

OCT 24 1963

GMELIN REFERENCE NUMBER

AED-Conf-63-029-2

CONF MASTER
OCT 25 1963
(2,c)✓

EFFECT OF ELECTRON IRRADIATION ON MECHANICAL PROPERTIES
OF ALUMINUM SINGLE CRYSTALS AT 80°K

by

K. Ono, M. Meshii and J. W. Kauffman

Materials Research Center
and Department of Materials Science
The Technological Institute
Northwestern University
Evanston, Illinois

CONF-49-3

Metallurgical Society of
A.I.M.E.,
Annual Meeting
Dallas, Texas
February 24-28, 1963

WITHDRAWN
BADGER AVENUE

ABSTRACTED IN NSA

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

Effect of Electron Irradiation on Mechanical Properties

of Aluminum Single Crystals at 80°K

K. Ono, M. Meshii and J. W. Kauffman

Materials Research Center and Department of Materials Science

Northwestern University, Evanston, Illinois

Aluminum single crystals of 99.999% initial purity were irradiated at 80°K with 1 MeV electrons to total dose of $1.2 \sim 8 \times 10^{17}$ el/cm². The flow stress was measured at 77°K over the strain rate range 5×10^{-4} to 4×10^{-3} and increased with irradiation. A larger increase for the higher strain rate was observed. The increase is interpreted to be due to interstitials which have migrated to dislocations and have been absorbed heterogeneously by making superjogs or kinks, which produce resistance to motion of the dislocations. An interruption of tensile test for a short time resulted in a yield drop which was found only in the irradiated crystals. This yield drop may be due to the Cottrell locking of the interstitials which were freed from impurity trapping sites by glide dislocations. Annealing at room temperature resulted in recovery of the hardening caused by the irradiation. Migration of vacancies or divacancies to the dislocation having interstitial jogs may produce the recovery.

INTRODUCTION

The effect of particle irradiation on various physical properties of metals has been investigated from many aspects. The basic process involved is, at least in part, due to the creation of vacancies and interstitials.

Effects of neutron irradiation on the mechanical properties of metals have been studied quite extensively and have been established as resulting from the presence of defect aggregates.⁽¹⁾ However, little attention has been paid, thus far, to the hardening effect of electron irradiation, which can produce the simplest types of defect, viz., single vacancies and interstitials.

Earlier studies^(2,3) were by no means complete, although a suitable energy of electrons was employed to obtain simple types of defect.

The most extensive work⁽⁴⁾ to date was performed on copper single crystals with 4 MeV electrons at irradiation temperatures between 77°K and 293°K. The critical shear stress was increased by the irradiation, but no change in rate of work-hardening was observed. It has been suggested from the dependence of the hardening on the irradiation temperature that homogeneously nucleated clusters of randomly migrating point defects were responsible for the process. It can be shown, however, by using the theory of electron-nucleus collision⁽⁵⁾ and the experimentally determined value of the threshold energy for displacement,⁽⁶⁾ that almost half of the collisions due to 4 MeV electrons have enough energy to create multiple defects.

Thus it may be necessary to modify the conclusion⁽⁴⁾ that the hardening produced by the electron irradiation is due to the introduction of isolated interstitial vacancy pairs.

The present investigation was aimed at the hardening effect of the simpler defect structure produced by a lower energy of incident electrons. Because of the difficulty in handling the very thin crystals required for electron penetration, aluminum was selected due to its relatively large penetration distance.

Recovery of electron induced resistivity of aluminum after a low temperature irradiation is known to occur in three major stages, as in copper. Stage I recovery includes recovery substages below 55°K, Stage II is between 55°K and 175°K and Stage III is between 175°K and 270°K. These are responsible for resistivity changes of 84%, 6% and 10%, respectively, of the total change due to irradiation near 4.2°K.⁽⁷⁾ As in copper, Stage I recovery is due to recombination of close-pairs and interstitial migration and Stage II is attributed to release of impurity trapped interstitials. Although the nature of Stage III recovery is yet to be established, we favor vacancy migration of some type, as will be discussed later.

Close pairs of vacancies and interstitials are by no means stable at the irradiation temperature of 80°K; interstitials move freely and recombine with vacancies or migrate to traps or sinks, namely impurity atoms and dislocations. The remaining immobile vacancies, whose pairing interstitials have migrated to the traps or sinks, stay dispersed in the matrix. By subsequent tensile testing, one can distinguish the responsible hardening mechanisms with the above-mentioned defect configuration in mind.

