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Mechanical energy losses in plastically deformed and electron plus neutron irradiated high purity single crystalline molybdenum at elevated temperatures

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Mechanical spectroscopy (MS) and transmission electron microscopy (TEM) studies have been performed in plastically deformed and electron plus neutron irradiated high purity single crystalline molybdenum, oriented for single slip, in order to study the dislocation dynamics in the temperature range within one third of the melting temperature. A damping peak related to the interaction of dislocation lines with both prismatic loops and tangles of dislocations was found. The peak temperature ranges between 900 and 1050 K, for an oscillating frequency of about 1 Hz.

1 Introduction In advanced fission or fusion reactors is critical the fully understanding of the physical processes which develop during the time of operation. The knowledge of environment-material reactions in terms of fundamental stability and kinetics, including the influences of composition, microstructure and system design are critical to predict the long time performance of nuclear materials [1]. Of particular interest are the drastic, irradiation-induced microstructural evolution and thermophysical property changes occurring as a result of energetic particle irradiation, which significantly impact the performance and lifetime of much of the reactor components [2].

Molybdenum exhibiting a high melting point, a high specific heat, a relatively low thermal neutron cross-section, good corrosion, creep resistance and strength at high temperatures results attractive for the use in the new generation of nuclear power reactors [1–7].

It has been previously reported that single crystalline deformed molybdenum exhibits two damping peaks in the temperature range of about one third of the melting temperature, $0.3\,T_m$ (~865 K). The physical mechanism which controls the damping peak appearing at around 800 K (the so called low temperature peak (LTP)) can be related with the dragging of jogs by the dislocation under movement assisted by vacancy diffusion. The damping peak which appears at higher temperatures of about 1000 K (the so called high temperature peak (HTP)) is controlled by the formation and diffusion of vacancies assisted by the dislocation movement [8].

It was also found for samples oriented for multiple slip ($\langle 110\rangle$ tensile axis) that annealing at temperatures of stage V ($>$ 850–900 K) is required for restoring the characteristic of the LTP after neutron irradiation ($<10^{-5}$ dpa) [8]. Indeed, the damping peak in neutron irradiated samples ($\langle 110\rangle$) is less intense and appears at smaller temperatures (600 K) than in only pre-strained samples (800 K). Subsequent annealing during the mechanical spectroscopy (MS) tests at 973 K did not change too much the peak position of the relaxation in

irradiated samples. However, an annealing at 1073 K displaces the peak position to around 800 K, due to the recovery of the structure in stage V [8, 9]. Moreover, in samples oriented for single slip (⟨149⟩ tensile axis) the LTP was not affected by the neutron irradiation. This behaviour was explained by the smaller quantity of jogs in the ⟨149⟩ samples than in the ⟨110⟩ ones.

It should be mentioned that the temperature range around 0.3 Tm, usually related to the stage V of recovery, is particularly interesting in molybdenum due to the strong influence on the mechanical properties in both, the pure metal and technological molybdenum-based alloys [10–12].

In the present work we have study the interaction processes between dislocations and vacancies in the temperature interval around 0.3 Tm, in samples oriented for single slip, irradiated with electrons and neutrons. The obtained results contribute to the knowledge of the mechanisms which produce mechanical losses in irradiated and cold worked molybdenum, in particular in samples oriented for single slip where the effects of neutron irradiation could not be clearly resolved.

2 Experimental

2.1 Samples The single crystals used in this work were prepared from zone refined single-crystal rods of molybdenum in A.E.R.E., Harwell, England. The high purity molybdenum crystals were prepared from a single batch of 'Amax Specialty Metals Corporation' low carbon 3/8″ diameter rods. The specifications of this starting material were: C 0.004%, O_2 0.0004%, H_2 0.0001%, N_2 0.0001%, Fe 0.002%, Ni 0.001% and Si 0.002%. Samples were prepared from 6-pass zone refined molybdenum, which was decarburized at 1873 K for 48 h and annealed at 2073 K for 3 h in a vacuum better than 10^{-5} Pa. The total final content of (C, O_2, H_2, N_2, Fe, Ni, Si, W) was under 10 at ppm., being the main residual impurity tungsten. The residual resistivity, RR, of the samples was 8000. RR is calculated from the ratio between the electrical resistivity value measured at room temperature and the value of the electrical resistivity measured at liquid helium temperatures. Samples with the ⟨149⟩ crystallographic tensile axis were used to favour deformation only by single slip. The samples were sheets of 20 mm length, 0.2 mm thickness and 2 mm width.

