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MASTER

PRESSURE INDUCED RECOVERY
OF IRRADIATION DAMAGE
IN MOLYBDENUM*

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PRESSURE INDUCED RECOVERY OF IRRADIATION DAMAGE
IN MOLYBDENUM

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and B. Mastel

INTRODUCTION

Thermally induced annealing of interstitials and vacancies in neutron irradiated metals is known to proceed in at least five major stages. Unfortunately there is no general agreement on the specific defect responsible for each of the stages. Most of the controversy has centered around the defect (or defects) responsible for the third recovery stage in irradiated metals. For fcc metals, one school of thought⁽¹⁻⁶⁾ holds that vacancies are migrating at this temperature, while several other investigators⁽⁷⁻¹⁰⁾ feel that it is the result of interstitial migration. In bcc molybdenum, the situation is much the same with as many advocates of the vacancy argument⁽¹¹⁻¹⁵⁾ as the interstitial mechanism.⁽¹⁶⁻²⁰⁾

Because an irradiated crystal occupies more volume than a perfect crystal, the application of pressure provides a driving force to anneal out the defects. However, at the same time that pressure is increasing the strain energy associated with each type of defect, it is also affecting the defect mobility. This latter effect has been studied quite extensively with respect to vacancy controlled diffusion in unirradiated solids, and it has been shown that the pressure effect on mobility far outweighs the driving force for single vacancy and vacancy cluster annealing. Diffusion coefficients have generally been found to be reduced by an order of magnitude or more at 20 kbars (1 kbar = 1000 atms.),⁽²¹⁾ while recrystallization of Cu²² and the annealing of prismatic vacancy loops in Al²³ requires higher temperatures under pressure than at ≤ 1 bar.

There have been no similar experiments performed on a metal containing only metal interstitial atoms because of the extreme difficulty of producing a sufficient number of interstitial atoms without drastically increasing the vacancy concentration. Theoretical treatments of pressure effects on the diffusion of single interstitial atoms have also been lacking.

The objects of the present investigation were to:

- determine whether pressure assists or retards the annealing of irradiation produced point defects in Mo,
- measure the activation volume of the defect involved in the third annealing stage, and
- determine the effect of pressure on the thermal stability of defect clusters in irradiated Mo.

EXPERIMENTAL

Polycrystalline, high purity (99.99%) Johnson Matthey molybdenum foil, 0.0075 cm thick, containing less than 10 ppm carbon, was used as starting stock. After the foils were cut into an appropriate geometry for four probe resistance measurements and for transmission electron microscopy studies, they were vacuum annealed at 1760°C for 20 minutes. The samples were irradiated to a fast fluence of 6.2×10^{18} n/cm² (E > 1 MeV) at a temperature of less than 40°C.

Samples were subjected to low pressure (< 1 bar) isothermal and isochronal anneals in a variety of ways: below 200°C they were heated in a silicone oil bath, at 210°C in a KNO₃-NaNO₃ bath, and from 300 - 1000°C in a vacuum at 10⁻⁵ torr by means of radiant heat lamps. The errors in the temperature of the oil baths and the vacuum anneals amounted to less than $\pm 5^\circ\text{C}$. The time of the isochronal anneals was two hours.

Identical samples were isochronally annealed under a hydrostatic pressure of 20 ± 1 kbars at temperatures from 25 to 490°C. The high pressure equipment, consisting basically of a piston-cylinder unit, and the calibration techniques are described elsewhere.⁽²⁴⁾ The samples were contained in a collapsible can arrangement⁽²⁴⁾ filled with a 50-50 mixture of n- and isopentane below 300°C and with a eutectic mixture of NaK above 350°C. Because unirradiated control specimens included in each test showed essentially no resistance change over the entire temperature range explored, the pressure was considered truly hydrostatic at all times. The error limits on the temperatures were $\pm 5^\circ\text{C}$ after the effect of pressure on the thermocouple readings had been taken into account.

