Effects of 14 MeV Neutron Irradiation on Mechanical Properties of High-Purity Molybdenum Single Crystals

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High-purity molybdenum single crystals with the antitwinning axial orientation were irradiated with 14 MeV neutrons at room temperature, 473 K and 673 K under low neutron fluences of $(2.1 \sim 2.7) \times 10^{21} \text{ n/m}^2$ using the Rotating Target Neutron Sources (RTNS-II), and then were deformed in tension at 290 K. The irradiations led to the inhomogeneous yielding accompanied by large yield drops. The active slip plane, which of the unirradiated specimen was the plane (101), was shifted more and more towards the maximum shear stress plane (211) with decreasing irradiation temperature. By the isochronal annealing, the pronounced yield drop disappeared above 973 K. Furthermore, the flow stress of the specimens irradiated at room temperature changed from softening to hardening near 500 K and the strain-rate sensitivity also responded to this change, which was related to the migration of vacancies. Especially for the specimens irradiated at 673 K, it was found that the irradiation effects still remained above 973 K.

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I. Introduction

In the development of materials for fusion reactors, it is necessary that the fusion neutron damage in materials is more exactly understood by the accumulation of data and the disclosure of new fundamental aspects. At present, experimental data on fusion neutron irradiation have been confined to those at low fluences obtained from 14 MeV neutron irradiation at the Rotating Target Neutron Source (RTNS-II) which is currently the strongest source of D-T neutrons. In recent years, however, studies on the fusion-fission damage correlation have been actively developed, including the fusion damage prediction from experimental and theoretical aspects⁽¹⁾. Particularly, the fusion-fission correlation to the mechanical properties or the comparison between their annealing characteristics, in which we are mostly interested, have been extensively discussed in high-purity irons and iron alloys (including low alloy steels) from more fundamental aspects utilizing irradiations at relatively low temperatures⁽²⁾⁻⁽⁶⁾. However, in molybdenum which is one of the refractory materials and especially one of the attractive materials for the first wall and protection components of fusion reactors, such a discussion has not been sufficiently made so far because data on the fusion neutron irradiation was extremely less and most of the results of fission neutron irradiations have been obtained from impure samples irradiated at temperatures higher than 773 K⁽⁷⁾⁻⁽⁹⁾.

Changes in mechanical properties and the annealing behaviour of irradiated materials depending on neutron

energy are closely related to the damage characteristics through the interaction of the radiation-induced defects with moving dislocations controlling plastic deformation. Plastic deformation may be regarded as a useful tool for judging defect properties because the movement of dislocation sensitively responds to the type and number density of point defects with large misfit strains⁽¹⁰⁾. However, the plastic behavior of BCC metals such as iron and molybdenum is significantly influenced by the existence of a small amount of interstitial type impurities such as carbon, nitrogen and oxygen because screw dislocations controlling the plastic deformation are strongly interacted with them. Furthermore, the existence of these impurities drastically changes the properties of defects formed at given irradiation temperatures through the defect-impurity interaction and thereby the mechanical properties. In order to extract the intrinsic properties of radiation damage from neutron irradiated materials, it is very important to use high-purity metals.

In this paper, before practical investigation on irradiation problems of molybdenum or molybdenum alloys in a fusion reactor mentioned above, the plastic behavior of high-purity molybdenum single crystals, irradiated with 14 MeV fusion neutrons at relatively low fluences at lower irradiation temperatures, is experimentally disclosed and is discussed from a fundamental viewpoint on the basis of the intrinsic properties of defects. The use of single crystals simplifies the phenomenon of plastic deformation and makes the irradiation effects more prominent.

II. Experimental

1. Materials

The starting material used for producing high-purity molybdenum single crystals was pure molybdenum supplied by Johnson Matthey Co. These molybdenum rods of 5 mm in diameter and 150 mm long are reported to contain about 150 mass ppm metallic impurities as listed in Table 1. Important information about gaseous impurities, which strongly influence mechanical properties of BCC metals, is not given by the supplier. In order to remove gaseous impurities, therefore, further purification was carried out using an electron-beam floating-zone apparatus; one outgassing pass just below the melting point in a vacuum of 10^{-4} Pa and five zone-melting passes under a low-hydrogen pressure of 5×10^{-3} Pa. Finally, a purified single crystal of about 10 mm in diameter and about 100 mm long was produced.

