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Hardness and electrical resistivity of molybdenum in the post-irradiated and annealed conditions

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Abstract

Hardness and electrical resistivity were measured for LCAC molybdenum following irradiation at 270–1100 °C and neutron fluences of $10.5–64.4 \times 10^{24}\,\mathrm{n/m^2}$ ($E > 0.1$ MeV) to provide a basic assessment of the influence of irradiation and post-irradiation annealing on recovery. Hardness was a more sensitive measure of defect density than electrical resistivity. Irradiation at 935–1100 °C resulted in little hardening and therefore no recovery was observed. Recovery of the radiation hardening for molybdenum irradiated at 270–605 °C was observed to begin at Stage V recovery temperatures (≈600 °C) and was completed at 980 °C and 1100 °C, respectively. Isothermal annealing was performed at 700 °C and Meechan–Brinkman analysis indicated that the activation energy for recovery was 4.07–4.88 eV ± 0.83 eV, which is comparable to values for molybdenum self-diffusion. These results indicate that the recovery of molybdenum irradiated at temperatures $\leqslant$605 °C occurs by the solid-state diffusion of vacancies to coarsen the dislocation loops and voids.

1. Introduction

Molybdenum is a refractory metal with excellent strength and creep-resistance at high temperatures making it of interest for many nuclear and other energy system applications [1–5]. Low carbon arc cast (LCAC) molybdenum is a wrought unalloyed molybdenum form that possesses a high degree of tensile ductility at room-temperature when the grain size is fine, the carbon content is below 100 ppm, and the carbon to oxygen ratio is high [6–10]. Neutron irradiation of molybdenum at temperatures <700 °C can result in radiation embrittlement that is typically characterized by an increase in the ductile to brittle transition temperature (DBTT), as determined by either tensile testing or bend testing, from below room-temperature to values between 600 °C and 700 °C following irradiation [1–5]. The radiation embrittlement of molybdenum results from the formation of a high number density of finely spaced point defect clusters, dislocation loops, and/or voids, which limit the homogeneous movement of dislocations and is the result of the slow self-diffusion rate of point defects limiting defect annihilation at irradiation temperatures <700 °C. The greater mobility of point defects at higher temperatures results in diffusion of point defects to sinks at a higher rate and generally produces a lower number density of coarse voids and little hardening and

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embrittlement following irradiation of molybdenum at temperatures >800 °C.

The embrittlement of molybdenum at irradiation temperatures <700 °C has also been characterized by large increases in hardness and tensile strength [1–5,11]. Electrical resistivity measurements may also be used as an indirect measure of defect density, where larger increases in electrical resistivity are observed following irradiation at lower temperatures due to the presence of a higher number density of defects [12–14]. The purpose of this work is to determine the change in hardness and electrical resistivity of wrought LCAC molybdenum sheet following irradiation and post-irradiation annealing and to use this data as an indirect measure of defect density for determining the kinetics of recovery and defect annihilation following irradiation between 270 °C and 1100 °C.

2. Materials and experimental procedure

The LCAC molybdenum sheet was obtained from H.C. Starck, Inc. [5]. The chemistry of this material is shown in Table 1 to meet ASTM B386-365 specifications for LCAC [6]. Tungsten, iron, chromium, titanium, and nickel are the most abundant tramp elements, with the highest levels observed for tungsten. The LCAC molybdenum was rolled into sheet using standard commercial processing methods, and then given a final stress-relief anneal at 900 °C for 1 h [15,16]. A subset of LCAC specimens were recrystallized by heat treatment at 1150 °C for 1 h. Subsized SS-1 tensile specimens were machined in the longitudinal or transverse orientation, laser-scribed for identification, pickled in a solution of 10 parts acetic acid + 4 parts nitric acid + 1 part HF acid for 5–15 s to remove 25–51 μm of material, and electropolished [5,15]. Four metallurgical conditions were used for the tensile specimens: (1) longitudinal stress-relieved (LSR), (2) transverse stress-relieved (TSR), (3) longitudinal recrystallized (LR), and (4) transverse recrystallized (TR).

The irradiation of the LCAC tensile specimens has been described elsewhere [5]. Ten tensile specimens for each irradiation capsule were loaded into a rectangular axial opening machined into a 5.08 cm diameter × 5.6 cm long cylindrical specimen holder. Holders were made of aluminum for the 270 °C irradiation and vanadium for the 605 °C, 935 °C, and 1100 °C irradiations. The tensile holders were centered within the capsule using a thimble and then hermetically sealed by welding the capsules in an inert helium atmosphere. A thermal model was used to determine the temperature of the tensile specimen during irradiation, which is primarily dependent on the size of gas-gaps. The temperature gradients in the tensile specimens were determined from thermal calculations to be on the order of 10 °C at an irradiation temperature of 270 °C and on the order of 25 °C at an irradiation temperature of 1100 °C. The irradiation temperatures were verified by taking post-irradiated electrical resistivity measurements from passive silicon carbide (SiC) temperature monitors after isochronal anneals performed in increments of 20 °C [5,15,17]. The attained irradiation temperature was generally within 100 °C of the target irradiation temperature, as summarized in Table 2 [5]. Thermal analysis results indicated that the variation of the irradiation temperature along the length of the tensile specimen was ±25 °C at the higher irradiation temperatures, and ±15 °C at the irradiation temperature of 270 °C [5].

The capsules were irradiated in the peripheral target tube position (PTP) of the high flux isotope reactor (HFIR) under the conditions given in Table 2 in one to three cycles at 85 MW of power [5]: (1) cycle 380 for 2230 MW days (26.2 days of operation), (2) cycle 381 for 2123 MW days (25.0 days of operation), and (3) cycle 382 for 1755 MW days (20.6 days of operation). No ef-

Table 1
Chemical analysis of the LCAC molybdenum sheet [5] (in weight ppm)

Material/Lot#	C	O	N	Ti	Zr	Fe	Ni	Si	La	Al	Ca	Cr	Cu	Other
LCAC sheet Ingot40386A2 Heat#C18605 0.51 mm sheet	90	3	4	NA	NA	10	<10	<10	NA	NA	NA	NA	NA	NA
LCAC specification[a]	$\leqslant$100	$\leqslant$15	$\leqslant$20	NA	NA	$\leqslant$100	$\leqslant$20	$\leqslant$100	NA	NA	NA	NA	NA	NA
GDMS data														
LCAC sheet	$\sim$100	$\sim$45	$\sim$2	8	0.95	20	2.2	1	<.005	1.7	0.1	3.9	0.3	150 W

1. NA = Not available.
2. All material was obtained from H.C. Starck, which was formerly known as CSM Industries, Inc., Cleveland, OH.
3. Trace GDMS composition for elements not listed was <1 ppm.

[a] ASTM B386-365 for arc-cast LCAC [6].