EXPERIMENTAL PROCEDURE

Aluminum single crystals of 99.999% initial purity, 30 cm long, were grown by the strain-anneal method. Sheet specimens of 0.8 x 6 x 38 mm were cut from as-grown crystals and annealed at 400°C for twenty-four hours. The orientations of the crystals were determined by the back reflection Laue method and are shown in Fig. 1.*

The specimens were then electropolished in an ethyl-alcohol-perchloric acid (4:1) solution to a final thickness of ~ 0.75 mm. Irradiations were performed in the Northwestern University Van de Graaff with an electron energy of (1.0 ± 0.1) MeV. A schematic drawing of the cryostat used is shown in Fig. 2. The electron beam was about 3 cm in diameter and collimated by a thick aluminum window to irradiate a specimen area of 15 x 6 mm. A beam current of ~ 10 μ a and not more than 25 μ a was maintained. A current integrator was used to measure the total dosage. Thus the irradiation time to attain a dose of 10^{17} el/cm² was about an hour.

Temperature of the specimen during irradiation was measured by a calibrated copper-constantan thermocouple, and the specimen was kept below 80°K. The specimen was set in the specimen chamber of the cryostat which was filled with helium gas and then immersed in liquid nitrogen. The electron beam was introduced into the chamber through two iron foils (total thickness of 0.076 mm) and a liquid nitrogen layer of 1 mm thickness. The cryostat was designed to allow tensile tests at 77°K after the irradiation without moving or heating the

*The crystals of the orientation A were used for the earlier experiments with a faster strain rate and those of orientation B for the later experiments with a slower strain rate.

specimen. Stress was measured by an electrical strain gauge load cell and the relative displacement of specimen grips was measured by a differential transformer transducer. The outputs were led to carrier amplifiers and subsequently to an X-Y recorder. The stress and strain sensitivities were $\pm 2\text{g/mm}^2$ and $\pm 5 \times 10^{-5}$, respectively. The rates of strain employed were $2 \sim 4 \times 10^{-3} \text{ sec}^{-1}$ and $5 \times 10^{-4} \text{ sec}^{-1}$.

In practice, a series of irradiation experiments was carried out on specimens of the same orientation. Irradiation was done on a specimen which had been strained to 0.3 ~ 1.5% of glide strain at 77°K before the irradiation. After the irradiation the tensile test was continued, thus enabling us to examine the effect of irradiation on hardening with the same dislocation configuration. In some instances, second and third irradiations were carried out after a small strain which followed the previous irradiation. The effects of interrupting the test and unloading were examined in both irradiated and unirradiated specimens. When the test was interrupted, the load was maintained to about 90% of the flow stress which is known to be enough to differentiate the unloading effect.⁽⁸⁾ To study the effect of annealing, the liquid nitrogen bath was emptied and the specimen was allowed to warm up. After recooling, the subsequent tensile test was performed at 77°K. During the irradiations as well as the annealing treatment, specimens were in the unloaded condition. Resolved shear stress-shear strain curves were calculated by the standard method.

EXPERIMENTAL RESULTS

Typical stress-strain curves of irradiated and unirradiated crystals tested at 77°K are shown in Fig. 3.

The shear stress and strain, at which the irradiation was carried out, ranged from 140 g/mm² to 210 g/mm² and from 0.3% to 1.5%, respectively.

An increase in flow stress was always found after irradiation, and the amount of the increase was dependent on total electron dose and the rate of strain. The fractional increase in resolved shear stress with respect to the electron dose is plotted in Fig. 4.

A larger increase was observed for the higher strain rate. For example, with a strain rate of $5 \times 10^{-4} \text{ sec}^{-1}$, the fractional increase in the shear stress was about 6%, but it was about 20% for $2 \sim 4 \times 10^{-3} \text{ sec}^{-1}$ at the same dose of $1.2 \times 10^{17} \text{ el/cm}^2$.

Although the effect of irradiation on the rate of work-hardening was not very apparent, a slight increase in the rate of work-hardening was observed in the early stage of deformation. As the deformation proceeded to the stage of more rapid work-hardening, however, the difference between stress-strain curves of irradiated and unirradiated crystals became smaller. Table I shows the rate of work-hardening before and after the irradiation together with electron dose and the strain at which the specimen was irradiated.