Tensile specimens with an axial orientation shown in Fig. 1 were prepared by cutting the single crystal rod and electropolishing the cut rectangular bars to put them into their final form with shoulders. The selection of this crystal orientation is based on the prevention of the occurrence of the unexpected slip phenomena, i.e., the multiple slip⁽¹¹⁾ and the anomalous slip⁽¹²⁾⁻⁽¹⁵⁾ which have been recognized widely in the group V and VI metals including molybdenum. The antitwinning orientation, $\lambda=48^\circ$ and $\chi=30^\circ$, is known to give the single slip system $(\bar{1}01)$ $[111]$ for the deformation below room temperature and more correctly reflect the irradiation effects through the interaction of dislocations with radiation-induced defects, where λ is the angle between the tensile axis and the primary slip direction $[111]$ and χ is the angle between the maximum shear stress plane and the plane $(\bar{1}01)$. The final dimensions of the specimens were about 0.2 mm in square cross-section and 5 mm long for the deformed part and were about 0.35 mm in square cross section and 3 mm long for the shoulders. The details of the specimen preparation mentioned above have been reported somewhere⁽¹⁶⁾.

The residual resistivity ratio of these specimens was in the range of 5000–6000. This level of residual resistivity ratio is considered to be mainly due to tungsten impurity with a lower vapor pressure and a higher melting point than molybdenum. However, it is surmised that the floating-zone melting under low pressure hydrogen leads to the reduction of gaseous impurities such as carbon, nitrogen and oxygen and metallic impurities with lower melting points than that of molybdenum because the present molybdenum single crystals are easily deformed at 4.2 K⁽¹⁶⁾⁽¹⁷⁾.

Table 1 Impurity concentration of molybdenum given by the supplier (mass ppm)

W	Cr	Sn	Mg	Si	Mn	Al	Ca	Fe	Cu	Ni
<50	30	50	1	—	2	1	1	20	5	—

Fig. 1 Stereographic projection of crystal orientation of molybdenum single crystals.

2. Irradiation and tensile tests

Irradiation was performed with 14 MeV fusion neutrons at the RTNS-II under conditions shown in Table 2. The present irradiation was designed to be carried out under similar neutron flux and fluence at three different irradiation temperatures. That is, tensile specimens, packed in a Al foil, were set on the inner wall of the irradiation chamber and were irradiated at a given temperature controlled by heating the irradiation chamber. The variations of flux and temperature along the specimens during irradiations was predicted to be small enough to be negligible to the investigation of irradiation effects on the mechanical properties. Actually, the tensile tests of three specimens irradiated under a given irradiation condition indicated the nearly similar plastic behavior.

Tensile test was carried out at 290 K at a nominal strain-rate of 1.7×10^{-4} /s. Irradiation effects of molybdenum were investigated mainly through the irradiation temperature dependence of slip system and

Table 2 Conditions of neutron irradiation

Irradiation temperature (K)	Fluence (n/m^2)	Flux ($\text{n}/\text{m}^2/\text{s}$)
RT	2.7×10^{21}	2.5×10^{15}
473	2.4×10^{21}	2.1×10^{15}
673	2.1×10^{21}	1.9×10^{15}

the post-irradiation isochronal annealing experiments including thermal activation analysis. The annealing experiments were conducted by annealing the irradiated specimens in a vacuum below 7×10^{-5} Pa under conditions of the increment of annealing temperature $\Delta T = 50$ K and annealing time $\Delta t = 20$ min and by deforming the annealed specimens at 290 K. In thermal activation analysis, activation area A^* , through which dislocations sweep as they thermally overcome obstacles, is evaluated by the strain-rate sensitivity $\Delta \tau / \Delta \ln(\dot{\gamma})$ (or simply $\Delta \tau$) obtained from the strain-rate change tests ($\dot{\gamma}_1 \rightarrow \dot{\gamma}_2$) and is given by the following equation⁽¹⁸⁾:

$$A^* = (1/b)kT \cdot \ln(\dot{\gamma}_2/\dot{\gamma}_1) / \Delta \tau, \quad (1)$$

where b is the Burgers vector of molybdenum, k is the Boltzmann constant and $\Delta \tau$ is the increment of shear stress after strain-rate change ($\dot{\gamma}_2/\dot{\gamma}_1 = 10$).

III. Experimental Results

1. Stress-strain curve

Figure 2 shows the feature of typical stress-strain curves for high-purity molybdenum single crystals irradiated at RT, 473 and 673 K together with that for an unirradiated single crystals, where three specimens were actually used for tensile test in all cases, respectively. In the deformation at 290 K, the unirradiated specimen exhibited a rounded yield point and parabolic work-hardening. The total elongation was within 15–18%. On the other hand, neutron irradiations caused a large yield drop, followed by inhomogeneous deformation due to the propagation of Lüders fronts. The magnitude of the lower yield point increased with increasing irradiation temperature. However, the behavior of the upper yield point was not simple. That is, the value for the specimen irradiated at 673 K was small than that of the specimen irradiated 473 K. The lower yield point was near 3%

plastic strain for all the irradiation temperatures. The work-hardening rate was positive for the irradiations at room temperature and 473 K, but at 673 K it was negative. The elongation was slightly enhanced by the irradiation at room temperature (17–20%), while the increase in irradiation temperature led to the decreasing trend (11–14%). At $T_{\text{irr}} = 473$ and 673 K, furthermore, the occurrence of serrated yielding was more clearly observed. This is associated with the intermittent formation of localized slip bands accompanied by the propagation of Lüders fronts. From the observation of the slip band formation mentioned in next section, we understand that the plastic deformation of the specimens containing structural defects such as radiation-induced defects is very difficult, in particular in the early stage. Both the phenomena of the enhancement of elongation and the serrated yielding have also been found in RTNS-II irradiated high-purity iron⁽⁶⁾. An interesting result was the ductile fracture with 100% reduction of cross section for all the irradiated specimens as well as the unirradiated one.

The annealing effect on yielding of the specimens irradiated at room temperature is shown in Fig. 3. The details of annealing conditions are shown in previous section. The general features of the annealing effect were that the amount of yield drop gradually decreased with increasing annealing temperature (from about 95 MPa for the as-irradiated specimen consequently to 4 MPa at annealing temperature $T_{\text{ann}} = 973$ K) and that the lower yield point has a maximum magnitude within the range from 673 and 873 K. In details, it was characterized that the serrated yielding was observed at $T_{\text{ann}} = 473$ and 673 K, which was similar to the behavior at specimens irradiated at $T_{\text{irr}} = 473$ and 673 K, and that the sharper yielding tended to disappear above 873 K. The specimens irradiated at 473 K also showed a similar annealing behavior to that of the specimen irradiated at room temperature.

Fig. 2 Effect of 14 MeV neutron irradiation on the load-elongation curves at 290 K in molybdenum single crystals. The irradiation temperatures are room temperature, 473 K and 673 K.

Fig. 3 Annealing effects on the initial stage of the load-elongation curves at 290 K in molybdenum single crystals irradiated with 14 MeV neutrons at room temperature.