Table 2
Irradiation temperature, neutron fluence, neutron flux, and calculated DPA values for LCAC molybdenum following irradiation in HFIR

Target irradiation temperature[a] [°C]	Tensile specimen irradiation temperature[b] [°C]	Neutron fluence [$E > 0.1$ MeV], $\times 10^{24}$ n/m^2/(estimated molybdenum DPA)[c] [18]			Average fast neutron flux [$E > 0.1$ MeV] $\times 10^{18}$ n/m^2 s	Peak thermal neutron flux[d] [$E < 0.1$ MeV] $\times 10^{19}$ n/m^2 s
300	270 ± 15	10.5/(0.6)	N/A	N/A	4.64	1.0
600	605 ± 25	16.2/(0.9)	27.0/(1.4)	N/A	6.80	1.5
1000	935 ± 25	18.0/(1.0)	44.6/(2.4)	62.7/(3.3)	10.09	2.2
1200	1100 ± 25	22.9/(1.2)	44.7/(2.4)	61.3/(3.3)	10.04	2.2

N/A indicates that irradiations were not performed at these conditions.

[a] The target irradiation temperatures were used for the design of the irradiation test.

[b] The specimen irradiation temperatures are the temperatures that were determined based on confirmatory data from SiC temperature monitors and thermal modeling, with an estimated error of ±15 °C for irradiations at 270 °C, and an error of ±25 °C for the irradiations at 605 °C, 935°C, and 1100 °C.

[c] The conversion from neutron fluence to molybdenum DPA for the HFIR spectrum was determined using SPECTER [18].

[d] Peak unperturbed values in the PTP location for fast and thermal neutron flux are estimated to be 10×10^{18} n/m^2 s ($E > 0.1$ MeV) and 2.2×10^{19} n/m^2 s ($E < 0.1$ MeV), respectively. The estimates of peak thermal fluence are based on the axial factor for the location of the capsules. Detailed calculations of the change in thermal flux as a result of the capsule environment were not performed. No determination of thermal fluence was made as thermal flux was not believed to be important in this work due to the high fast flux.

fort was made to shield the capsules or specimens from the neutron spectrum that is produced by HFIR, and a nominal peak fast neutron flux of 10×10^{18} n/m^2 s ($E > 0.1$ MeV), and a peak thermal neutron flux of 2.2×10^{19} n/m^2 sec ($E < 0.1$ MeV) was estimated for the PTP locations. Aluminum holders were used for the 270 °C irradiations while vanadium was used for the 605 °C, 935 °C, and 1100 °C irradiations, which could have a slight effect on the thermal flux, but detailed calculations of the change in thermal fluence with the different holder materials were not determined. Small changes in thermal fluence can slightly change the amount of transmutation products produced in some metals, such as copper, and the difference in holder materials for the 270 °C and 605–1100 °C irradiations could slightly affect the measurements of electrical resistivity but would be expected to have no measurable effect on the hardness measurements. The irradiation damage and dpa produced by the irradiation was primarily the result of the fast flux, and thermal fluence values were not a significant contribution to the irradiation damage. Transmutation of molybdenum after irradiation in HFIR to the maximum fluence values reported in Table 2 (3.3 dpa) is calculated to produce a very low concentration (<0.2 wt%) of transmutation products that are primarily Tc and Ru with 3–4 ppm amounts of Zr and Nb [5,38]. Independent calculations were performed using an ORIGEN-S point depletion program, and the results were comparable to those reported by Greenwood and Garner [38]. These low levels of transmutation products are expected to have a negligible effect on hardening in comparison to the defects produced by irradiation in this work, but transmutation products could have a significant effect on changes in electrical resistivity.

The hardness and electrical resistivity of LCAC molybdenum were determined at room-temperature from a single tensile specimen in each of the non-irradiated, post-irradiated, and post-irradiated + annealed conditions. Electrical resistivity measurements were performed on tensile specimens using the standard 4-point method [19] using a Keithley potentiometer. A constant current of 100 mA was used for the measurement of resistance, using an average of at least five measurements. Commercially available spring-loaded mini-hook clips that were beveled to create a well-defined contact point were epoxied into four (4.2 mm diameter) through-holes in a Plexiglas fixture. Stainless steel wire was used as guide pins for pin holes in the tensile specimens. The distance between the contacts always remained constant during the measurements. All specimens behaved in an Ohmic fashion (i.e., $V = IR$, where V is the voltage, I is the current, and R is the resistance). Room-temperature (21.6 °C) was chosen as the reference temperature, and a temperature correction was made for any deviation from this reference temperature. The dimensions of the tensile specimen were measured (±1 μm) using a Mitutoyo digital micrometer.

A Buehler microhardness tester was used for Vicker's hardness measurements on the grip region of tensile specimens that had previously been tested at room-temperature. A dwell time of five seconds was used with a 500 g load. No additional surface preparation was given to the samples. The hardness measurements are made normal to the surface of the specimen and the longitudinal or transverse orientation of the tensile specimen used for this measurement has no relevance. The hardness measurements are identified as being performed on either stress-relieved and recrystallized material with no distinction given for the longitudinal or transverse orientation of the tensile specimen. The distance from the specimen edge was greater than ten (10) diagonals of the Vicker's hardness indenter to avoid plastic interaction with a free surface [20]. The hardness values reported represent an average of four to 10 measurements.

Annealing was performed in a vacuum furnace ($<6 \times 10^{-5}$ MPa) that was equipped with molybdenum heating elements and molybdenum heat shields. Heating to the annealing temperature was typically achieved in 30–45 min. All isochronal anneal holds were for 1-h, while the isothermal anneal was performed at 700 °C for periods of time ranging from 0.5 to 64 h. This work represents the first effort to anneal molybdenum that has been irradiated to high fluences at temperatures close to Stage V recovery temperatures. Metallographic examination of the unirradiated specimens was performed using a Murakami etch (10 g potassium ferri-cyanide + 10 g potassium hydroxide + 100 ml water).

