HYDROSTATIC PRESSURE INDUCED
ANNEALING OF DEFECT CLUSTERS IN
IRRADIATED MOLYBDENUM

G. L. Kulcinski and B. Mastel

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HYDROSTATIC PRESSURE INDUCED ANNEALING OF DEFECT

CLUSTERS IN IRRADIATED MOLYBDENUM

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ABSTRACT

Irradiation of high purity molybdenum to 6.2×10^{18} n/cm² ($E > 1$ MeV) at 40°C produces 3.7×10^{16} observable defect clusters/cc. The average cluster diameter is 25-35 Å. Postirradiation annealing at ≤ 1 bar causes the cluster density to decrease during stage III (150°C), increase during stage IV-A (400°C) and decrease to zero during final recovery at stage V (750°C). Isochronal recovery under 20 kbars of hydrostatic pressure shows that stage III is lowered to 90°C and stage V to 450°C. The recovery stages are compared to those obtained from previous electrical resistance and lattice parameter studies to establish a tentative model for radiation damage annealing in molybdenum.

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INTRODUCTION

When molybdenum is irradiated at $\sim 40^{\circ}\text{C}$ to fast neutron fluences in excess of 10^{18} n/cm², small observable defect clusters begin to appear in the lattice.⁽¹⁾ Transmission electron microscopy shows that initially these clusters are 25-30Å in diameter. As the fast fluence increases to 10^{19} n/cm², the density of clusters increases to $\sim 3-4 \times 10^{16}$ /cc and the average size increases to $\sim 50-70\text{Å}$.⁽¹⁾ As irradiation proceeds to still higher fluences, the number of clusters remains relatively constant but their size continues to increase. Downey and Eyre⁽²⁾ find that exposure to 2.5×10^{20} n/cm² results in clusters up to 200Å in diameter. Defect cluster size is also known to be a sensitive function of purity^(1,3,5) and irradiation temperature.^(4,5)

Postirradiation studies of these defect clusters show that rather high temperatures are required for their recovery. Mastel and Brimhall⁽¹⁾ show that the small clusters are stable $\sim 800^{\circ}\text{C}$ with little or no coarsening after which they rapidly anneal out. Downey and Eyre⁽²⁾ report much the same behavior up to 800°C on more heavily irradiated material but the recovery takes place over a larger temperature range and many of the clusters grow into loops. Brimhall et al⁽⁵⁾ have demonstrated that small clusters ($< 70\text{Å}$) formed during high temperature irradiation are annealed out above 700°C whereas much larger loops ($> 70\text{Å}$) actually grow in size at temperatures up to 1000°C .

Identification of the clusters has been carried out by many investigators.^(1,5,6) For material irradiated below 100°C it is found that the clusters are predominantly interstitial in character with Burgers Vector $a/2 \langle 111 \rangle$. In material irradiated above the $0.15T_m$ (stage III) recovery temperature^(4,5) vacancy clusters have also been observed and, in one case, after a one hour $\sim 760^\circ\text{C}$ anneal of irradiated molybdenum.⁽⁶⁾

In recent studies on the effect of hydrostatic pressure on the isochronal recovery of irradiation induced electrical resistance increases⁽⁷⁾ and lattice parameter increases,⁽⁸⁾ it was shown that pressure caused these properties to recover at temperatures considerably lower than those in similar material annealed at low pressures. The 150°C, 425°C, and 575°C electrical resistance recovery stages in molybdenum were reduced to only two stages (90°C and 375°C) when the samples were annealed under pressure. Similarly, the 150°C, 400°C, and 750°C lattice parameter recovery stages were also reduced to only two stages at 90°C and 375°C. The loss of one of the stages and the fact that the final electrical resistance and lattice parameter recovery occurred at different temperatures at low pressure but at the same temperature at high pressure was difficult to explain on the basis of only two types of measurements. Since it is known that defect cluster annealing at low pressure takes place at $\sim 750^\circ\text{C}$, the microstructure revealed by transmission electron microscopy was studied in both the low and high pressure annealed samples to see if the loss of one of the recovery stages at high pressure could be explained. This report summarizes the results of the microscopy studies.