The electrical resistance was measured at liquid helium temperatures with a high sensitivity (0.01 microvolt) voltage measuring system.⁽²⁵⁾ The lattice parameter measurements were made with Cu radiation, and precision values were obtained by using a Nelson-Riley extrapolation.⁽²⁶⁾ The Vickers hardness measurements are reported for a 50 g load⁽²⁷⁾ and the electron microscopy was performed in a manner described previously.⁽²⁸⁾ The defect cluster density was determined by electron microscopy of foils for which the thickness was established by the interference fringe technique; 400 - 1000 clusters were counted for each data point (except at the very high temperatures where only a few clusters per micrograph were found).

RESULTS

The results of the electrical resistance measurements are summarized in Figure 1. Significant features of the low pressure curve as determined from differential annealing curves⁽²⁵⁾ are the rather large recovery at stage III (150°C), a very small recovery peak at 425°C which we choose to

call stage IV-A for reasons which will be explained later, and a final recovery at a stage IV-B (575°C).

Combination of low pressure isothermal and isochronal resistance data into a Meecham-Brinkman⁽²⁹⁾ plot gives an activation energy of 1.25 ± 0.09 eV for stage III annealing. This analysis, reported in more detail elsewhere,⁽²⁵⁾ showed the reaction in order to vary with temperatures from 3.4 at 135°C to 2.0 at 149°C and 1.7 at 164°C.

In the samples annealed under 20 kbars pressure, stage III recovery of resistance is lowered by 60 - 90°C. Only one other resistance recovery stage was found, at 375°C, and recovery was complete at 430°C under pressure. The results of the lattice parameter recovery studies⁽²⁶⁾ are summarized in Figure 2. Like the resistance measurements, there is a fairly large amount of recovery at 150°C (stage III) and 400°C (stage IV-A) at low pressure. The lattice parameter does not show a stage IV-B recovery, but instead anneals at 750°C (stage V). The high pressure results are also similar to the resistance studies in that only two recovery peaks are noted at 90°C and at 350°C.

The results of the transmission electron microscopy studies reported elsewhere⁽²⁷⁾ are summarized in Figure 3. The plot of the percentage of clusters remaining versus isochronal annealing temperature shows some rather odd behavior in that cluster density initially declines and then increases slightly. This is followed by a broad annealing band from 400 - 800°C in the low pressure anneals and from 300 - 490°C in the samples annealed under pressure. Furthermore, the application of pressure apparently increases the number of clusters observable at room temperature. Although the errors involved in counting cluster densities ($\sim \pm 10-15\%$) are greater than any other measurement errors reported here, the fact that these recovery peaks correspond to property changes observable by other more accurate techniques supports the

belief that they are real. Regardless of the exact shape of the curve, it is found that the temperature at which the 25-40A diameter defect clusters anneal out in vacuum is lowered by $\sim 300^{\circ}\text{C}$ by the application of 20 kbars hydrostatic pressure.

The hardness recovery data reported in more detail elsewhere⁽²⁸⁾ are summarized in Figure 4. During the low pressure anneal, the hardness shows a gradual increase over the as-irradiated value up to 600°C , after which it recovers in a $300\text{--}400^{\circ}\text{C}$ temperature interval. The recovery peak here is 750°C (stage V), and it corresponds to the final recovery of the defect clusters (Figure 3) and the lattice parameter (Figure 2). In the high pressure annealed samples, it is found that the hardness increases by some 70% after a 20 kbar room temperature test. Further isochronal annealing produces a slight increase at stage III, after which the hardness is unaffected up to 400°C . Rapid annealing then takes place in the region between 400 and 490°C , again coincident with the final defect cluster annealing.

DISCUSSION

From the data in Figures 1-4, it is obvious that pressure has a considerable effect on the annealing of both point defects and defect clusters. To better compare the information from the four types of measurements, the differential annealing peaks calculated from Figures 1-4 have been plotted in Figure 5.