3. Results and discussion: pre- and post-irradiation and isochronal annealing

The starting microstructure of stress-relieved LCAC molybdenum sheet is shown in Fig. 1 to consist of elongated, pancaked grains that are nominally 1–15 μm in diameter (average width = 3.9 μm) and aligned in the longitudinal direction with grain length ranging from 50 to 325 μm (average = 172 μm). Grain size in the transverse direction ranged from 2 to 15 μm in diameter (average width = 5.0 μm) with length from 20 to 170 (average = 78.1 μm). Recrystallization of LCAC molybdenum produces larger equiaxed grains (16.2 μm average diameter (range of 5–30 μm) and 74.1 average length for longitudinal (range of 15–210 μm); 17.5 μm average diameter (range of 8–32 μm) and 47.1 μm average length (range of 10–166 μm) for transverse). Recrystallization of the material results in lower hardness and tensile strength in the non-irradiated condition [5] in comparison to the stress-relieved condition, see Table 3. A low

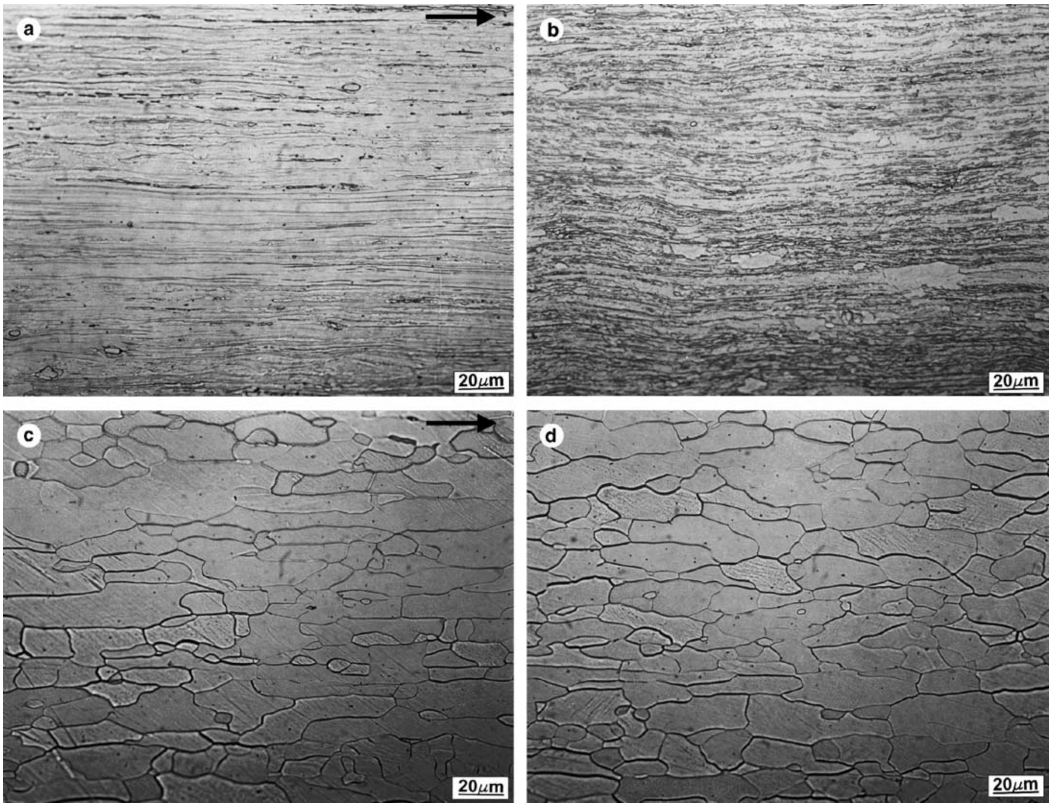

Fig. 1. Optical metallography of the LCAC molybdenum microstructure: (a) longitudinal orientation in the stress-relieved condition (LSR), (b) transverse orientation in the stress-relieved condition (TSR), (c) longitudinal orientation in the recrystallized condition (LR), and (d) transverse orientation in the recrystallized condition (TR). A Murakami etch was used. The arrow identifies the longitudinal direction.

Table 3
Summary of average Vickers hardness values for LCAC molybdenum sheet in the non-irradiated and post-irradiated condition

Alloy condition	Irradiation temperature [°C]	Average Vickers hardness value ± standard deviation [MPa] for a given neutron fluence value × 10^{24} n/m^2 [$E > 0.1$ MeV]/dpa			
		0	10.5–22.9/0.5–1.2 dpa	27.0–44.7/1.4–2.4 dpa	61.3–62.7/3.3 dpa
Stress-relieved	270	257.4 ± 3.6	401.9 ± 4.4	NA	NA
		259.6 ± 5.8	394.0 ± 3.9		
		265.6 ± 3.6			
Recrystallized		172.9 ± 5.3	368.0 ± 2.6	NA	NA
			349.4 ± 8.7		
Stress-relieved	605	See above	324.4 ± 5.4	379.3 ± 1.5	NA
			325.6 ± 5.1	402.2 ± 8.3	
			337.4 ± 8.9		
Recrystallized			281.6 ± 4.9	337.9 ± 11.5	NA
			297.3 ± 5.9	347.4 ± 6.5	
Stress-relieved	935	See above	194.8 ± 1.7	184.8 ± 4.2	152.6 ± 4.1
			206.2 ± 5.4	196.2 ± 4.0	160.6 ± 6.1
				204.3 ± 3.9	169.0 ± 4.0
Recrystallized			204.6 ± 6.5	192.7 ± 2.7	155.3 ± 4.5
			209.3 ± 6.7	199.6[a]	157.8 ± 1.6
					173.7 ± 6.7
Stress-relieved	1100	See above	167.2 ± 3.3	167.4 ± 2.0	175.3 ± 4.5
				170.6 ± 6.2	178.9 ± 3.7
Recrystallized			164.5 ± 3.4	168.0 ± 2.8	181.2 ± 4.7
			165.2 ± 4.2	175.0 ± 4.2	

1. Each value is the average of 4–10 measurements.
2. Multiple values for each condition represent duplicate measurements made on the same or alternate specimens.

[a] A single value indicates that only one value was measured in this case.

density of small particles was observed within the microstructure of LCAC molybdenum that were assumed to be carbides [5].

3.1. Change in hardness after irradiation

Non-irradiated and post-irradiated hardness values are summarized in Table 3. The repeat measurements were generally identical statistically within a 95% confidence interval, but scatter was observed in some cases. Since the microhardness measurements are taken from the surface of a specimen, local variations in the microstructure, such as carbides and grain boundaries, likely contribute to the variation in hardness observed. Direct correlations between irradiated hardness and tensile strength have been reported for irradiated molybdenum [1,11]. A direct correlation between the hardness data and tensile strength values were not resolved in this work, but the relative trends for the hardness and yield strength data were similar [5]. The highest post-irradiated hardness and tensile strength values [5] were observed for the 270 °C irradiation temperatures, with slightly lower values for the 605 °C irradiations, while the values measured for the 935 and 1100 °C irradiations were lowest and were generally close to data for non-irradiated recrystallized LCAC molybdenum. Since recrystallization of stress-relieved LCAC would be expected during the 935–1100 °C irradiations, comparisons with non-irradiated recrystallized data are appropriate. For irradiations at temperatures $\leqslant$605 °C, the hardness values for stress-relieved LCAC were consistently higher than those observed for recrystallized LCAC, and consistent with trends in tensile data [5].