2. Morphology of slip bands

Figure 4 shows the development of slip bands formed during yielding for the specimen irradiated at room temperature. At the plastic strain $\epsilon_p=0.6\%$, just after the upper yield point (see Fig. 2), the coarse slip bands began to form near the grip ends and propagated towards the center of the specimen. On the top surface (T), the slip line was wavier, while on the side surface it was straight. As illustrated in the figure, this slip morphology is closely related to the relationship between the slip direction $[111]$ of screw dislocations which control the low-temperature deformation of molybdenum and the surface orientation. Although screw dislocations in the unirradiated specimen can slip on the equivalent elementary planes

($\bar{1}01$) and ($\bar{1}10$)⁽¹⁵⁾⁽¹⁶⁾ and the very fine and diffusive slip lines were traced on the unirradiated specimen surface, the observation of slip lines for the irradiated specimens show that the maximum shear stress plane is ($\bar{2}11$) as the mean slip plane. This indicates that the irradiation induces the cross-slip of screw dislocation between the ($\bar{1}01$) and ($\bar{1}10$) planes. Therefore, on the top surface, where the slip direction intersects with a large angle, the wavy slip traces due to the cross-slip were observed. At both the front of slip bands, however, the slip lines of the ($\bar{1}01$) plane were still recognized.

The propagation of the slip bands did not complete at $\varepsilon_p=1.3\%$. Above 3% the slip bands covered on the whole surface and the steady state deformation began. As shown in Fig. 4(c), the $(\bar{2}11)$ slip became more domi-

Fig. 4 Slip patterns on the surfaces of the specimens irradiated at room temperature. (T) and (S) denote the top and side surfaces, respectively. The development of slip band in the early stage of deformation is shown at (a) $\epsilon_p = 0.6\%$, (b) 1.3% and (c) 3.3% . The active slip planes are obtained by the two-face analysis. Furthermore, the slip planes are geometrically analyzed in connection with the tensile axis, surface orientations and the direction of a screw dislocation.

Fig. 5 Slip patterns on the surfaces of the specimens irradiated at room temperature, 473 and 673 K. The amount of deformation at 290 K is about 3% in every case. The mean slip planes are denoted with ψ .

nant as a result of the frequent occurrence of cross-slip. The density of slip bands more and more increased in the work-hardening region until the necking began to occur. It appears that the secondary slip system does not operate.

The formation of slip bands for the 473 K irradiated specimens was characterized by finer slip lines than those for the R.T. irradiated specimens and the slip planes near the $(\bar{2}11)$ plane. On the other hand, the slip morphology for the specimen irradiated at 673 K indicated that the slip traces became diffusive and the slip occurred between the $(\bar{2}11)$ and the $(\bar{1}01)$ planes. Figure 5 shows the slip morphology near 3% plastic strain depending on irradiation temperature, where ψ in the figure expresses the observed slip plane and is defined by the angle to the $(\bar{1}01)$ plane. $\bar{\psi}$ denotes the mean slip plane. In every case the slip bands covered the whole specimen above 3% plastic strain. The annealing effects on active slip planes were examined only for the specimens irradiated at room temperature, as shown in Table 3. It is found that the slip plane largely recovers towards the $(\bar{1}01)$ plane at tempera-

Table 3 Active slip planes after annealing the specimens irradiated at room temperature.

T_{ann} (K)	R.T.	473	673	873	≥ 973
$\bar{\psi}$	30°	27°	15°	8°	0°

tures between 473 and 673 K.

3. Post-irradiation isochronal annealing

Figure 6 shows the results of the the post-irradiation annealing experiments in high-purity molybdenum single crystals irradiated with the similar neutron fluence level, $2 \times 10^{21} \text{ n/m}^2$ at room temperature, 473 and 673 K. The annealing behaviour was examined from the viewpoint of the change in the mechanical properties, i.e., the flow stress τ_f and the strain-rate sensitivity $\Delta\tau$ (or the activation area A^*). The analysis of annealing data is fairly complicated as follows. First of all, every shear stress in the figure, except those above 973 K, is resolved on the slip planes ($\bar{\psi}$ in the figure) observed at room temperature, although the slip planes, for example those for the room temperature irradiation actually varied with annealing as measured at some annealing temperatures (see Table 3). The criterion of flow stress given here, instead of the yield criterion, is the stress level at 3% plastic tensile strain. As mentioned in previous Sections 1 and 2, the yielding of the irradiated specimens occurred by the inhomogeneous deformation and was accompanied by the large yield drop, in contrast to that of the unirradiated specimens. Therefore, we could not exactly define the conventional yield criterion. The 3% plastic strain mentioned above approximately corresponds to