A sharp yield point or yield drop was not found at the beginning of plastic deformation after irradiation.

After a small strain of 0.2 ~ 0.7% following the irradiation, however, an interruption of the tensile test without unloading

resulted in an unexpectedly large yield drop of about $2 \sim 10 \text{ g/mm}^2$ or of a few percent of the flow stress for the interruption period of 5 sec \sim 5 min at 77°K, as shown in Fig. 5. The corresponding experiment was carried out in unirradiated crystals, but the yield drop failed to appear for the same condition. The interruption yield drop after the irradiation was larger for the higher strain rate. After continuing the test, another interruption was examined at the interval of 0.3 \sim 2.5% shear strain. The yield drop was again found. Since the experiments on the effect of interruption time on the yield drop were carried out with varying parameters, direct comparison could not be made. However, experimental results under similar conditions indicate that an interruption period longer than ten seconds results in little increase in the magnitude of the effect.

The yield drop due to the unloading was found to be similar for both irradiated and unirradiated crystals. This yield drop was observed with the unloading above a shear stress of 300 g/mm^2 , which is smaller than that reported by earlier investigators.⁽⁹⁾

The hardening effect of multiple irradiations on a crystal is shown by broken curves in Fig. 3. The fractional total increase in shear stress was notably larger at the same integral dose than that which is expected from a single irradiation, at least in the case of the faster strain rate.

Annealing of a specimen to room temperature was carried out after the effect of the fourth irradiation was measured. The result is shown in Fig. 6. The annealing time was about twelve hours. The flow stress decreased 15% which is about half of the

total increase due to the previous irradiations. Further annealing at room temperature after 4% strain did not lead to an additional decrease in flow stress except the recovery which is normally observed.

TABLE I
Change in Rate of Work-Hardening
due to Electron Irradiation

Electron Dose (el/cm ²)	Rate of Work Hardening (kg/mm ²)		Strain at the irradiation (o/o)	Strain ₁ rate sec
	before	after		
1.2 x 10 ¹⁷	1.4	1.5	1.46	2 ~ 4 x 10 ⁻³
1.2	1.5	2.5	1.43	"
8.0	1.1	2.0	0.81	"
1.2	1.5	2.4	0.75	5 x 10 ⁻⁴
1.2	1.2	2.5	0.31	"

DISCUSSION

A. Distribution of Defects after Irradiation.

The average energy of electrons at the middle of the specimen was calculated to be 0.8 MeV from the standard formula⁽¹⁰⁾. The displacement cross section, σ_d , of a primary recoil atom is obtained by integrating the McKinley-Feshbach approximation⁽¹¹⁾ for the differential cross section between the displacement threshold energy, T_d , and the maximum energy, T_m , which can be transmitted to an atom by an incoming electron, giving

$$\sigma_d, (\text{in cm}^2) = 2.5 \times 10^{-25} Z^2 \frac{(1-\beta^2)}{\beta^4} \left[\left(\frac{T_m}{T_d} - 1 \right) - \beta^2 \ln \left(\frac{T_m}{T_d} \right) + \pi \alpha \beta \left\{ 2 \left[\left(\frac{T_m}{T_d} \right)^{\frac{1}{2}} - 1 \right] - \ln \left(\frac{T_m}{T_d} \right) \right\} \right] \quad (1)$$

where Z is the atomic number, β is the ratio of the velocity of an electron to that of light, and $\alpha = Z/137$.

With the energy of electrons, used in this study, T_m is 117 eV. The value of T_d was experimentally determined by Lucasson and Walker⁽⁶⁾ to be 32 eV. A calculation with these values gives $\sigma_d = 15.4 \times 10^{-24} \text{ cm}^2$. Lucasson and Walker⁽⁶⁾ have concluded that the Seitz-Koehler model for multiple defect production⁽⁵⁾ is most suitable for the case of aluminum. With this model, the displacement cross section is calculated to be $15.9 \times 10^{-24} \text{ cm}^2$, at an electron energy of 0.8 MeV.

The difference between the two cross sections is due to secondary recoil atoms which would result in the formation of two vacancies, some of which may form divacancies. In the present case, the difference is only a few percent of the total cross section and thus the divacancies must be less than a few percent of the total defects. The concentration of trivacancies must be negligibly small, but, since T_m/T_d is about 3.7, few of them should be formed.