The % difference in pre- and post-irradiation hardness values for the 935–1100 °C irradiations of both stress-relieved and recrystallized LCAC molybdenum ranged from −12% to +18% with little net change in hardness, which is similar to the small changes in yield strength (−11% to +29%) observed for 935–1100 °C irradiations [5]. The small differences in hardness between 935 and 1100 °C irradiated and non-irradiated LCAC likely represent sample to sample variability as well as variability introduced by the in-reactor recrystallization. For recrystallized LCAC molybdenum, the % difference in hardness values between non-irradiated and irradiated conditions for the 270 °C irradiations (102–113%) and 605 °C irradiations at the highest fluence (93–101%) were relatively close to the % difference in the reported room-temperature tensile strength values for 270 °C irradiations (88–98%) and 605 °C irradiations (54–64%) [5]. For stress-relieved LCAC, the differences in hardness for the 270 °C irradiations (48–56%) and

605 °C irradiations at the highest fluence (43–56%) were generally much lower than the % difference in tensile strength values for 270–605 °C irradiations (39–100%) [5]. Although the % increase in yield strength values for irradiated stress-relieved and recrystallized LCAC were generally comparable [5], a larger % increase in hardness was observed for irradiated recrystallized LCAC. Recrystallized LCAC has a larger spacing between strengthening barriers (larger grain size, coarser size of carbide inclusions, and lower dislocation density) that results in a lower non-irradiated hardness, and larger % increases in hardness are possible after irradiation.

3.2. Change in hardness after isochronal annealing

Plots of hardness versus isochronal annealing temperatures for 270 °C and 605 °C irradiated LCAC show in Figs. 2(a) and 3(a), respectively, that recovery of the hardness begins at 600–900 °C, which is close to or slightly above the reported Stage V recovery temperature (≈600 °C) for molybdenum [21]. Coarsening of voids in irradiated molybdenum is reported to occur at annealing temperatures (600–1100 °C) that are comparable to or higher than Stage V recovery temperatures [1–3,13,21–32]. The hardening of LCAC molybdenum irradiated at temperatures ⩽605 °C likely results from a fine spacing of small voids and/or dislocation loops [13]. Recovery of molybdenum irradiated at temperatures ⩽605 °C is reported to result from the coarsening of loops/voids into larger voids by the solid-state diffusion of vacancies with a decrease in the number density of voids that provides less hardening [3,11,13,21–32]. Stage V is the temperature at which the solid-state diffusion of vacancies becomes fast enough for such recovery to occur.

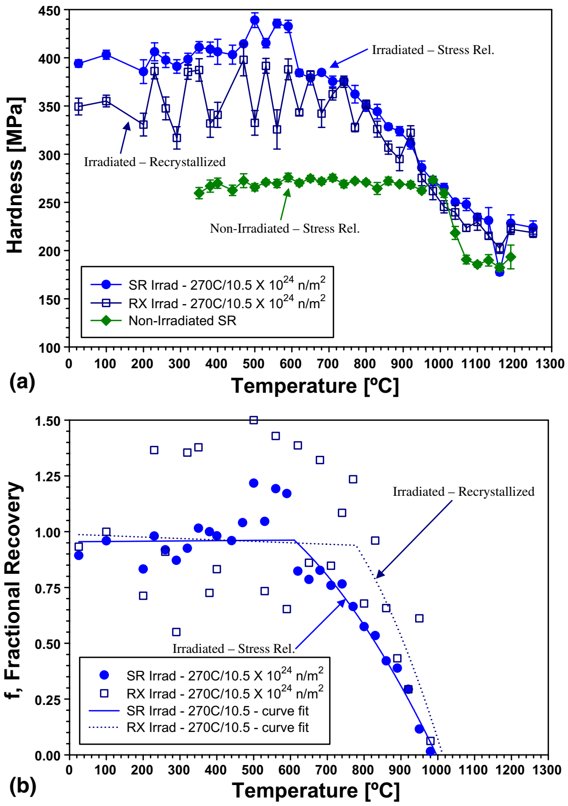

Fig. 2. Isochronal annealing results (1 h anneals) for LCAC molybdenum irradiated at 270 °C to a nominal fluence of 10.5×10^{24} n/m^2 ($E > 0.1$ MeV)/0.5 dpa for the stress-relieved and recrystallized material compared to a non-irradiated control: (a) plot of average hardness versus isochronal annealing temperature with the error bars representing one standard deviation, and (b) plot of fractional recovery, *f*, from Eq. (3) versus isochronal annealing temperature.

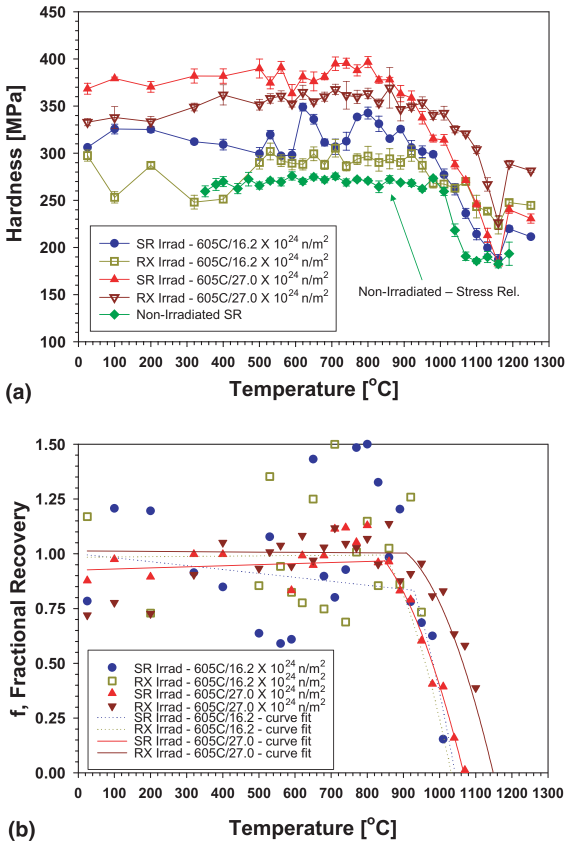

Fig. 3. Plots of hardness recovery during the isochronal annealing (1 h anneals) of LCAC molybdenum irradiated at 605 °C to a nominal fluence of 16.2×10^{24} n/m^2 and 27.0×10^{24} n/m^2 ($E > 0.1$ MeV)/0.9 and 1.4 dpa for stress-relieved and recrystallized material compared to a non-irradiated stress-relieved control: (a) average hardness with error bars represent one standard deviation, and (b) fractional recovery, *f* (Eq. (3)).