EXPERIMENTAL

The sample material, preirradiation heat treatments, irradiation data, and postirradiation low and high pressure techniques have been

previously described.⁽⁷⁾ Briefly, polycrystalline, high purity molybdenum containing less than 10 ppm of carbon was irradiated to 6.2×10^{18} n/cm² ($E > 1$ MeV) at a temperature of $\sim 40^\circ\text{C}$. The postirradiation annealing of these samples was carried out at two pressures, ≤ 1 bar* and 20 kbars. The isochronal anneal was two hours.

Specimens used for the transmission electron microscopy were the same as those for which the lattice parameter had been measured.⁽⁸⁾ The specimens were 0.0075 cm thick foils, 1 cm long and 0.5 cm wide.

The samples were thinned in an electrolyte of 25 ml HCl, 10 ml H₂SO₄, and 75 ml methyl alcohol cooled to 10°C . The applied voltage between the specimens and the electrodes was 18V and current densities were 0.9 amp/cm².

The average cluster size and density was determined from electron micrographs at a magnification of 120,000X. Accurate thickness values were obtained by noting the operating g value and by using the interference fringe method. Approximately 400-1000 clusters were counted at each temperature interval except for the very high temperature at which only a few clusters per micrograph could be found.

RESULTS

Figures 1-5 show representative micrographs of the low and high pressure annealed samples after various isochronal anneals. It should be noted that the cluster density of the as-irradiated sample is apparently increased slightly by a room temperature, 20kbar anneal, Figure 1. After a 300°C anneal, the cluster density is slightly reduced after both the low and high pressure treatments, Figures 2a, 2b. At 400°C the low pressure anneal causes little change in cluster density, Figure 3a, while the high

* 1 bar $\approx$ 1 atmosphere

pressure treatment causes a considerable drop in the defect cluster density, Figure 3b. Annealing at approximately 500°C causes all the defects to be removed from the sample subjected to 20 kbar pressure annealed, Figure 4b, while there is still a significant number of clusters remaining in the sample annealed at low pressure, Figure 4a. At 700°C the cluster density is further reduced during the low pressure anneal, Figure 5a, and at 800°C the clusters are essentially all removed, Figure 5b.

An important point to note in Figures 1-5 is the low dislocation density in both the low and high pressure annealed samples. This indicates in the latter case that the pressure was truly hydrostatic at all times and that channeling of dislocations⁽⁹⁾ could not account for the cluster removal.

The density of clusters is shown in a more quantitative fashion in Table I and Figures 6-8 show these results in a graphical form. The cluster density measurements are assumed to be accurate to within 10-15%. The data is shown in three different ways to point out that the method of display can have a significant effect on the interpretation of the recovery. First, in semi-log plot (Figure 6) it appears that the defect cluster density is relatively constant with temperature at low pressure, but anneals rapidly between 700-800°C. Much the same is true for the high pressure data except that the cluster density does show a decline with increasing temperature up to 400°C after which the recovery is fairly rapid.

Plotting the same data from Table I in linear fashion gives the curves in Figure 7. Here the low and high pressure anneals show recovery peaks at 150°C and 90°C, respectively. After the stage III recovery, Figure 7 shows an increase in defect concentration followed by a broad annealing

TABLE ISummary of Cluster Density in Irradiated Mo After Two HourIsochronel Anneals at ≤ 1 Bar and at 20 Kbars

<u>≤ 1 Bar</u>	
Annealing Temperature °C	Average Number of Clusters/cc $\times 10^{-16}$
As Irr.	3.7
100	3.5
150	3.1
200	3.1
300	3.2
400	3.5
500	2.6
605	1.9
700	1.3
800	0.009
 <u>20,000 Bars</u>	
25	4.2
80	4.0
90	3.8
110	2.9
205	2.4
300	2.6
350	1.9
400	1.2
430	0.9
490	0.002

band (200-400°C) in the samples annealed under both low and high pressures. Figure 8 is similar to Figure 7 except that the recovery is plotted as a function of the as-irradiated defect density. The significance of these curves will be pointed out later.

It was also discovered that the average cluster size does not vary significantly over the temperature and pressure range studied, being 25-35Å for the low pressure anneals and 30-40Å for the high pressure anneals. There was an apparent slight coarsening of clusters at higher temperatures, but it is difficult to substantiate this observation because of the extremely small size involved.