The concentration of the defects produced can be calculated by

$$C = n\sigma_d \quad (2)$$

where n is the total number of electrons per unit area. Because of the finite thickness of the specimens, it was necessary to integrate Eq. (2) along the specimen thickness. At the front and the back surfaces of the specimen, the energy of electrons differed by about 0.1 MeV from that in the middle of the specimen. This value corresponds to a change in the displacement cross section of $\pm 2.5 \times 10^{-24} \text{ cm}^2$. In this energy range, the change in σ_d is fairly close to linear with respect to the electron energy. The intensity of the electron beam also decreases as the beam passes through the specimen. From the data given by Marshall and Ward,⁽¹²⁾ changes of the beam intensity in aluminum with an incident beam energy of 0.9 MeV can be approximated as

$$n = n_0 (1 - 0.575 X^{2.5}) \quad (3)$$

where n_0 and n are the total number of electrons per unit area at the thickness of zero and X mm, respectively. The intensity of electrons, thus, decreased to 72% of the original value at the back surface of the specimen. Therefore, it is assumed that the distribution of

defects is homogeneous throughout the specimen.

The average concentration of the defects produced is obtained by integrating Eq. (2) over the specimen thickness, giving

$$C_{ave} = (13.2 \times 10^{-24}) n_0 \quad (4)$$

In our present investigation C_{ave} is not the concentration of the defects present as most of recovery of the radiation-induced resistivity is already completed at the irradiation temperature of 80°K, as mentioned above. The concentration of di-interstitials and other interstitial clusters which would be formed at the impurity trapping sites is negligibly small, since the instantaneous concentration of free interstitials is very small and the number of traps is greater than the interstitials which have been trapped at impurity atoms⁽¹³⁾.

By assuming that a vacancy and an interstitial contribute to the resistivity change by a comparable amount and that interstitials which have migrated to dislocations contribute to the resistivity by a negligible amount, the numbers of the interstitials which have migrated to the dislocations, the impurity trapped interstitials, and the vacancies dispersed in the matrix can be estimated to be 10%, 6% and 16% of C_{ave} , respectively.

In the present experiment, the specimens were deformed before the irradiations by a small amount. Therefore, the density of dislocations is about $10^8/\text{cm}^2$ (14). Although the density of dislocations in the previous resistivity experiments was not reported, it would have been comparable to or even larger than one in the annealed polycrystalline aluminum wire. Thus, it may be possible that the

number of interstitials which have migrated to the dislocations is larger in the present experiment than the above estimate. The increase, however, would not be larger than a factor of two, considering the data on irradiated cold-worked and annealed copper⁽¹⁵⁾.

B. Interpretation of the Observed Hardening

Dispersed vacancies can contribute to hardening through their dilatational field and modulus change, as discussed by Mott⁽¹⁶⁾, Nabarro⁽¹⁷⁾, and Fleischer⁽¹⁸⁾. The concentration of the vacancies, however, is on the order of 10^{-7} for the present case and a reasonable choice of constants shows that the effect is really negligible. Another possible source of hardening, viz., dislocation tangling due to the interaction of gliding dislocation and vacancies⁽¹⁹⁾, is also excluded since the reaction is very unlikely under the existing conditions. As vacancies are immobile at the testing temperature, coagulation of vacancies at the glide dislocations will not take place.

Solid solution hardening due to impurity trapped interstitials cannot explain the observed effect. Since the volume change of a Frenkel pair is estimated to be 1.25 atomic volume from the data of Blewitt, Coltman and Klabunde⁽²⁰⁾ with a Frenkel pair resistivity of $3.4 \mu\Omega\text{-cm}$ per atomic percent⁽⁶⁾, volume change of an interstitial would be 1.75 atomic volume when we assume relaxation around a vacancy to be 0.5 atomic volume. The estimated concentration of the interstitials is on the order of 10^{-8} , so the hardening effect is negligible if the volume change is spherical⁽¹⁶⁾. If a tetragonal distortion of an interstitial is present as assumed by Fleischer⁽²¹⁾, the magnitude of hardening by the tetragonal distortion is about the same as the

present result. His calculation, however, did not include thermal vibrations which are very important for such a short range effect as the dislocation-interstitial interaction. Therefore, the calculation overestimates the hardening effect when it is applied to our case, that is, 80°K as the test temperature. Moreover, the impurity trapped interstitials will be freed from the trapping sites and migrate to a dislocation, when they experience large enough interaction with the dislocation. Assuming the binding energy, B , to be roughly 0.1 eV⁽⁷⁾, it is shown in the Appendix that the impurity trapped interstitials within about 50 Å of a slip plane will be released from the sites by a moving dislocation. Consequently, strong solid solution hardening cannot be expected in terms of the impurity trapped interstitials.