Low Pressure Annealing

In the low pressure differential annealing curves of Figure 5, four distinct recovery peaks occur above room temperature. The results are summarized in Table I. The recovery of electrical resistance is in complete agreement with previous investigations,^(12,13,16-19,30,31) as are the lattice

parameter measurements.⁽³²⁾ The activation energy agrees quite well with that found previously,^(11-13,16-19,30) and the complex kinetics of stage II annealing corresponds to work by Nihoul,⁽¹⁶⁾ Gray,⁽³²⁾ and DeJong.⁽¹⁹⁾ The hardness increase with increasing temperature is somewhat different than that reported by Downey and Eyre,⁽¹⁴⁾ but the difference might be explained by the fact that the total exposure to their samples was 40 times greater than that in the present tests. On the other hand, the general annealing of the irradiation induced hardness at 750°C (0.35 Tm) does agree with their data.

The annealing of the clusters depicted in Figure 3 does not appear so strange if it is plotted on a semi-log plot after the fashion of Gower and Eyre⁽¹⁴⁾ or Mastel and Brimhall.⁽³³⁾ However, a semi-log plot can be misleading when associating the defect cluster change with the linear change in other properties, such as electrical resistance, lattice parameter, etc. It is interesting to note the same sort of behavior shown in Figure 3 has recently been observed⁽³⁴⁾ in W irradiated to seven times the fluence in the present study. The effect in W is more pronounced than observed here, and the mechanical properties seem to reflect first a decrease, then an increase, and finally a decrease in the number of clustered defects.

High Pressure Annealing

In general, the affect of annealing under pressure is to lower stages III and V and perhaps even stage IV. The shift in stage III is approximately -3°C/kbar, and for stage V it is about -15°C/kbar. Both the direction and magnitude of these shifts is somewhat surprising, as the compressibility of molybdenum is very small.⁽³⁵⁾ The direction is opposite to what one might expect based on previous diffusion experiments.⁽²¹⁾ Furthermore, the effect seems to apply to point defects (which are generally assumed to

migrate in stage III) and clustered defects as well.

One method of analyzing the pressure effect on stage III is to assume that the migration of the defect responsible for the observed property changes is an activated event. The rate at which the concentration of defects disappear at any temperature T is given by⁽³⁶⁾

$$\frac{dn}{dt} = F(n)K_0 \exp(-H/kT) \quad (1)$$

where n = concentration of defects, $F(n)$ = a continuous function of n , K_0 = pre-exponential constant, and H = activation enthalpy of the process. Since $H = E + P\Delta V$, where E is the activation energy, ΔV is the activation volume of the process, and P is the applied pressure, it is obvious that the application of pressure during the annealing of defects at a constant temperature may considerably alter their rate of disappearance. The effect will be to accelerate the process if ΔV is negative and to retard the process if ΔV is positive. If the annealing is performed isochronally, curves determined at different pressures will be displaced from one another in a manner determined by the magnitude of the $P\Delta V$ term. As a result, if a horizontal line is drawn at a constant defect concentration, for example n_i , then the quantity

$$-\frac{1}{K_0} \int_{n_0}^{n_i} \frac{dn}{F(n)} = t \exp\left(-\frac{E + P_j \Delta V}{kT_j}\right) \quad (2)$$

should be constant for any T_j and P_j where T_j is the temperature at which the defect concentration reaches n_i in an annealing time t under a pressure of P_j . This assumes that the activation energy, the pre-exponential term, and the order of the reaction is not changed by the applied pressure. Since the time of isochronal annealing is constant by definition, Equation (2) reduces to Equation (3) at constant n_i .

$$\exp\left(-\frac{E + P_j \Delta V}{kT_j}\right) = \text{constant} \quad (3)$$

Rearranging,

$$\Delta V = \frac{E(T_{j+1} - T_j)}{P_{j+1}T_j - P_jT_{j+1}} \quad (4)$$

where the T_{j+1} is now the temperature at which the defect concentration reaches n_1 in time t under a pressure of P_{j+1} . Equation (4) can then be used to calculate the activation volume from the measured shift, with pressure, in the isochronal annealing curves and the activation energy of the process which can be measured at very low pressure.