Radiation anneal hardening (RAH) has been reported in literature to occur in molybdenum by the coarsening of point defect clusters into larger, more effective strengthening barriers at annealing temperatures between 300 °C and 800 °C [26,33–36]. Increased scatter and small increases in hardness above the as-irradiated value are resolved in some cases for 270 °C and 605 °C irradiated LCAC at annealing temperatures where RAH is expected (300–800 °C). The RAH observed (1–14% increase in hardness) is a small fraction of the % increase in hardness from irradiation alone (39–113%), which indicates that the strengthening barriers produced by irradiation are controlling the mechanical properties and are of most interest [26].

The annealing temperature at which the hardness returns to the non-irradiated value for 270 °C irradiated LCAC is 980 °C, see Table 4. Full recovery of the 605 °C irradiated LCAC is observed at 1010 °C and 1070–1130 °C for the neutron fluences of 16.2 and 27.0×10^{24} n/m^2 (0.9 and 1.4 dpa), respectively. Although the starting hardness and % change in hardness of stress-relieved and recrystallized LCAC are different following irradiation at 270 °C and 605 °C, the temperatures at which recovery begins and full recovery is observed for the respective irradiation temperatures are generally the same. The recovered hardness values were slightly higher than the hardness for non-irradiated recrystallized LCAC molybdenum, which likely results from some fraction of coarse voids that remain after annealing [3,11,22–32]. The temperatures for full recovery of the hardness for the 270 °C and 605 °C irradiated LCAC are generally within the range of temperatures reported in literature (1000–1200 °C) for molybdenum irradiated between 70 °C and 700 °C to similar fluences, see Table 4.

The slightly lower temperature for full recovery of the relatively low neutron fluence 270 °C irradiation (980 °C) may indicate that full saturation of the irradiated properties has not been achieved [5]. Irradiation of molybdenum to high neutron fluences results in the formation of a void lattice, and the spacing of voids is much finer at lower irradiation temperatures with finer void sizes observed at lower irradiation temperatures [39,40]. Although a void lattice has not been observed

Table 4
Summary of the temperature of recovery determined after a series of 1 h isochronal anneals, as determined from the hardness and electrical resistivity data

Irradiation temperature (°C)	Neutron fluence [$\times 10^{24}$ n/m^2, $E > 0.1$ MeV]	Full recovery temperature (°C), determined from	
		Hardness	Electrical resistivity
LCAC molybdenum			
270	10.5	980	920
605	16.2	1010	1010C
605	27.0	1070	1130[a]
935	18.0	RT[b]	RT[c]
935	44.6	RT[b]	RT[c]
935	62.7	RT[b]	RT[c]
1100	22.9	RT[b]	RT[c]
1100	44.7	RT[b]	RT[c]
1100	61.3	RT[b]	RT[c]
Literature data[d]			
455 – [3]	250	1050	NA
70 – [1,11]	1.1	1200	NA
430 – [13]	~100[e]	1450	NA
70 – [22]	1.4	1000	NA
40 – [23]	0.4[e]	800	NA
40 – [24]	0.062[e]	1000	NA
465 – [25]	140	1050	NA
450 – [27]	0.7[e]	1050	NA
470 – [28]	40[e]	1100	NA

N/A means this property was not measured.

[a] True recovery was not resolved for this specimen.

[b] The hardness values for the as-irradiated specimens were within the range of values for the as-received control of recrystallized LCAC.

[c] These specimens exhibited electrical resistivity values close to the control specimen in the as-irradiated condition.

[d] The reference number is indicated for irradiation temperature.

[e] The neutron fluence for these earlier studies is in terms of $E > 1$ MeV. A rough estimate for fluence in terms of $E > 0.1$ MeV can be obtained by multiplying the fluence ($E > 1$ MeV) times a factor of 2.

at irradiation temperatures ⩽420 °C, the formation of finely spaced, small voids and/or dislocation loops has been reported [12,13,22,26,32,39]. Irradiation of molybdenum at 270 °C would be expected to result in the formation of finely spaced dislocation loops and voids that are small in size, which would be expected to recover at lower isochronal annealing temperatures as the diffusion distance between the loops and voids is shorter. A slightly higher temperature for full recovery was identified for the higher neutron fluence 605 °C irradiation, which may indicate that saturation of the defect density has been achieved under these conditions [5]. Irradiation of molybdenum at 605 °C would be expected to result in no dislocation loops and a coarser distribution of voids than observed for the 270 °C irradiations, and coarsening of these larger voids would therefore be expected to occur at higher annealing temperatures of 800–1100 °C [12,13,22,26,30].

No resolvable change in hardness was observed in Fig. 4(a) and (b) for the isochronal annealing of the 935 °C and 1100 °C irradiated LCAC, respectively, and the hardnesses are close to the values for the non-irradiated control after recrystallization at 1070 °C. Thus, recovery temperatures were not identified for LCAC molybdenum irradiated at 935–1100 °C. The variation in hardness values for LCAC irradiated at temperatures ⩾935 °C are likely the result of the coarse void distribution and differences in microstructure (grain size and carbide distribution) that are produced by the thermal exposure during irradiation, which can result in hardness values that are higher or lower than the non-irradiated control. Coarse voids that are widely spaced are reported to be formed at irradiation temperatures between 900 °C and 1100 °C [26,39,40], which results in little increase in hardness, and recovery by decreases in hardness were not detected. These results indicate that irradiation of LCAC molybdenum at temperatures ⩾935 °C results in little hardening and therefore no recovery during 1 h isochronal anneals to temperatures of 1200 °C.

3.3. Change in electrical resistivity after irradiation

A summary of the change in electrical resistivity values after irradiation shows in Table 5 that the largest increase (10.1–12.0%) was observed at the 270 °C irradiation temperature with a more modest increase for the 605 °C irradiations (2.8–5.3%). Although the neutron fluences for the 935–1100 °C irradiations were a factor of 2–6 larger than for the irradiations at 270 °C and 605 °C, the changes in electrical resistivity were the lowest (0.5–2.6%). The change in electrical resistivity after irradiation for the four different metallurgical conditions of LCAC (LSR, LR, TSR, and TR) appeared similar and no trend was resolved with respect to metallurgical condition.