DISCUSSION

LOW PRESSURE ANNEALS

The variation of cluster density with isochronal annealing temperature is consistent with previous results published by Mastel and Brimhall⁽¹⁾ and Downey and Eyre⁽²⁾ when plotted on a semi-log diagram (Figure 6). However, it can be misleading to look at the cluster annealing from this standpoint alone as indicated by Figures 7 and 8. In these latter Figures, it can be seen that considerable annealing does take place before the final "apparent" annealing stage at the 700-800°C and 400-500°C temperature ranges for the low and high pressure anneals respectively. Quantitatively, it is found that almost 2/3 of the clusters have annealed prior to the steep drops or annealing stages indicated by Figure 6. Although there are considerable uncertainties involved in counting defect clusters, even error limits of $\pm 15\%$ allow a certain amount of fine structure to emerge. The first is an apparent drop in cluster density

during stage III annealing^{*}; the second is the apparent increase in defect clusters at stage IVA; and the third is the final recovery centered around stage V. Figure 9a shows how the absolute value of these recovery peaks correlates with the previous electrical resistance and lattice parameter measurements. It is difficult to state that there is no recovery peak at stage IVB because of the broad temperature range over which cluster annealing takes place. For the present discussion, we will not assign a recovery peak to that temperature, but the possibility can not be ruled out completely.

The rather close association of the defect cluster annealing temperature with those found by more accurate techniques leads to the belief that the defect cluster density does change at the various recovery stages. In addition, similar behavior has been recently discovered in W⁽¹⁰⁾ irradiated to seven times the fluence considered in this study. In the former work on W, it was found that the mechanical properties closely followed the decrease, then increase, and finally the recovery of the defect clusters. Because the clusters in the present study are so small (25-35Å in diameter), a slight reduction in size could cause them to "disappear" because of low contrast and the finite resolution limit of the microscope. Similarly, clusters which are just below the resolution limit could "appear" by the addition of a few defects. Hence, it is not necessary to postulate the complete disappearance or the formation of clusters from very small nuclei to explain the variations.

* The notation of various stages will follow that outlined in

Reference 8, namely: Stage III $\approx 0.15T_m$
 Stage IVA $\approx 0.24T_m$
 Stage IVB $\approx 0.29-31 T_m$
 Stage V $\approx 0.35T_m$

Since the majority of clusters are assumed to be interstitial in character, the above arguments would indicate that interstitial clusters are being annihilated at stage III. In view of the existing controversy and confusion concerning the defects responsible for stage III recovery, it seems unlikely that this stage could be due to interstitial migration. Although the migration of interstitials with subsequent annihilation at vacancies could explain the large electrical resistance⁽⁷⁾ and lattice parameter⁽⁸⁾ recoveries, those interstitials which are not annihilated should add on to or form more clusters, not less as observed.

The migration of vacancies or divacancies could explain resistance and unit cell recovery in stage III and those vacancies not annihilated at trapped interstitials could react with interstitial clusters to reduce the cluster size below the resolution limit of the microscope. Such a mechanism has also been indicated by previous studies on this material.^(7,8)

The apparent increase in clusters at stage IVA could be due to the formation (or appearance) of either interstitial or vacancy clusters. Since this recovery peak also corresponds to a very small electrical resistance and a moderate lattice parameter recovery, one cannot rule out the release of interstitials from traps. An alternate mechanism would be the migration of vacancies to annihilate the remaining trapped interstitials and the coalescence of those unreacted vacancies into defect clusters. This mechanism would require that another type of vacancy migrate in stage III, possibly a divacancy. As pointed out previously,⁽⁷⁾ divacancy migration in stage III is favored by the high

pre-exponential factor for migration and as a more plausible explanation for the apparent negative activation volume of migration.⁽⁷⁾

The fact that the final defect cluster recovery peak corresponds to that of the final lattice parameter recovery (Figure 9) indicates that the interstitial defect clusters contribute a small but measurable amount to the lattice parameter increase. It is also observed that the electrical resistance increase is completely recovered while at least half of the defect clusters are still present. This implies that the interstitial clusters contribute very little, if anything, to the electrical resistance increase.