Insertion of appropriate values for the terms in Equation (4) ($E = 1.25$ eV, $P_{j+1} = 20$ kbar, $P_j = 0$, $T_j = 423^\circ\text{K}$, and $T_{j+1} = 363^\circ\text{K}$) gives $\Delta V = -0.9V^*$ where V^* is the atomic volume. This implies that the defect responsible for stage III annealing has a negative activation volume for migration almost equal to the molar volume. The physical significance of such a value is hard to determine, although negative activation volumes have been measured experimentally in a few isolated cases,⁽³⁷⁻³⁹⁾ and they have also been predicted by the computer calculations of Johnson for vacancies in gamma Fe⁽⁴⁰⁾ and interstitials in copper.⁽⁴¹⁾ In these latter cases, the calculated values range from -0.01 to $-0.05 V^*$, much lower than indicated by Equation (4).

One possible defect which might have such a large negative activation volume is a divacancy. Beeler⁽⁴²⁾ has shown that considerable numbers of divacancies exist after computer-simulated irradiation at 0°K , and their relative numbers actually increase as the "solid" is allowed to heat up. Since ΔV_m may be interpreted as a measure of the relaxation of many atoms around a migrating defect, it is possible that the saddle point configuration corresponds to a volume decrease.

Another explanation of the pressure effect on stage III would be a negative pressure coefficient of the binding energy between an interstitial and a trap. This trap could be an impurity atom such as Fe, which would be substitutional in Mo and is undersized. In this case, an interstitial Mo atom might be accommodated at the impurity site easier than in an interstitial site surrounded by like Mo atoms. It has recently been calculated⁽⁴³⁾ that a considerable binding energy exists between a substitutional iron impurity atom and a Mo interstitial. Since the close proximity of the two atoms means that they are in a position where the potential energy is varying rapidly with distance, a slight shortening of the lattice parameter might have disproportionately large effects.

One other possibility is the forced recombination of vacancies and interstitials. It has been postulated⁽⁴⁴⁾ that there is a radius around any defect such that if a defect of opposite type comes within that distance, the two will immediately interact and annihilate one another. Under the action of pressure, this radius could be increased such that defects do not have to move as far to be captured. Something similar to this has been observed in the stress induced annealing of chemical radiation damage in various compounds.⁽⁴⁵⁻⁴⁸⁾

This latter argument might well apply to the apparent reduction in the temperature required to anneal defect clusters. Barnes⁽⁴⁹⁾ has postulated that the disappearance of defect clusters during low pressure anneals is due to cluster migration and interaction. Since the size of the clusters in the present tests do not change significantly during the test, this argument would require that vacancy clusters are present. In recent high temperature irradiations, Brimhall, et. al.,⁽⁵⁰⁾ have observed vacancy clusters in Mo, and Rao and Thomas⁽⁵¹⁾ also have found some large vacancy loops after postirradiation anneals.

Assuming that there were both vacancy and interstitial loops available, then one would have to examine the effect of a hydrostatic pressure on the glide and/or climb of such loops. Norris⁽²³⁾ has concluded that for climb of prismatic vacancy loops, the reduction in the pipe diffusion coefficient would be greater than the strain energy induced by the pressure. Presumably the same would apply to interstitial loops, but in view of the negative shift in stage III annealing, this should be examined in greater detail.

The effect on glide would be connected with the effect of pressure on the critical resolved shear stress which would be expected to increase with pressure. This would make it harder for the loops to glide, but the effect of pressure on the interaction distance may override this.