Fig. 6 Post-irradiation isochronal annealing curves of the flow stress and strain-rate sensitivity (or the activation area) for the 14 MeV neutron irradiated molybdenum. The deformation temperature is 290 K. The close and open symbols denote the results of the virgin specimens and the successive deformation, respectively. The flow stress is resolved on the slip plane (ψ) actually observed in the deformation at room temperature. Above 973 K, however, the (101) plane is used in the all cases.

the ending of the large yield drop for all the irradiated specimens and to the beginning of the homogeneous deformation. Furthermore, a technique of the successive deformation using one tensile specimen was used for making more recovery data because of the confined number of irradiated specimens. The definition of the successive deformation is as follows; the amount of straining after annealing is below 1% in each deformation and the increment of work-hardening during successive deformation is always subtracted from because of the evaluation of the defined flow stress. Although the application of this successive deformation to the annealing experiment should be made with care because of the influence of deformation-induced dislocations on the evolution of radiation-induced defects during annealing, it seems that results of the virgin specimens in Fig. 6 (closed symbols) are in good accord with those obtained from the successive deformation.

The specimens irradiated at room temperature showed the irradiation softening in the annealing temperature range 300–500 K. Above 500 K the flow stress varied to hardening. In response to this variation, the strain-rate sensitivity also varied largely at annealing temperatures 500–550 K. It began to decrease with increasing annealing temperature above about 750 K, which corresponds to the change in the flow stress. It is considered that both, as well as the slip plane, recover above 973 K according to data analyzed with $\bar{\psi}=0^\circ$.

In the 473 K irradiation, the irradiation hardening is recognized through annealing temperature. However, the annealing behavior of the flow stress and strain-rate sensitivity was similar to that of the room temperature irradiation. In any case, the magnitude of hardening will be greatly reduced at higher temperatures in the specimens irradiated at room and 473 K if the variation of slip plane with annealing (to $\bar{\psi}=0^\circ$) is taken into account. On the other hand, the specimens irradiated at 673 K, also characterized by irradiation hardening, showed no annealing effects up to 823 K. This temperature corresponded to the peak regions of the $\tau_f(T_{\text{ann}})$ and $\Delta\tau(T_{\text{ann}})$ curves for the room temperature and 473 K irradiations. However, both of the flow stress and strain-rate sensitivity had a peak near 900 K. The irradiation effects still remained above 973 K. The influence of variation of slip plane on the values of τ_f and $\Delta\tau$ is smaller in this range.

IV. Discussion

The changes in mechanical properties of molybdenum by 14 MeV fusion neutron irradiation were experimentally disclosed using high-purity single crystals. The 14 MeV fusion neutrons very effectively damaged the crystals, although the fluences were low. As shown in Fig. 2, the irradiations did not easily lead to the homogeneous deformation which is observed in the unirradiated high-purity single crystals. The difficulty of the plastic deformation in the early stage, i.e., the occurrence of a large yield drop, results from the low mobility at 290 K of screw dislocations controlling deformation of molybdenum

and the strong resistance of radiation-induced defects against the dislocation movement⁽⁵⁾⁽¹⁰⁾⁽¹⁹⁾. The similar behaviour has been also reported in the high-purity iron single crystals RTNS-II irradiated with almost the same fluence⁽⁶⁾. In this case, the above behaviour occurred below 200 K. At room temperature, however, the irradiated iron single crystals were more ductile than the unirradiated ones.