The only remaining source of the hardening is the interstitials which have migrated to dislocations during the irradiation. The number of interstitials at the dislocations is about $9.5 \times 10^{15}/\text{cm}^3$ with the electron dose of $1.2 \times 10^{17} \text{ el}/\text{cm}^2$, and is the same order of magnitude as the number of atomic sites along the dislocations.

It has not been clear how interstitials behave at dislocations. If they can be absorbed easily at dislocations of an edge orientation, the dislocations will climb up uniformly by a few atomic distances⁽²²⁾. There will be little change in the nature of the dislocations. Therefore, little or no hardening can be expected.

An interpretation of the interruption yield drop, as will be discussed later, seems to suggest that interstitials cannot be absorbed immediately at the dislocations, instead, they form an

atmosphere at the dislocations. If this is the case, the interstitials may precipitate by forming superjogs on the dislocations when a critical supersaturation is reached*. The resulting dislocation is less mobile than a straight dislocation and a hardening effect is expected. The heterogeneous climb of the dislocation may be enhanced here, since the specimens are strained by a small amount prior to irradiation⁽¹⁴⁾. Weertman has shown that helical dislocations are the equilibrium form regardless of the initial orientation of dislocations after a deformation⁽²³⁾. Some of the superjogs are efficient anchoring points which contribute to formation of tangled dislocations. Others will be dragged along with the dislocations⁽¹⁹⁾. As long as the size of the superjogs is of the order of only several atomic distances, thermal activation may assist the superjogs in jumping from one position to the next by an atomic distance⁽²⁴⁾.

Although an estimate of hardening effect due to the formation of tangled dislocation cannot be made, the effect due to the dragging of a superjog may be estimated by assuming that the thermally activated motion of the superjogs is responsible for the observed hardening.

If we set the activation energy for the process to be U , a rate of shear strain $\dot{\gamma}$ can be given by⁽²⁵⁾

$$\dot{\gamma} = NAbv_0 \exp(-U/dT)$$

where N is the number of dislocation segments bounded by superjogs in a unit volume, A is the area swept out by a dislocation in one jump,

*The situation is similar to that discussed in reference 19. It should be noted, however, that interstitials and stationary dislocations are involved here, whereas vacancies and gliding dislocations were treated.

b is the Burgers vector, v_0 is the frequency factor (10^{13} sec^{-1}), k , the Boltzmann's constant and T , the temperature. When we take the average distance between the superjogs to be ℓ and the dislocation density ρ , N is $2\rho\ell^{-1}$. This is taken since the total length of the dislocation in unit volume is given by $2\rho^{(26)}$, and $A = \ell b$. The activation energy U can then be written as

$$U = U_0 - v(\tau' - \tau) \quad (6)$$

where U_0 is the activation energy for a jump without the external stress, and τ and τ' are the shear stresses before and after the irradiation, respectively. v is an activation volume which is given by $v = b\ell\lambda$ where λ is a jumping distance. Since $\lambda = b$ and $v = b^2\ell$, Eq. (6) may be rewritten as:

$$\dot{\gamma} = 2\rho b^2 v_0 \exp[v(\tau' - \tau) - U_0]/kT \quad (7)$$

Assuming v_0 and U_0 to be independent of $\dot{\gamma}$, we obtain the activation volume as a function of strain rate, $\dot{\gamma}$, and the shear stresses τ' and τ :

$$v = \frac{kT}{\Delta\tau_1 - \Delta\tau_2} \log \frac{\dot{\gamma}_1}{\dot{\gamma}_2} \quad (8)$$

where $\Delta\tau = \tau' - \tau$, which is the change due to irradiation.