General Observations

This study represents the first instance where electrical resistance, lattice parameter, hardness, and electron microscopy measurements have been made on the same material. Direct correlations between defects and property values can be drawn from the recovery behavior of this material, whereas they would be extremely difficult and risky to make from materials of different purities, fabrication histories, or irradiation conditions. It is apparent that there are at least four recovery stages above room temperature instead of the three normally discussed. It is felt that the designation of the 0.24 Tm peak as IV-A and 0.31 Tm as IV-B would reduce the confusion that occurs when the 0.31 Tm peak is called stage IV and the 0.15 Tm peak stage III.

It appears that changes in cluster density do accompany changes in other properties, such as electrical resistance and lattice parameters, which are normally attributed to point defect migration. This should not be unexpected, however, because there is a high density of defect clusters, $3-4 \times 10^{16}/\text{cc}$ in the material used,⁽²⁷⁾ and considerable numbers of defects

can interact with these clusters.

The observable ($> 25 \text{ \AA}$)^o defect clusters contribute very little, if anything, to the electrical resistance increase. This is substantiated by the fact that the resistance increase is completely recovered, while some 30-50% of the observable defect clusters are still present in both the high and low pressure annealed samples (Figures 1 and 3).

The complete recovery of electrical resistance in the low pressure annealed sample before the final lattice parameter recovery stage signifies that the defect clusters, which are presumed to be mainly interstitial at high temperature, contribute some small amount to the lattice parameter. Why the lattice parameter anneals completely before the final cluster recovery or breakup of clusters under pressure occurs is not known at the present time, but the clusters remaining may have collapsed to loops or configurations where they contribute very little to the lattice parameter.

Finally, the hardness values seem to be associated, in some complex manner, with the cluster density. It is not known why the hardness is fairly constant over the temperature range where the cluster density is going through a minimum, especially in the high pressure case. In addition, it is found in both cases that there is a certain amount of hardness increase remaining even after the clusters are completely recovered.

CORRELATION WITH RECOVERY MODELS

Models proposed for recovery of irradiation damage seem to follow one of two general patterns. One model, referred to as model A, involves the migration of interstitials (either free or trapped) in stage III, migration of vacancies in stage IV-A, vacancy cluster break-up in stage IV-B, and interstitial defect cluster annealing in V. A second model, B, would attribute stage III to vacancy migration, stage IV-A to some effect due to

impurities, stage IV-B with vacancy cluster break-up, and stage V with interstitial cluster annealing. A third model, C, which we feel is equally as acceptable, if not more probable, would attribute stage III to divacancy motion, IV-A to vacancy migration, IV-B to vacancy cluster break-up, and V to interstitial cluster annealing. All three models can explain electrical resistance, lattice parameter changes, and stored energy releases in stage III. However, it is difficult for model A to explain the stage III recovery found in quenched molybdenum.⁽¹⁶⁾ It is also hard to understand why the defect cluster density would decrease in stage III when, if anything, the migration and coalescence of those interstitials which are not annihilated would form more clusters. Models B and C could very well explain the latter observation by assuming those divacancies or vacancies which do not annihilate trapped interstitials, interact at interstitial clusters, and reduce their size below the resolution limit of the microscope. The negative activation volume for migration tends to rule out model B because of the previous results in high pressure diffusion studies.

The recovery at stage IV-A which shows a lattice parameter decrease and a corresponding cluster density increase is difficult to explain by model A, because it assumed that interstitials had already been released from traps; moreover, model B would have difficulty in explaining the increased cluster density, since it is assumed that the clusters are not groups of impurity atoms. The migration of a vacancy in this stage is compatible with quenching experiments⁽¹⁶⁾ and with increased cluster density (assuming that the clusters were vacancy in nature).

The observation by Brimhall, et. al.,⁽⁵⁰⁾ of small vacancy cluster break-up at the IV-B stage would support all three models, as would the lack of any further lattice parameter decrease.