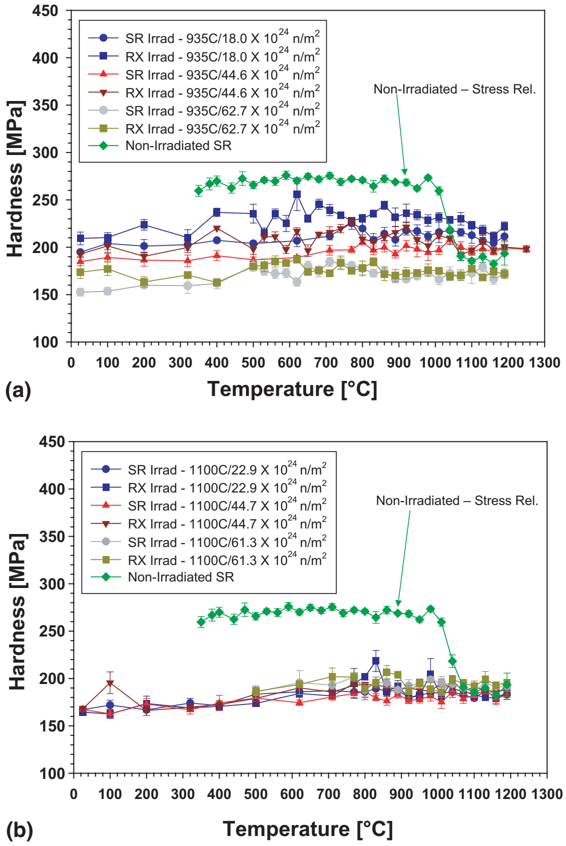

Fig. 4. Plot of Vicker's hardness versus isochronal annealing temperature (1 h anneals) for stress-relieved and recrystallized LCAC molybdenum irradiated at: (a) 935 °C to a nominal fluence of 18.0, 44.6, and 62.7 × 10^{24} n/m^2 (E > 0.1 MeV) or 1.0, 2.4, and 3.3 dpa, and (b) 1100 °C to a nominal fluence of 22.9, 44.7, and 61.3 × 10^{24} n/m^2 (E > 0.1 MeV)/1.2, 2.4, and 3.3 dpa. The points plotted are an average of at least five measurements with error bars representing one standard deviation. Results for non-irradiated stress-relieved LCAC molybdenum are also shown.

The increase in electrical resistivity can result from the creation of transmutation products and the creation of defects such as vacancies and interstitials, defect clusters, dislocation loops, and voids, while the change in hardness after irradiation results from the formation of defects that have a strong influence on dislocation mobility, such as loops and voids [11,13,26,37]. At temperatures >800 °C, the mobility of the vacancies and interstitials is high enough that these defects rapidly diffuse to sinks (grain boundaries, precipitate boundaries, pores, and dislocations) where annihilation occurs and only coarse voids are formed [13,26], which is consistent with the small increases in electrical resistivity for the 935–1100 °C irradiations. Most of the small increase in electrical resistivity for the 935–1100 °C irradiations

Table 5
Summary of the percent change in electrical resistivity values measured at room-temperature for LCAC molybdenum sheet

Alloy condition	Irradiation temperature [°C]	Average % change in electrical resistivity [%] (range of data) for a given neutron fluence value × 10^{24} n/m^2 [$E > 0.\ 1$ MeV]/dpa		
		10.5–22.9/0.5–1.2 dpa	27.0–44.7/1.4–2.4 dpa	61. 3–62.7/3.3 dpa
LSR	270	10.1 (10.4–10.0)	N/A	N/A
LR		11.3 (12.0–10.6)	N/A	N/A
TSR		12.0, 10.6	N/A	N/A
TR		11.2, 10.1	N/A	N/A
LSR	605	2.4 (2.9–1.9)	4.1 (4.3–3.8)	N/A
LR		1.5 (1.8–1.3)	3.3 (4.1–2. 8)	N/A
TSR		1.8	5.3	N/A
TR		1.7	3.6	N/A
LSR	935	−0.4 (−0.2 to −0.5)	0.9 (1.1–0.7)	N/A
LR		0.5 (0.9 to −0.1)	1.9 (2.2–1.5)	N/A
TSR		−0.7 (−0.1 to 1.6)	0.7, 0.1	N/A
TR		0.8 (1.2 to 0.1)	2.6, 2.3	N/A
LSR	1100	−0.7 (0.2 to −1.7)	1.2 (2.2–0.2)	1.2 (1.3–1.0)
LR		−0.3 (0.4 to −1.6)	2.7 (3.0–2.6)	2.1 (2.6–1.6)
TSR		−1.2 (0.2 to −2.9)	1.8, 1.6	1.0, 0.5
TR		−0.1 (0.5 to −0.9)	2.8, 2.4	1.9, 1.9

The percent change was determined by comparison of pre- and post-irradiated electrical resistivity measurements on the same specimen: $[(\rho_{\text{post}}-\rho_{\text{pre}})/\rho_{\text{pre}}\times 100\%]$.

1. A single value means that only one data point was measured.
2. Two values mean that only two data points were measured.
3. Average values with a minimum and maximum value are reported when three or more data points were determined.
4. N/A means that measured values were not available.

likely results from the formation of transmutation products with some contributions from a low number density of coarse voids [13,26]. At irradiation temperatures <700 °C where the mobility of point defects is slower, annihilation at sinks occurs at a much slower rate than the rate at which point defects are created. This results in a higher net accumulation rate of point defects that produces smaller sizes of loops and voids with a higher number density [13,26]. The net result is larger increases in electrical resistivity for the 270 °C and 605 °C irradiations. Transmutation products formed at any irradiation temperature would also be expected to have a large effect on electrical resistivity, but may be distributed very differently at the various irradiation temperatures. The formation of irradiation defects, such as voids and dislocation loops, also produces an increase in electrical resistivity that has been quantified in terms of 1% swelling results in a 1% increase in electrical resistivity [39]. Literature data indicates that the swelling of molybdenum would be expected to be very low at the neutron fluences in this work (<1%) [2,3,26,39], which indicates that the contribution of voids may be a small fraction of the total change in electrical resistivity, and transmutation products and irradiation defects play a dominant role. The strong dependence of irradiation temperature on the % changes in electrical resistivity indicates that the changes in electrical resistivity are likely dominated by the irradiation defects produced at low temperatures that have little effect on swelling, or transmutation products that are distributed very differently at lower irradiation temperatures. The strong role of irradiation temperature on hardness implies that the formation of finer irradiation defects that have little effect on swelling, such as dislocation loops, are likely the dominant microstructural feature that produces the larger changes in electrical resistivity at lower irradiation temperatures. However, more study is required to quantify the effect of transmutation products on electrical resistivity. The largest increase in electrical resistivity for the 270 °C irradiations indicates that the highest number density of point defect clusters/dislocation loops is produced. The change in electrical resistivity observed for irradiation at 270 °C and 605 °C is much lower than the change in hardness, which implies that most of the point defects produced by irradiation at these temperatures have formed dislocation loops or voids that are strong barriers to dislocation motion.

These results suggest that irradiation produces small changes in the physical properties of molybdenum that are controlled by electronic conduction transport mechanisms, such as electrical resistivity and thermal conductivity. The thermal conductivity (K) and electrical resistivity (p) at a specific temperature (T) are directly related using the Wiedemann–Franz relationship [41],

$$K = LT/\rho, \tag{1}$$

where the Lorenz number (L) can be determined for metals by using the Boltzmann constant (k_B) and charge of an electron (e),

$$L = (\pi^2/3)(k_B/e^2) = 2.45 \times 10^{-8}\ \mathrm{W\,ohm/deg^2}. \tag{2}$$

The small changes in electrical resistivity after irradiation to the neutron fluences in this study indicate that the changes in thermal conductivity would also be small.