Some samples were also annealed at 1973 K during 24 h and at 2273 K during 3 h, both under high vacuum. These samples will be called hereafter annealed samples. In contrast, samples without this last annealing treatment will be called as-supplied, *i.e.* with some remainder strains introduced during the preparation of the samples.

Samples were deformed in tensile at a constant speed of 0.03 cm/min, followed by 1% torsion at room temperature, see Table 1. Single crystals, after plastic deformation and damping tests, were checked by means of Laue photographs and metallographical studies. Laue and light microscopy results indicated that the single-crystalline state was not changed by the plastic deformation or annealing to the work temperatures.

Electron irradiation process was performed in the J. J. Thomson Physical Laboratory, Reading, England, at 80 K, using 2 MeV electrons, achieving a dose (fluence) of 1.4×10^{16} electron/cm^2 [13–15]. The dose was sufficient to fully pin the dislocations. This was monitored by measuring 'in situ' the values of the damping and modulus with irradiation until then gradually reached a constant saturated value [16].

Neutron irradiations were performed at room temperature, at the Siemens SUR 100 nuclear reactor, RA-4, of the National University of Rosario – National Atomic Energy Commission of Argentina. An estimation of the irradiation dose in dpa (displacement per atom) less than 1×10^{-5} could be done, which corresponds to a vacancy concentration of about 5 ppm (see for details Refs. [8, 17]).

Neutron irradiation was performed after the plastic deformation process and, in some samples, after electron irradiation. In contrast, in electron irradiated samples, the irradiation was carried out before plastic deformation. The characteristics of the samples studied in this work are shown in Table 1.

2.2 Measurements Mechanical spectroscopy (MS), referred to as the internal friction (IF) method in early literature, offers unique opportunities to study the mechanical losses due to the interaction, for instance, between dislocation and point defects produced during neutron or electron irradiation in materials, as molybdenum [17–19]. MS, damping (Q^{-1} or IF) and natural frequency were measured in an inverted torsion pendulum for free-decaying vibrations, under high vacuum of about 10^{-5} Pa; see Ref. [20] for a description of the experimental setup. The maximum strain on the surface of the sample was 5×10^{-5}. The heating and cooling rates employed in the tests were of 1 K/min. A heating and its corresponding cooling run will be called hereafter a thermal cycle. There was no hold time once the maximum temperature had been achieved during the thermal cycle.

Table 1 Status of the used high purity single crystalline samples oriented with tensile axis in the direction ⟨149⟩.

sample	type	elongation (%)	irradiation type	sequence of deformation and irradiation
a	annealed	5	no	only plastically deformed
b	annealed	5	neutron	plastic deformation followed by irradiation
c	as-supplied	5	electron	irradiation followed by plastic deformation
d	annealed	4	electron + neutron	electron irradiation followed by plastic deformation followed by neutron irradiation

For transmission electron microscopy (TEM) examinations, thin foils were prepared with the double jet technique using 12% H_2SO_4 in methyl alcohol. Observations were carried out in a Phillips CM200 transmission electron microscope, operated at 200 kV.

3 Results

3.1 Mechanical spectroscopy We will show firstly the behaviour of the damping spectra during different thermal cycles for a neutron irradiated sample, in order to help with the discussion of the results of the present paper. Figure 1 shows the damping spectra measured for neutron irradiated molybdenum (sample b in Table 1) during successive thermal cycles, by means of empty symbols. During the first heating, the neutron irradiated sample showed an increasing background with temperature. Nevertheless, on cooling a well developed IF peak, at about 800 K, was present (curve A in Fig. 1). After thermal cycles up to 973 K, the peak temperature and peak height are changing a little. The damping spectrum after three thermal cycles up to 973 K is shown in the curve labelled B in the figure. An increase in temperature up to 1223 K, leads to both the shifting in the peak temperature towards higher temperatures and the decrease in the peak height, see curve C in the figure. Also shown in Fig. 1 by means of crosses (curve D) is the damping curve corresponding to the first thermal cycle for an unirradiated sample (sample a in Table 1). The evolution of the damping spectra during different thermal cycles for the unirradiated sample has been already reported in the literature [21, 22], and it results completely similar to the behaviour exhibited by sample b.