All the models assume interstitial cluster annealing for the fifth stage, and this is amply supported by the recovery of hardness, observable cluster density, and final lattice parameter recovery at this temperature.

It appears that model C can most readily explain the observation of this study as well as some from other investigators, but the evidence is not conclusive. Further experiments utilizing both low and high pressure techniques are required.

CONCLUSIONS

From the low pressure experiments, it is concluded that:

- There are four recovery peaks above room temperature at approximately 0.15, 0.24, 0.31, and 0.35 Tm.
- The activation energy for stage III annealing is 1.25 ± 0.09 eV.
- The contribution of observable defect clusters to electrical resistance is very small.

From the 20 kbar pressure experiments, it is found that:

- The lattice parameter, electrical resistance, and cluster density stage III recovery peaks are lowered by 60°C.
- The activation volume of migration for the defect migrating in stage III is $-0.9V^*$.
- The number of recovery peaks is reduced from 4 to 3 under pressure.
- Both the electrical resistance and lattice parameter increases are completely recovered at 400°C under pressure.
- The defect cluster density and hardness stage V recovery peaks are lowered by 300°C.

TABLE 1

SUMMARY OF ANNEALING STAGES FOR ISOBARIC-ISOCRONAL ANNEALING
OF IRRADIATED MOLYBDENUM

<u>Stage</u>	<u>1 bar</u>			<u>20 kbar</u>		
	<u>Temp.</u> <u>Range, °C</u>	<u>T/T_m*</u>	<u>Property**</u> <u>Changing</u>	<u>Temp.</u> <u>Range, °C</u>	<u>T/T_m*</u>	<u>Property**</u> <u>Changing</u>
III	150	0.15	ER, LP, CD	90	0.13	ER, LP, CD
IV-A	400-450	0.24	ER, LP, CD	350-375	0.22	ER, LP, CD
IV-B	575	0.29	ER	450-475	0.25	CD, H
V	750	0.35	LP, CD, H			

* Used T_m as melting temperature at 1 bar

** ER = Electrical Resistance

LP = Lattice Parameter

CD = Cluster Density

H = Hardness

*Put parentheses
around year.*

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Figure No.

Caption

- 1 Effect of Hydrostatic Pressure on the Isochronal Recovery of the Irradiation Induced Electrical Resistance Increase in Molybdenum.
- 2 Effect of Hydrostatic Pressure on the Isochronal Recovery of the Irradiation Induced Lattice Parameter Increase in Molybdenum.
- 3 Effect of Hydrostatic Pressure on the Isochronal Annealing of Observable Defect Clusters in Irradiated Molybdenum.
- 4 Effect of Hydrostatic Pressure on the Isochronal Annealing of the Irradiation Induced Hardness Increase in Molybdenum.
- 5 Comparison of the Differential Recovery Curves for Irradiated Mo Isochronally Annealed at (a) Low ≤ 1 Bar and (b) High, 20,000 Bars Pressure.

ELECTRICAL RESISTANCE

KE 10 X 10 TO THE CENTIMETER 46 1510
18 X 25 CM. MADE IN U.S.A.
KEUFFEL & ESSER CO.

PER CENT RESISTANCE INCREASE REMAINING

100

90

80

70

60

50

40

30

20

10

0

0

100

200

300

400

500

600

700

800

TEMPERATURE °C

≤ 1 BAR

20,000 BARS

LATTICE PARAMETER

2

KE 10 X 10 TO THE CENTIMETER 46 1510
18 X 25 CM. KEUFFEL & ESSER CO.
MADE IN U.S.A.

PER CENT ORIGINAL CLUSTERS REMAINING

HARDNESS

DIFFERENTIAL ANNEALING RATE (ARBITRARY UNITS)

ELECTRICAL RESISTANCE

LATTICE PARAMETER

CLUSTER DENSITY

HARDNESS

DIFFERENTIAL ANNEALING RATE

TEMPERATURE °C