From the experiments, we have $\Delta\tau_1 = 36 \text{ g/mm}^2$ and $\Delta\tau_2 = 10 \text{ g/mm}^2$ for the electron dose of $1.2 \times 10^{17} \text{ el/cm}^2$. Since $\dot{\gamma}_1/\dot{\gamma}_2 = \dot{\epsilon}_1/\dot{\epsilon}_2$, we obtain $v = 1 \times 10^{-20} \text{ cm}^3$ or $\ell = 2.9 \times 10^2 b$. If we substitute these values together with $\rho = 10^8$ and $v_0 = 10^{13}$ into Eq. (6) we obtain 0.15 eV for the value of U_0 . For the present case, the number of the interstitials which have arrived at the edge dislocation of length ℓ is about 6.6×10^2 . If we assume about 10% of them form a superjog,

its size is about $8b$ in both height and width. The size of the superjog may be larger or smaller than this, but there is no possible way to determine it accurately. As to the activation energy for motion of the superjog, it can be estimated, at least to some extent, if we have data on the stress necessary to move a dislocation on other than close-packed planes. The theory of the Peierls stress⁽²⁷⁾ is too incomplete to predict this, however. On the basis of experiments we have an indication that the stress is higher on non-close-packed planes. Thus, we must be content with the fact that the above value of U_0 is not unreasonable when comparing it to an activation energy for migration of an interstitial.

As pointed out earlier, the nature of Stage III has not been understood clearly. Various models have been proposed for Stage III recovery, as recently discussed by Sosin and Rachal⁽⁷⁾. None of them, however, can explain all of the observed features of Stage III. All of the interstitial models are in conflict with the present observation for the following reasons: 1) The observed hardening can only be explained by assuming that a part of their vacancy counterparts are left in the matrix, which would contribute to the residual resistivity after Stage III. No such residual resistivity is actually observed^(6,7). 2) Furthermore, theoretical calculations predict that a body centered interstitial or a crowdion is not a stable configuration^(28,29). Thus, the two-interstitial model^(7,15), which appears to explain the kinetics of resistivity recovery after irradiation, is not justified, and further studies are necessary to understand the nature of Stage III fully. However, the decrease in flow stress observed after the room temperature

annealing in the present experiments can be best explained by migration of vacancies or divacancies which result in destruction of the interstitial jogs.

C. Interruption Yielding

We will now discuss the yield drop resulting from an interruption of the tensile test. The phenomenon has been reported on polycrystalline aluminum and also other f.c.c. metals after straining to certain amounts (5% or more) at low temperatures^(8,30,31). It has been interpreted to be due to the Cottrell locking of dislocations by the interstitials which are formed during the straining, by the following reasons: 1) the magnitude of the yield drop was larger for a larger amount of prestrain, 2) only the defect mobile at the testing temperature was an interstitial, and 3) stress-strain curves after the yield point were identical to that obtained in the absence of aging. In the present experiment, the interruption yield drop which is observed at the early stage of deformation in only the irradiated crystals, has very similar characteristics, namely, the short aging time and the shape of the yielding phenomenon⁽⁸⁾. Since the strain rate dependence of the yield drop is also observed, it appears that the Cottrell effect of point defects is responsible for the observed yield drop, although the aging kinetics has not been studied enough to give a definite time law. Since unirradiated crystals have not shown a yield drop at the early stage of deformation with comparable interruption time and the vacancies are immobile at the temperature of testing, the defects responsible must be the interstitials formed by the irradiation. Therefore, the interstitials which are released from the trapping sites at impurity atoms by the

glide dislocations are suggested to be responsible for the yield drop.

If all the impurity trapped interstitials have been released from the sites and attracted to the dislocations, the number of the interstitials for an electron dose of $1.2 \times 10^{17} \text{ el/cm}^2$ is about $6 \times 10^{15} \text{ cm}^{-3}$, which compared to the number of sites along edge dislocations of $3.4 \times 10^{15} \text{ cm}^{-3}$. This number of interstitials strongly depends, of course, on the interaction and the distribution of the trapped interstitials and dislocations and not all of them will be attracted to the dislocations. Assuming the volume change of an interstitial, Δv , is spherical, the energy of the elastic interaction U_I , between the interstitial and an edge dislocation is given by⁽³²⁾:

$$U_I = \frac{Gb\Delta v}{\pi} \cdot \frac{\sin \alpha}{R} \quad (9)$$

where R and α are the polar coordinates about the dislocation.