Figure 2 shows the behaviour of the electron irradiated sample in the as-supplied state (sample c in Table 1) during different thermal cycles performed up to different maximum temperatures. Similarly to the previously showed case, during the first heating up to 973 K, the electron irradiated sample shows an increasing background with temperature. However, on cooling a well developed IF peak, at about 820 K, is present (curve A in Fig. 2). After thermal cycles up to 973 K, the peak temperature and peak height change slightly, see curve B in Fig. 2. An increase in temperature up to 1073 K, leads to both the shifting in the peak temperature towards higher temperatures and the decrease in the peak height (curve C in Fig. 2). A further increase in the maximum temperature during the thermal cycles up to 1190 K, leads to a next shifting in the peak temperature and to other decrease in the peak intensity (curve D in Fig. 2). After thermal cycles performed up to 1223 K, the damping peak moves again towards higher temperatures of about 1100 K and a decrease in the peak intensity can also be observed, see spectrum E in the figure. Indeed, the peak intensity in the spectrum E decreases even if the damping values are higher than in spectrum D, for temperatures higher than 1000 K, since the background has increased.

It should be pointed out that the electron irradiation plus the subsequent plastic deformation leads to a higher damping peak at around 800 K which is almost two times higher than for unirradiated and neutron irradiated samples (see Fig. 1). In addition, electron irradiated samples during the thermal cycles performed at temperatures higher than 973 K reveal the appearance of two overlapped relaxations on the damping spectra, in contrast to the neutron irradiated samples.

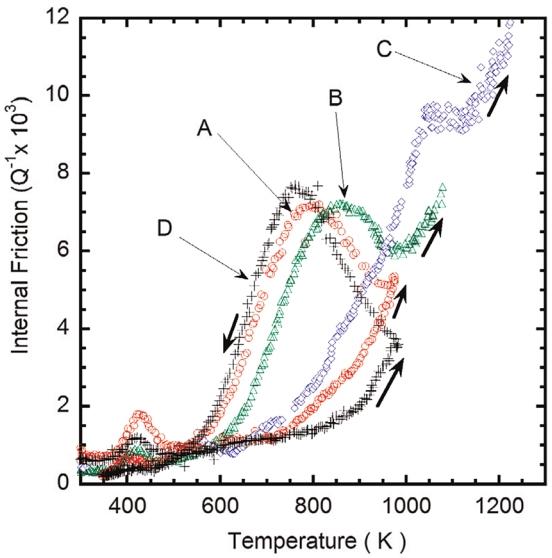

Figure 1 (online colour at: www.pss-a.com) Damping spectra measured for a sample of type (b) during different heating runs. A: after neutron irradiation; B: spectrum after three heating runs up to 973 K; C: spectrum after a heating to 1223 K. Curve D corresponds to an unirradiated sample of type (a) after deformation at room temperature. Arrows indicate the warming and cooling runs.

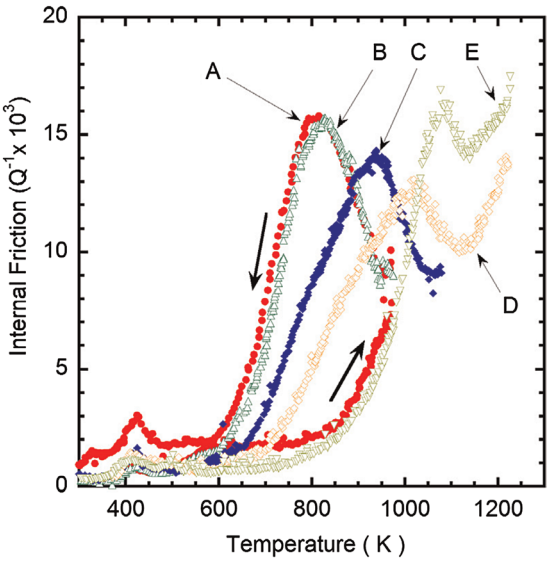

Figure 2 (online colour at: www.pss-a.com) Damping spectra measured for a sample of type (c) during different heating runs. A: after electron irradiation. B: spectrum after heating runs up to 973 K. C: spectrum after a heating run up to 1073 K. D: spectrum after a heating run up to 1190 K. E: spectrum after a heating run up to 1223 K. Arrows indicate the warming and cooling runs.