The mechanism for defect cluster annealing is not clear at this time, but Barnes⁽¹¹⁾ has proposed that the defect clusters themselves can migrate and grow if they meet a cluster of like defects or be annihilated if they meet a cluster of unlike defects. Since the annealing in this experiment takes place without significant cluster growth, annealing by Barnes' mechanism would require the presence of vacancy clusters. While they cannot be identified in the present study due to the small defect size (25-35Å), previous studies^(5,6) have shown that clusters do exist in Mo under the proper conditions. If our interpretation of stage IVA is correct, then the formation of vacancy clusters could explain the increase in defect cluster density, while at the same time could provide the mechanism for interstitial cluster annealing in stage V.

The recovery in stage IVB, which is only detected by electrical resistance measurements, is probably due to small vacancy cluster annealing, because the larger clusters do not significantly affect the electrical resistance (stage V). The vacancies released from the small

clusters could also account for the apparent reduction in observable clusters as indicated by the broad recovery band around stage IVB.

HIGH PRESSURE ANNEALS

The dramatic decrease in cluster density at the stage III recovery peak under high pressure also corresponds to large electrical resistance and lattice parameter recoveries.^(7,8) This indicates that similar to the low pressure anneals, the defect (or defects) moving in stage III not only interacts with other point defects but with the clusters as well. Again, this indicates that interstitials are not mobile during the third stage of recovery, and the most probable defects are divacancies or vacancies.

The cluster density annealing still has three recovery peaks at 20 kbars but in this latter case, the lattice parameter is completely recovered before the final defect clusters are annealed out. Since the electron microscopy was done on the same samples used for lattice parameter determinations, there is little chance that sample inconsistencies account for the difference. A possible explanation for their behavior could lie in the geometric configuration of the clustered defects. If the clusters were converted from three dimensional arrays to platelets by the applied pressure, one might expect the contribution to the lattice parameter to be minimized. Such a hypothesis is difficult to check because of the small defect size.

The close correlation between the finite electrical resistance, lattice parameter and the cluster recovery at $\sim 350^{\circ}\text{C}$ is very similar to that observed at low pressure during stage IVA. However, it cannot

be stated with certainty that the second recovery peak observed under pressure is indeed stage IVA because the pressure coefficient of IVB is unknown. Further work at lower pressures should be conducted to determine the initial direction of the IVA-IVB shifts.

Finally and probably most important of all is the large negative shift of the final cluster annealing temperature with pressure. If the pressure coefficient were linear over the 20 kbar region, the shift of the final annealing temperature is $-15^{\circ}\text{C}/\text{kbar}$. The mechanism by which this pressure induced annealing occurs is not known, just as the mechanism of low pressure annealing is unresolved. Some possibilities are:

- 1) Pressure induced migration of point defects which annihilate at the clusters.
- 2) Negative pressure coefficient of the binding energy per defect in a cluster.
- 3) Cluster migration and annihilation with cluster of the opposite type.

Assuming the clusters to be primarily interstitial, the first mechanism would mean that pressure would assist the migration of a vacancy through the metal. However, practically all of the high pressure diffusion studies have shown that just the opposite is correct. Emrick⁽¹²⁾ has actually measured the activation volume for migration of a vacancy in Au. He found that it is $+0.15 V^*$ where V^* is the atomic volume. Since the activation volume is positive, the migration of vacancies should be retarded with pressure, although it is possible that bcc metals present a special case.

The second possibility is a difficult one to assess because of the lack of information, both experimentally and theoretically. At the present time, there is no adequate theory which predicts how tightly an interstitial will be bound in a defect cluster at low pressure, let alone at high pressures. Since this is a complex problem, a computer approach would seem worthwhile. Qualitatively, one might expect that the binding energy per interstitial is reduced as the pressure is increased, thus requiring less thermal energy for the clusters to break up.

The third possibility was originally proposed by Barnes⁽¹¹⁾ to account for the 1 bar annealing behavior of irradiated material. He showed that there will be an attraction between like-like and between like-unlike defect clusters, and that the resultant coalescence will produce loop growth or annihilation, respectively. Since the loops (and we will now assume that the clusters we see are really interstitial or vacancy loops) could coalesce by either climb or glide one must examine the effect of pressure on both processes.