General trends in previously reported irradiated electrical resistivity data [12–14] after high dose exposure ($100–610 \times 10^{24}\,\mathrm{n/m^2}$, $E > 0.1\,\mathrm{MeV}$) are consistent with the results reported here: (1) the largest increase in electrical resistivity was observed at lower irradiation temperatures, and (2) small increases in electrical resistivity were observed at higher irradiation temperatures. However, decreases in electrical resistivity have been reported at relatively high irradiation temperatures (910 °C and 1050 °C). The larger changes in resistivity at all irradiation temperatures for previous studies [12,13] likely result from the higher neutron fluence and lower materials purity in comparison to this work. Irradiation to higher neutron fluences than used in this work would result in the accumulation of transmutation products and irradiation defects that could result in different trends than reported herein.

3.4. Change in electrical resistivity after isochronal annealing

The recovery in electrical resistivity with isochronal annealing was generally similar to that observed for hardness. For the 270 °C and 605 °C irradiated LCAC molybdenum, Fig. 5 shows recovery of electrical resistivity values at 400–500 °C, which is slightly lower than the Stage V recovery temperature [21]. Significant recovery

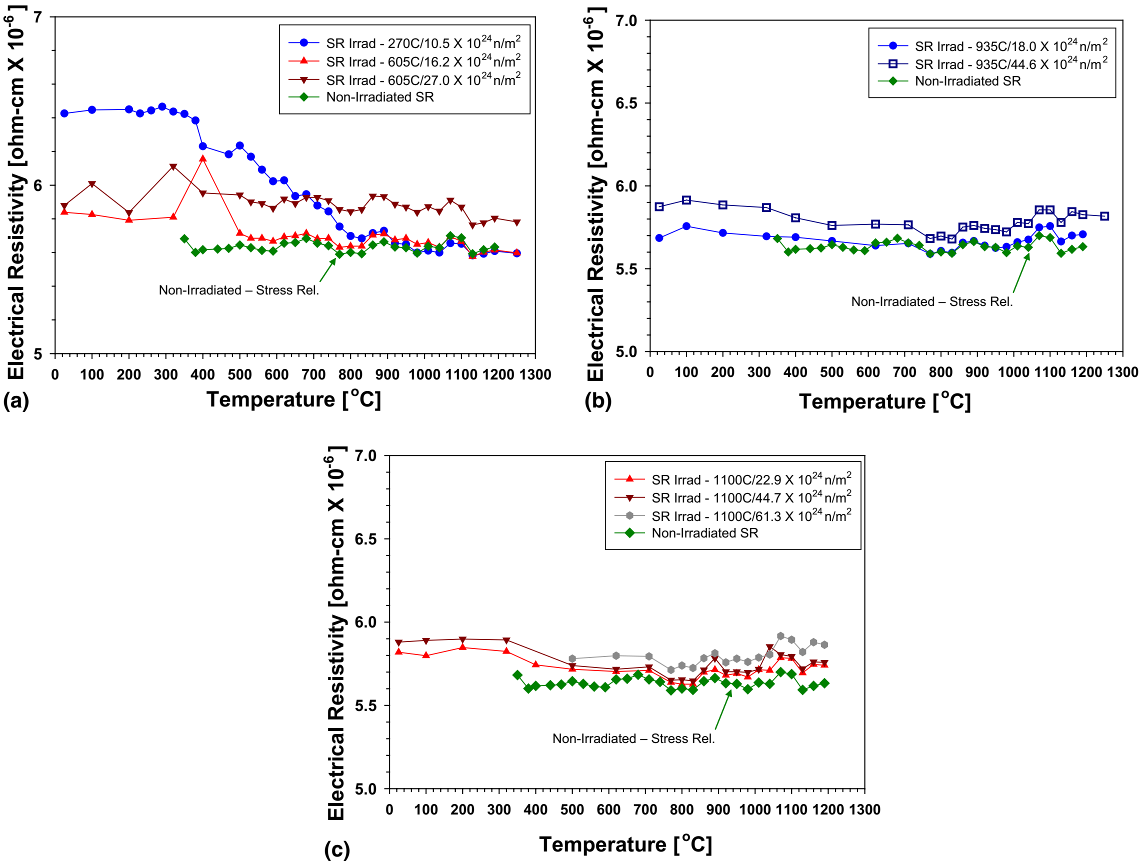

Fig. 5. Plot of electrical resistivity versus isochronal annealing temperature (1 h anneals) for stress-relieved LCAC molybdenum irradiated at: (a) 270 °C to a nominal fluence of $10.5 \times 10^{24}\,\mathrm{n/m^2}$/0.5 dpa and 605 °C to nominal fluences of 16.2×10^{20} and $27.0 \times 10^{24}\,\mathrm{n/m^2}$ ($E > 0.1\,\mathrm{MeV}$)/0.9 and 1.4 dpa, (b) 935 °C to a fluence of 18.0 and $44.6 \times 10^{24}\,\mathrm{n/m^2}$ ($E > 0.1\,\mathrm{MeV}$)/1.0 and 2.4 dpa, and (c) 1100 °C to a fluence of 22.9, 44.7, and $61.3 \times 10^{24}\,\mathrm{n/m^2}$ ($E > 0.1\,\mathrm{MeV}$)/1.2, 2.4, and 3.3 dpa. Results for a non-irradiated stress-relieved LCAC specimen are shown for comparison.

of electrical resistivity for irradiated molybdenum is reported to begin at Stage III recovery temperatures (≈160 °C) [26,42–46]. Since Stage III recovery in molybdenum would occur at a temperature less than the irradiation temperatures (270–605 °C), recovery of these defects would occur during the irradiations. The temperature determined for full recovery of the 270 °C irradiated LCAC to electrical resistivity values of the non-irradiated control (980 °C, see Table 4) was identical to the full-recovery temperature determined using hardness. The temperatures for full recovery of electrical resistivity for 605 °C irradiated LCAC to a fluence of 0.9 and 1.4 dpa were 1010 °C and 1130 °C, respectively, which are also close to the recovery temperatures determined from the hardness measurements. True recovery was not resolved for 605 °C irradiated LCAC at the fluence of 1.4 dpa, which may reflect either the formation of voids or the presence of transmutation products [13,21–26,39], and the recovery temperature was identified as the minimum value that was close to the non-irradiated control.

The electrical resistivity values for LCAC irradiated at 935 °C and 1100 °C were slightly higher than that of the non-irradiated control, but no change in electrical resistivity was observed and a recovery temperature cannot be determined, as observed for the hardness measurements. The slightly higher electrical resistivity values for the 935 °C and 1100 °C irradiated LCAC specimens in comparison to the non-irradiated control likely results from the coarsely spaced voids that are formed during irradiation, which are not removed by 1 h annealing times up to temperatures of 1200 °C, and the formation of transmutation products.