The interaction of moving screw dislocations with radiation-induced defects formed at different irradiation temperatures caused the change in the slip system of molybdenum. At room temperature and 473 K the active slip plane was nearly the maximum shear stress plane of $(\bar{2}11)$ ($\bar{\psi}=30^\circ$), and at 673 K it was close to the $(\bar{1}01)$ plane ($\bar{\psi}=0^\circ$) which is the slip plane of the unirradiated specimen. This result suggests that the defect structure change in the range of 473–673 K. On the other hand, the isochronal annealing experiments of the flow stress and strain-rate sensitivity for the specimens irradiated at room temperature showed the critical change in the annealing temperature range 500–550 K (see Fig. 6), which has been also reported in the annealing experiments for the same high-purity molybdenum irradiated with fission neutrons at 5 K⁽²⁰⁾. The above critical change is interpreted to result from the migration of vacancies near 500 K. This judgment was given by the results of the positron annihilation lifetime measurements for the same high-purity molybdenum irradiated at room temperature by the same neutron source (2.45×10^{16} n/m²)⁽²¹⁾. On one hand, the migrating vacancies react with the small interstitial loops which are formed at room temperature⁽²²⁾. The small interstitial loops can assist the kink-pair formation of screw dislocations on Peierls barrier as a lattice friction and can cause irradiation softening⁽⁵⁾⁽¹⁰⁾⁽¹⁹⁾. The above reaction consequently leads to the reduction of the loop density and the new growth of interstitial loops towards large size. The former results from the recombination reaction and the left interstitial loops. On the other hand, vacancies are newly able to form vacancy agglomerates. The formation of these growth-up defects with low number density by annealing accounts for the recovery of the slip plane and the change to hardening, respectively.

The recovery behavior of the 673 K irradiation is very complicated. Namely, the peak of hardening appeared near 900 K and the irradiation effects still remains above 1073 K. The former is difficult to be interpreted at present because the fundamental behaviour of the vacancy type defects, which are more dominant at higher temperatures, is not exactly understood. According to the positron annihilation lifetime measurements mentioned above, however, the annealing resistance is interpreted to be related to the stabilization of microvoids formed at higher irradiation temperature due to the nuclear reaction products (He or H) produced by high energy neutrons, i.e. to the formation of microvoid - He (or H) complexes. Although such gas atoms is very small in total

amount at the present neutron fluences, they might be more effectively supplied into microvoids in the single crystals free from grain boundaries. This fact has been also reported in the high-purity iron single crystals irradiated with the same 14 MeV neutron sources⁽⁵⁾.

The authors could discuss the damage correlation between fission and fusion neutron irradiations in high-purity iron by means of plastic deformation and positron annihilation lifetime measurement⁽⁶⁾. Unfortunately, authors obtain no data of fission neutron irradiation under the similar conditions using molybdenum single crystals with an equal purity level at present. The results of Abe⁽²⁰⁾ are based on the very low temperature irradiation and cannot be completely compared with those of the present 14 MeV neutron irradiation at temperatures more than room temperature. Also, the results by other investigators⁽⁷⁾⁽⁸⁾ is not available to the references of the fission-fusion correlation because the differences in purity of molybdenum and irradiation conditions. The authors will be able to discuss the fission-fusion correlation on molybdenum in the near future by carrying out the elaborate program for fission neutron irradiation.

V. Conclusion

(1) The 14 MeV neutrons effectively damaged high-purity molybdenum single crystals even under low neutron fluences of $(2.1 \sim 2.7) \times 10^{21} \text{ n/m}^2$.

(2) In the deformation at 290 K, all the specimens irradiated at room temperature, 473 and 673 K showed the inhomogeneous yielding accompanied by large yield drops.

(3) The slip planes at 290 K shifted more and more towards the maximum shear stress plane ($\bar{2}11$) with decreasing irradiation temperature.

(4) In the post-irradiation annealing experiments, the pronounced yield drop disappeared above 973 K. The specimens irradiated at room temperature showed irradiation softening below 500 K, while with increasing annealing temperature hardening occurred, which corresponded to the behaviour of the strain-rate sensitivity. The critical change near 500 K was explained to be

caused by the migration of vacancies.

(5) In the 673 K irradiation the annealing resistance against irradiation hardening was recognized and was related to the formation of the complexes consisting of a microvoid and He or H gas.

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