In fact, in the damping peak of curve B in Fig. 2, there is no sign of the overlapping of two relaxation processes. However, for the curves C and D the overlapping of the relaxation peaks becomes clear. In addition, as the temperature of the thermal cycles is increased to 1223 K, the damping peak becomes more as a single peak, indicating that the overlapping effect is diminished as the temperature is increased, see curve E in Fig. 2.

Figure 3 shows the damping behaviour for an electron plus neutron irradiated sample (sample d in Table 1), during different thermal cycles performed up to different maximum temperatures. The behaviour of the damping spectra during the successive thermal cycles is similar to the previous shown for (c) type samples.

After thermal cycles up to 973 K, the peak temperature and peak height change slightly, see curve B in Fig. 3, similarly as in Fig. 2. However, in the low temperature tail of the peak a small hump can be observed at around 790–800 K. An increase in temperature up to 1073 K during the thermal cycles, allows clearly, the split of the two overlapped relaxations (see curve C in Fig. 3). The relaxation at lower temperatures is the so called LTP. It has its centre at around 800 K, and the higher temperature one, labelled as X in Fig. 3, appears at around 990 K. A further increase in the maximum temperature during the thermal cycle up to 1190 K, leads to a shifting of the relaxation X towards higher temperatures and to a decrease in the intensity of the LTP (curve D in the figure).

Finally, after thermal cycles performed up to 1223 K, the peak X moves again towards higher temperatures of about 1100 K and a decrease in its peak intensity can also be observed. In addition, the LTP has disappeared in these spectra (curve E in Fig. 3).

It is interesting to note that after thermal cycles up to 1223 K, the LTP disappeared and the remainder spectrum is similar to the obtained for the (c) type sample (see curve E in Fig. 2).

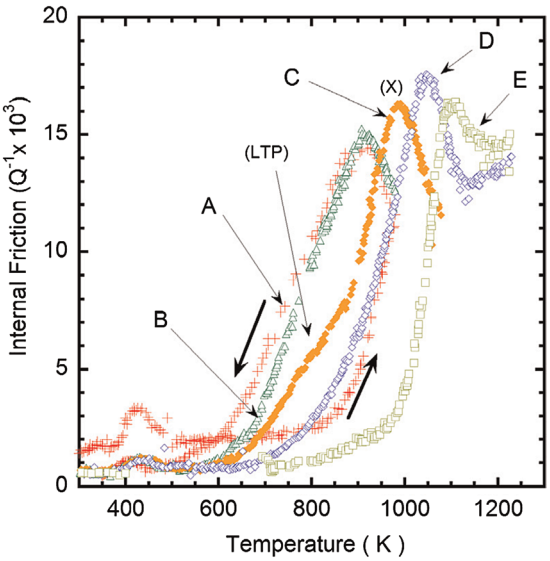

Figure 3 (online colour at: www.pss-a.com) Damping spectra measured for a sample of type (d) during different heating runs. A: after electron irradiation plus neutron irradiation (see Table 1). B: spectrum after heating runs up to 973 K. C: spectrum after a heating run up to 1073 K. D: spectrum after a heating run up to 1190 K. E: spectrum after a heating run up to 1223 K. Arrows indicate the warming and cooling runs. LTP and X, see explanation in the text.

3.2 Transmission electron microscopy The dislocation arrangements in deformed and neutron irradiated single crystalline ⟨149⟩ and ⟨110⟩ molybdenum was reported in Ref. [8]. An analysis of constructive interference, related the (110) plane as the sliding one and the Burgers vectors with the ⟨111⟩ direction. In addition, the dislocation density, determined by counting the dislocation lines, was larger in ⟨110⟩ than in ⟨149⟩ samples, in agreement with the multiple slip condition for ⟨110⟩ samples. Dislocations in samples with ⟨110⟩ orientation were shorter and present higher density of jogs than for the ⟨149⟩ orientation, which results in agreement with pervious reported works [23–27].

We have performed new TEM examinations in the neutron and electron irradiated samples prior to the starting of the MS measurements and after the successive thermal cycles with the aim of comparing the dislocation arrangements we are dealing with in the present work.