U_{max} is about 1.2 eV when $R = b$ and $\sin \alpha = 1$. With this interaction energy and the estimate of the binding energy between an interstitial and an impurity atom, it is shown in the Appendix that about 20% of the impurity trapped interstitials are released from the sites when an irradiated crystal is strained in shear by 0.5%. Once they are released, they are expected to migrate to dislocations. Thus, the yield drop of the observed magnitude can be caused due to the Cottrell locking by the interstitials.

In the above discussion, we have assumed that interstitials are not absorbed immediately at superjogs on dislocations. This seems valid, at least for the time interval of the test interruption.

If our assumption is correct, a yield drop would not be observed after a prolonged test interruption, but an increase in the flow stress should be expected. Experiments on this point will be carried out in the near future.

ACKNOWLEDGEMENTS

The authors would like to express their appreciation to Prof. T. Mura for valuable discussions. They would also like to thank Dr. T. H. Blewitt and Prof. J. B. Cohen and M. E. Fine for reading this manuscript. The research upon which this paper is based was supported by the Advanced Research Projects Agency of the Department of Defense. The irradiation was carried out in the Northwestern University Van de Graaff facility which was set up largely through U. S. Atomic Energy Commission support.

REFERENCES

- 1) G. H. Vineyard, "Strengthening Mechanisms in Solids," ASM seminar p. 109 (1962)
- 2) C. E. Dixon and C. J. Meechan, Phys. Rev. 91, 237 (1953)
- 3) H. Dieckamp, NAA - SR - 1452 (1955)
- 4) M. J. Makin and T. H. Blewitt, Acta Met. 10, 241 (1962)
- 5) F. Seitz and J. S. Koehler, "Solid State Physics," Vol. 2, F. Seitz and D. Turnbull, editors, Academic Press, New York, (1956) p. 307
- 6) P. G. Lucasson and R. M. Walker, Phys. Rev. 127, 485 (1960)
- 7) A. Sosin and L. H. Rachal, Phys. Rev. 130, 2238 (1963)
- 8) A. R. C. Westwood and T. Broom, Acta Met. 5, 77 (1957)
- 9) P. Haasen and A. Kelly, Acta Met., 5, 192 (1957)
- 10) L. D. Landau, J. Phys. (U.S.S.R.), 8, 201 (1944)
- 11) W. A. McKinley and H. Feshbach, Phys. Rev. 74, 12 (1948)
- 12) J. S. Marshall and A. G. Ward, Can. J. Research, A15, 39 (1937)
- 13) J. W. Corbett, R. B. Smith and R. M. Walker, Phys. Rev. 114, 1452, 1460, (1959)
- 14) H. G. F. Wilsdorf and J. Schmitz, J. Appl. Phys. 33, 1750 (1962)
- 15) C. J. Meechan, A. Sosin and J. A. Brinkman, Phys. Rev. 120, 411, (1960)
- 16) N. F. Mott, J. Inst. Metals, 72, 367 (1946); "Imperfections in Nearly Perfect Crystals," W. Shockley, editor, Wiley, New York (1952)
- 17) F. R. N. Nabarro, Proc. Phys. Soc. 58, 669 (1948)
- 18) R. L. Fleischer, Acta Met. 9, 996 (1961)
- 19) D. Kuhlmann-Wilsdorf, R. Maddin and H. G. F. Wilsdorf, "Strengthening Mechanisms in Solids," p. 137, ASM seminar (1962)

- 20) T. H. Blewitt, R. R. Coltman and C. S. Klabunde, Proc. Conf. of Crystal Lattice Defects, Kyoto, Japan, III, p. 283 (1962)
- 21) R. L. Fleischer, Acta Met. 10, 835 (1962)
- 22) R. M. Thompson and R. W. Balluffi, J. Appl. Phys. 33, 803 (1962)
- 23) J. Weertman, Trans. A.I.M.E., (to be published)
- 24) A. H. Cottrell, "Vacancies and Other Point Defects in Metals and Alloys," J. Inst. Metals, p. 1, (1958)
- 25) A. Seeger, Proc. Conf. Dislocations and Mech. Prop. of Crystals, J. Wiley and Sons, New York, p. 243 (1957)
- 26) G. Schoeck, J. Appl. Phys. 33, 1745 (1962)
- 27) A. Seeger, "Handbuch der Physik," Vol. 7/1 Springer, Berlin, p. 383 (1956)
- 28) R. A. Johnson and E. Brown, Phys. Rev. 127, 446 (1962)
- 29) K. H. Brennemann, Phys. Rev. 124, 669 (1961); Z. Physik 165, 445 (1961)
- 30) G. F. Bolling, Phil. Mag. 4, 537 (1959)
- 31) H. K. Birnbaum and F. R. Tuler, J. Appl. Phys. 32, 1403 (1961)
- 32) B. A. Bilby, Proc. Phys. Soc. A63, 191 (1950)