It has been shown⁽¹³⁾ that the annealing of prismatic vacancy loops in Al is retarded under pressure and this was interpreted as evidence that pressure reduces the pipe diffusion around the clusters. Because climb is very sensitive to pipe diffusion, one might then expect that it would be slower under pressure than at 1 atmosphere.

Slip is a sensitive function of the critical resolved shear stress and since Bridgman⁽¹⁴⁾ has shown that the shear strength of Mo increases with pressure, one would expect that slip would be retarded with pressure. A factor which has been neglected is the effect of pressure

on the strain energy characteristic of an interstitial loop in a compressed lattice. This effect could counteract the effect of pressure on slip and climb and may actually induce the loops (or clusters) to anneal.

In summary, the defect cluster density does appear to show fine structure in both low and high pressure anneals. The cluster density is reduced at stage III indicating the migration of a vacancy type defect, is increased at stage IVA, and shows a final recovery over stages IVB and V. Such behavior would be consistent, but not necessarily proof of, divacancy migration in stage III, vacancy migration in stage IVA, small vacancy cluster break up in stage IVB, and interstitial (and possibly large vacancy) cluster annealing in stage V.

CONCLUSIONS

At pressures ≤ 1 bar:

- Irradiation at 40°C to $6.2 \times 10^{18} \text{ n/cm}^2$ ($E > 1 \text{ MeV}$) produces 3.7×10^{16} defect clusters/cc.
- The defect cluster density shows a reduction at stage III, an increase at stage IVA (400°C), and final recovery at stage V (750°C).
- Observable defect clusters contribute a small but measurable amount to the lattice parameter increase.
- Observable defect clusters contribute very little to the electrical resistance increase.
- Stage V recovery in Mo is due to the final annealing of observable defect clusters.
- The cluster size is between $25\text{-}35\text{\AA}$ and does not vary significantly up to the temperature of final annealing.
- Final recovery is complete at 800°C .

After annealing at 20 kbars hydrostatic pressure:

- The observable defect cluster density increases during room temperature compression.

The cluster density shows a recovery stage at 90°C in accordance

- with previous electrical resistance and lattice parameter measurements.

The cluster density increases at 300°C and then is completely

- recovered at 490°C, more than 300°C lower than in the case of annealing in vacuum.

- The average defect cluster diameter is not significantly altered over the range of temperatures investigated.

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A

1000 Å

B

1000 Å

FIGURE 1
Representative Transmission Micrographs of Irradiated
Molybdenum a) As-Irradiated b) After a 2 Hour 20 kbar
Anneal at 25 °C

A

1000 Å

B

1000 Å

FIGURE 2
Representative Transmission Micrographs of Irradiated
Molybdenum Annealed for 2 Hours at 300 °C a) in Vacuum and
b) Under 20 kbars Pressure

A 1000 Å

B 1000 Å

FIGURE 3
 Representative Transmission Micrographs of Irradiated
 Molybdenum Annealed for 2 Hours at 400 °C a) in Vacuum and
 b) Under 20 kbars Pressure

A

1000 Å

Edge of
Foil

B

1000 Å

FIGURE 4

Representative Transmission Micrographs of Irradiated Molybdenum Annealed for 2 Hours at 500 °C a) in Vacuum, and b) Under 20 kbars Pressure. Note the Absence of any Clusters in the Interference Fringes of b)

A

1000 Å

B

1000 Å

FIGURE 5
 Representative Transmission Micrographs of Irradiated
 Molybdenum Which was Vacuum Annealed for 2 Hours at a) 700 °C
 and b) 800 °C

FIGURE 6
Semi-Log Plot of the Effect of Pressure on the Annealing of
Defect Clusters in Neutron Irradiated Molybdenum
2 Hours at Each Temperature

FIGURE 7
Linear Plot of the Effect of Pressure on the Annealing of
Defect Clusters in Neutron Irradiated Molybdenum
2 Hours at Each Temperature

FIGURE 8
Reduced Plot of the Effect of Pressure on the Annealing of
Defect Clusters in Neutron Irradiated Molybdenum
2 Hours at Each Temperature

FIGURE 9

Comparison of the Differential Annealing Rates for the Recovery of Electrical Resistance Increase, Lattice Parameter Increase and Defect Cluster Density under Various Pressures;

a) ≤ 1 Bar b) 20,000 Bars

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