Figure 4 shows a micrograph for the ⟨149⟩ plastically deformed plus neutron irradiated sample previous to the MS test (sample b in Table 1). The dislocations in sample (b) are rather straight. Dislocations loops promoted by neutron irradiation cannot be detected. In addition, the microstructure after thermal cycles exhibited a decrease in the dislocation density due to the structure recovery in agreement to already reported results [8].

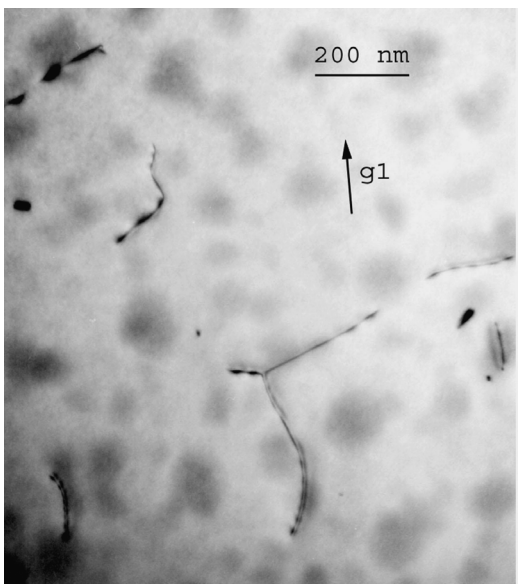

Figure 4 TEM micrograph for a plastically deformed plus neutron irradiated sample (b type).

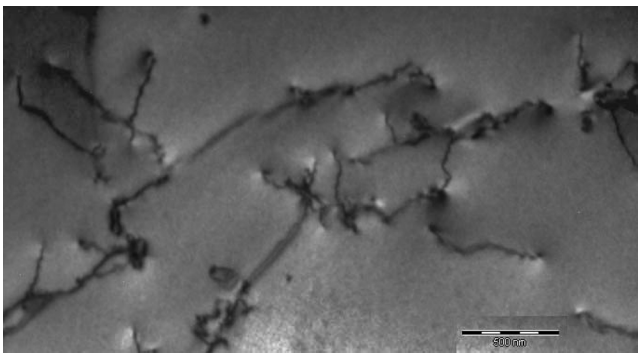

Figure 5 TEM micrograph for an electron irradiated sample of (c) type.

Figure 5 shows a micrograph for a sample of type (c), see Table 1. The resulting dislocation arrangement exhibits rather straight dislocations, but they present both, small loops and tangles of dislocations. Loops and tangles appear subsidiaries to the dislocations lines. The dislocation arrangement for a (d) sample (electron plus neutron irradiated, see Table 1), resulted similar to that exhibited for sample (c) (electron irradiated) in Fig. 5.

After thermal cycles to 1223 K, TEM examinations reveal that the quantity of loops and tangles of dislocations in samples (c) and (d) has decreased markedly, obtaining a structure more alike the one shown for samples (b) after the same thermal cycles.

4 Discussion It has been reported before [8] that single crystalline deformed molybdenum exhibits two relaxation peaks in the temperature range of around $0.3\,T_m$. The physical mechanism which controls the damping peak appearing at 800 K (the so called LTP) was related with the dragging of jogs by the dislocation under movement assisted by vacancy diffusion. Meanwhile the damping peak which appears at higher temperatures of about 1000 K (the so called HTP) was related with the formation and diffusion of vacancies assisted by the dislocation movement [8]. The activation energy (H) and pre-exponential factor (τ_0) of the relaxation time, for the LTP and HTP were: $H \approx 1.6$ eV, $\tau_0 \approx 10^{-11}$ s and $H \approx 2.7$ eV, $\tau_0 \approx 10^{-14}$ s, respectively [8, 22].

In the present work the damping spectra in (c) and (d) samples are similar and they are twice times higher than for unirradiated, (a), and only neutron irradiated, (b), samples (see Figs. 1–3). After annealing during the thermal cycles at temperatures of 1073 K, the damping spectrum of samples (c) and (d) stars to split in two components, see curves C in Figs. 2 and 3.

