( \alpha'' \), of 0.154 - 0.190 kpsi/wt ppm C. The primed coefficients have been used to denote possible hardening coefficients, in contrast to the actual solution hardening coefficients, \( \alpha_{IIA} \), as measured for each IIA. Values reported in the literature for the actual solution hardening coefficients for polycrystalline niobium \((86,91)\) are

\[
\alpha_O = 0.026 \text{ kpsi/wt ppm O}
\]

\[
\alpha_N = 0.066 - 0.138 \text{ kpsi/wt ppm N}
\]

Values for \( \alpha_C \) are not available in the literature but indications are that it is larger than that for nitrogen, \( \alpha_N \). In vanadium \( \alpha_O, \alpha_N \) and \( \alpha_C \) have the approximate values 0.02, 0.04 and 0.11 kpsi/wt ppm impurity atom, respectively \((87)\). Therefore, in niobium \( \alpha_C \) could well be double \( \alpha_N \). Comparison of the possible and actual hardening coefficients indicates that \( \alpha_O' \) is too high by a factor of about 3 but the \( \alpha_C' \) and \( \alpha_C'' \) could well be in the range of an \( \alpha_C \). It thus appears that solution hardening could account for the carbon peak in Figure 14, but not for the oxygen peak. The oxygen peak can be explained, however, by the Cottrell cloud mechanism whereby dislocation loops are immobilized by the diffusing oxygen providing the barrier strengthening as required in reference \((73)\). If we assume a defect structure roughly similar to that for the higher carbon sample where the mean interbarrier spacing, \( \bar{\lambda} \), is 990 Å \((= 1400 \text{ Å}/\sqrt{2})\) and the mean loop diameter, \( \bar{d} \), is 82 Å \((61)\), then a hardening increment due to the loops can be cal-
culated from Equations 15 and 17 as

$$\Delta \sigma_{\text{calc}} = \frac{1}{0.446} \frac{0.537K Gb}{\lambda} = 13.145K \text{ kg/mm}^2$$

where $G = 3.75 \times 10^{11} \text{ dynes/cm}^2$ is the shear modulus and $b = 2.85 \text{ Å}$ is the magnitude of the Burgers vector of niobium. We have used the mean Schmid factor rather than the mean Taylor factor as the samples are sheet material with a thickness of about 3 grain diameters. There would thus not be the geometric constraints imposed on the yield stress that occurs in larger tensile samples. The hardening coefficient, $K$, required to correlate $\Delta \sigma_{\text{calc}}$ with the measured $\Delta \sigma$ ($=4.17 \text{ kg/mm}^2$) has a value of 0.317. From the $K(\phi_c)$ analysis we have shown that for a loop diameter to interbarrier spacing ratio $d/\lambda = 0.06$, $K$ has a maximum value of about 0.6. This value of $K$ is indicative of maximum strength barriers where the Orowan mechanism is operative. Thus, with the dispersed barrier model, the maximum hardening increment that may be obtained is

$$\Delta \sigma_{\text{calc}}(\text{max}) = 13.145 \times 0.6 = 7.89 \text{ kg/mm}^2$$

Adding this to an unirradiated yield stress of 3.47 kg/mm$^2$ yields a maximum attainable yield stress of 11.36 kg/mm$^2$. This is in excess of the oxygen peak at 9.75 kg/mm$^2$ by 1.61 kg/mm$^2$ which may be interpreted as there being insufficient oxygen to completely immobilize the loops. The oxygen peak can thus be explained by considering dislocation loop immobilization by Cottrell clouds as the hardening mechanism in a
dispersed barrier model.

In addition, a little more hardening can still be obtained from the loops by diffusing carbon atoms in the carbon peak region. This would then reduce the hardening requirements of the carbon atoms returning to solution such that the possible solution hardening coefficients become $\alpha'_c = 0.10$ ksi/wt ppm C and $\alpha''_c = 0.085 - 0.11$ ksi/wt ppm C. A value of $\alpha_c$ obtained from the differences of the yield stresses of the two unirradiated samples used in (73) is also of this range, viz. 0.10 ksi/wt ppm C. These results, although showing excellent agreement, are rather fortuitous as the radiation produced defect structure of this sample was only an estimate.

An approach similar to the above has been applied to the primary data under consideration, viz. the vanadium work of Bajaj (11). As indicated previously, it appears justifiable to assume the primary loop immobilization or hardening is due to oxygen migration. In this case, though, there is sufficient oxygen present in the material to totally immobilize the loops, as shown in Figure 16 where the barrier strengths asymptotically approach their upper limits. The additional hardening peaks observed at 500°C, therefore, cannot come from any loop hardening contributions. These peaks, as before, are believed due to carbon atoms, their release from vacancy complexes being delayed as in the $\alpha$-iron work of Damask (79). The hardening increments above the asymptotic limits ($K =$
0.625) at 500°C are 6.0, 6.0 and 5.3 kpsi for the 95, 300 and 500 wt ppm samples, respectively. From Appendix B, the corresponding carbon concentrations are approximately 35, 50 and 70 wt ppm, respectively. The small carbon peak of the high oxygen sample can be explained by the carbon going into solution. In the low and intermediate oxygen cases, however, the carbon peaks are too large to be explained completely by the solution hardening of carbon although the standard deviation on the defect loop sizes for these data points is large (30%). The lower limit of these standard deviations decreases the hardening increments well within the range of solution hardening by the available carbon (35 and 50 wt ppm). Whether or not the carbon peaks can be attributed directly to the resolution of carbon is certainly debatable. Substitutional impurities of iron or nickel are present at levels of 40-50 wt ppm so that various types of carbides may be forming to effect the hardening. Thus in both the vanadium and niobium cases, the hardening has been interpreted in terms of two mechanisms: a dispersed barrier hardening model in which oxygen immobilizes the defect dislocation loops to form hard barriers and an additional hardening increment considered to be determined by the carbon impurity of the material. Although the hardening mechanism of the 500°C peak in vanadium cannot be identified, its dependence on the carbon atoms appears to be justified.