FIGURE CAPTIONS

- Fig. 1. Axial orientations of crystals tested.
- Fig. 2. Schematic drawing of the cryostat.
- Fig. 3. Typical stress-strain curves of irradiated and unirradiated crystals.
- Fig. 4. Fractional increase in shear stress against electron dose.
- Fig. 5. Effect of test interruptions in an irradiated crystal.
- Fig. 6. Effect of annealings at room temperature.

APPENDIX

Suppose an impurity trapped interstitial is at the origin and an edge dislocation which lies on a slip plane $y = y_0$ runs parallel to the z -axis. When the dislocation is at x_0 , the interaction energy of Eq. (9) gives

$$U_I = A \sin \alpha / R = A y_0 / (x_0^2 + y_0^2) \quad (\text{A.1})$$

where $A = Gb\Delta v/\pi$.

The probability of release of the interstitial with the above interaction can be written as

$$P = v \exp(-E_m - B + U_i)/kT. \quad (\text{A.2})$$

Taking $v = 10^{13}/\text{sec}$, $E_m = 0.12 \text{ ev}$ and $B = 0.1 \text{ ev}$,⁽⁷⁾ the probability is unity for $U_i = 0.02 \text{ ev}$ at 77°K , which corresponds to $R = 60b$ for $\sin \alpha = 1$. This implies that impurity trapped interstitials are freed only from sites near dislocations.

During a small strain interval before test interruption, however, dislocations move rather extensively. The strain rate $\dot{\gamma}$ is equal to $v_0 \rho b$ where v_0 is an average velocity of the dislocations which is about $3.5 \times 10^{-4} \text{ cm/sec}$ in our case.

Suppose a dislocation moves on $y = y_0$ from $x = -\infty$ to $+\infty$. Then the total probability of release, P_t , can be given by an integral:

$$P_t = \int_{-\infty}^{\infty} P \, dt. \quad (\text{A.3})$$

Noting $v_0 = dx/dt$,

$$P_t = \frac{v}{V} \exp(-E_m - B)/kT \int_{-\infty}^{\infty} \exp U_i/kT dx$$

and substituting (A.1)

$$P_t = \frac{v}{V} \exp(-E_m - B)/kT \int_{-\infty}^{\infty} \exp A y_o/kT (x^2 + y_o^2) dx \quad (A.4)$$

The function in the integral falls off rapidly and a graphical integration gives $P_t \approx 1$ for $y_o = 48 \text{ \AA}$. Therefore, the interstitials are released from the trapping sites when they are within about 50 \AA above and below the slip plane.

When we have a strain interval of 0.5%, total area swept by dislocations can be given by $\Delta\gamma/b = 1/570 \text{ \AA}^{-1}$. Thus, about 20% of all the interstitials can be expected to contribute to the interruption yield drop phenomena.

In this calculation, random motion of dislocations is assumed, which tends to overestimate the total area swept. This assumption, however, is reasonable since dislocations leave their initial slip planes rather randomly in aluminum as observed by transmission electron microscopy.

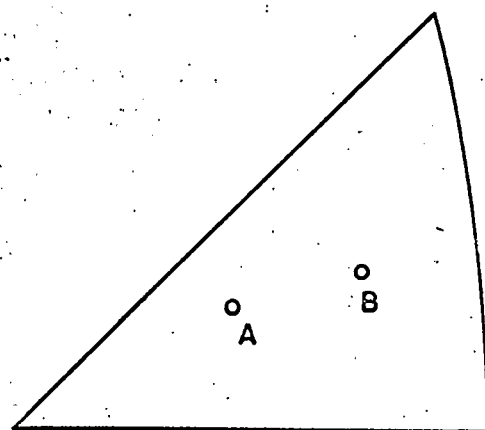


FIG 1