The damping spectra of electron, (c), and electron plus neutron irradiated, (d), samples, exhibit a new relaxation peak on the high temperature side of the LTP, see relaxation marked X in Fig. 3. This relaxation is also present as a component of the D run in Fig. 2. The activation energy and τ_0 of the relaxation time for the X peak, calculated from the shift of the peak temperature with the change in the oscillating frequency [18], result around (1.9 ± 0.5) eV and $1 \times 10^{(-11.0 \pm 0.5)}$ s, respectively. Consequently, considering the followings experimental facts: (i) the activation parameters and peak temperature of this relaxation, (ii) the damping values in electron irradiated samples, which are twice higher than for unirradiated and neutron irradiated samples, (iii) the TEM examinations which reveal that the electron irradiation followed by plastic deformation in tensile plus torsion have promoted the development of prismatic loops and tangles of dislocation subsidiaries to the dislocations lines, – we propose that this new relaxation is controlled by the overlay of the following interaction processes: (I) dislocation–prismatic loops and (II) dislocation–tangles of dislocations. As this relaxation appears at an intermediate temperature between the LTP and HTP peaks of the molybdenum, it will be called hereafter intermediate temperature peak (ITP) of the molybdenum, ITP.

Then, during the dislocations movement which gives rise to the LTP, they drag prismatic loops and tangles of dislocations, which were created as a consequence of the electron irradiation plus the plastic deformation. This leads to an increase in the frictional dissipation during the dislocations movement giving rise to this intense new peak, at higher temperatures than the LTP. The physical mechanism for the ITP will have an activation energy and relaxation time very close to the values of the LTP. A higher value of H for the ITP than for the LTP is reasonable since the system under movement is more complex for the ITP peak. Regarding to the τ_0, it results within the usual bandwidth for relaxation time involving dislocations mechanisms (10^{-10} s $< \tau_0 < 10^{-14}$ s) [18].

In contrast, the HTP in molybdenum has different activation parameters ($H \approx 2.7$ eV and $\tau_0 \approx 10^{-14}$ s) where the pre-exponential factor is clearly indicating another kind of physical mechanism [8].

It is important to mention here that, for samples (c) and (d) the damping peak at highest temperature (curves E in Figs. 2 and 3), which are obtained after thermal cycles up to 1223 K, are described by means of an activation energy of (2.9 ± 0.3) eV and a pre-exponential factor of around $1 \times 10^{(-14.0 \pm 0.5)}$ s. Therefore, this remainder peak after thermal cycles up to the highest temperature is the HTP.

On the other side, the behaviour of the damping spectra during the thermal cycles results in agreement with the proposed physical mechanism controlling the ITP. In fact, after an annealing during the thermal cycles at temperatures of 1073 K, the damping spectrum stars to split in two components, see curves C in Figs. 2 and 3. Moreover, for the (d) type of samples it can be clearly seen that the damping spectrum is composed for more than one elementary peak, see curve C in Fig. 3. A temperature of 1073 K was already reported for the first recovery of the internal stresses promoted by the neutron irradiation process [8, 9]. Then, above 1073 K the dislocations gain enough mobility and at the same time the loops and tangles star a process of recovery

and annihilation, assisted by vacancy diffusion. It leads to both, small loops and tangles of dislocations with a higher pinning strength, giving rise to the shifting of the peak temperature of the ITP peak towards higher temperatures. A further increase in the annealing temperature during the thermal cycles gives rise to even stronger pinning points and then the peak temperature is increased again, see Figs. 2 and 3.

After annealing at 1223 K, during the thermal cycles, the quantity and size of loops and tangles interacting with the dislocations decrease markedly as indicated by the strong decrease in the peak height of the ITP, see curves E in Figs. 2 and 3. This behaviour in the damping spectra is in agreement with TEM results. It corroborates that the dislocations arrangements after annealing to 1223 K exhibit a markedly decrease in the quantity of loops and tangles of dislocation and also in their size, resulting in an arrangement more alike the one showed in Fig. 4 for neutron irradiated sample.

5 Conclusions A new damping peak related to the interaction of dislocations lines with both prismatic loops and tangles of dislocation was described in electron irradiated and plastically deformed samples, oriented for single slip. The peak temperature ranges between 900 and 1050 K, for an oscillating frequency of about 1 Hz, and then it is called intermediate temperature peak (ITP) of molybdenum. The calculated activation energy for the ITP was around (1.9 ± 0.5) eV.

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