These results show that both electrical resistivity and hardness measurements can be used to evaluate the recovery of irradiated molybdenum. However, the difference in pre- and post-irradiation electrical resistivity values was very small for the neutron fluences used in this work (maximum of 12%), but a much larger difference between pre- and post-irradiation values was observed for the hardness measurements (maximum of 113%). Since the irradiation temperatures are above the Stage III recovery temperature where most of the recovery of electrical resistivity occurs, the change in electrical resistivity after irradiation at 270–1100 °C is relatively small. As a result, recovery of the defects produced by irradiation conditions in this work can be more clearly resolved using hardness measurements.

4. Results and discussion: recovery kinetics for LCAC irradiated at 270 °C and 605 °C

Isothermal annealing of 270 °C and 605 °C irradiated LCAC was performed at 700 °C to determine the activation energy for recovery using the Meechan–Brinkman method of analysis [47–49]. The fraction of recovery, *f*, during isochronal annealing in Figs. 2(b) and 3(b) was defined as

$$f = (H_a - H_{NI})/(H_{AR} - H_{NI}), \tag{3}$$

where H_a is the hardness at the isochronal annealing temperature, H_{AR} is the as-irradiated hardness, and H_{NI} is the non-irradiated hardness. The recovery rate has the general form [48]

$$-\mathrm{d}f/\mathrm{d}t = K(T)F(f), \tag{4}$$

where $F(f)$ is an Arrhenius function for a single recovery process given by the rate constant

$$K(T) = v_0 \exp(-E_a/KT), \tag{5}$$

where E_a is the activation energy of the recovery process, v_0 is the frequency factor, and k is Boltzmann's constant. Substitution of Eq. (5) in Eq. (4) with integration gives the equation used in the Meechan–Brinkman analysis,

$$(1/V_0)g(f) = (1/v_0)\int \mathrm{d}f/F(f) = t\exp(-E_a/kT), \tag{6}$$

The fractional recovery for the isothermal annealing was determined using the difference in hardness between the annealed irradiated specimen and non-irradiated control, ΔH

$$f = \Delta H/(H_{AR} - H_{NI}). \tag{7}$$

Plots of fractional recovery for the isochronal anneals show in Figs. 2(b) and Fig. 3(b) the RAH, start of recovery, and finish of recovery, as discussed in the previous section. The recovery during isochronal annealing was fit to a second order equation. Gradual recovery is shown in Fig. 6 for the isothermal annealing of 270 °C

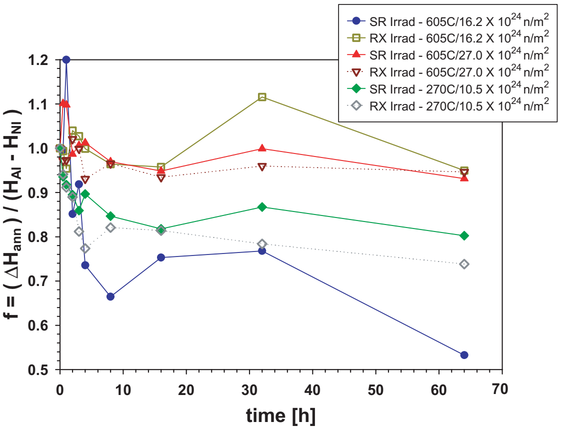

Fig. 6. Plot of fraction of recovery (Eq. (7)) versus annealing time for the 700 °C isothermal annealing of stress-relieved and recrystallized LCAC molybdenum irradiated at 270 °C to a nominal fluence of 10.5×10^{24} n/m^2 ($E > 0.1$ MeV)/0.5 dpa and 605 °C to nominal fluences of 16.2×10^{24} and 27.0×10^{24} n/m^2 ($E > 0.1$ MeV)/0.9 and 1.4 dpa.

and 605 °C irradiated LCAC. Scatter in the data is observed that is likely related to variability associated with measuring microhardness on the surface of a specimen with inhomogeneities in microstructure, but a general trend of recovery is resolved. Faster recovery was typically observed for 270 °C irradiated LCAC during the isothermal annealing, which is consistent with the expected irradiated microstructure where a higher number density of defects and finer defect size would result in a smaller diffusion distance. Irradiation at 605 °C would be expected to produce a coarser spacing of voids, and recovery during isothermal annealing is shown in Fig. 6 to generally occur at a slower rate during the isothermal annealing. Increases in hardness or RAH are observed in some cases for 605 °C irradiated LCAC at the shorter annealing times (generally 0.5–3 h), which likely results from the coarsening of point defects to form larger defects that are more effective barriers to dislocation motion. RAH of irradiated molybdenum was observed for 605 °C irradiated LCAC at 700 °C for the 1 h isochronal annealings, and RAH has been reported to occur for irradiated molybdenum at annealing temperatures between 300 °C and 800 C [26,33–36], which is consistent with these results observed for the isothermal annealing. The competition between recovery and RAH may contribute to the moderate change in hardness observed for the 700 °C isothermal annealing of 605 °C irradiated LCAC at annealing times <3 h, but recovery appears to dominate at longer annealing times. Little recovery is observed during the isothermal anneal for 605 °C irradiated recrystallized LCAC with the lower fluence of 0.9 dpa, which may be explained by the fact that the radiation hardening and defect density were not saturated for this condition [5]. Saturation of defects in irradiated molybdenum at high fluences has been observed to result in a well defined void lattice with a maximum swelling level of 4% [39,40]. More study is needed to define the neutron fluences and exact defect structure that leads to saturation of hardening in molybdenum. The highest recovery was observed for stress-relieved 605 °C irradiated stress-relieved LCAC with the shorter fluence of 0.9 dpa, suggesting that more examinations are required to explain these differences.

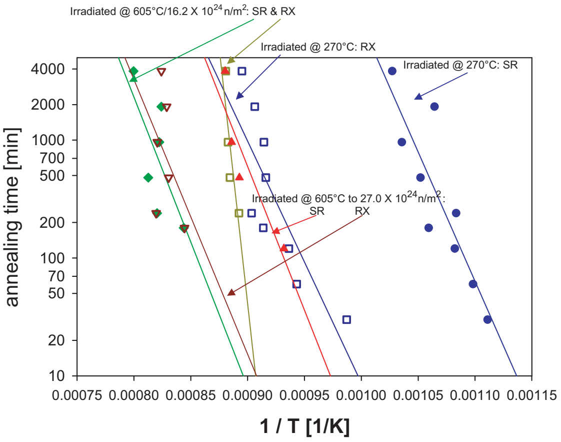

Fig. 7. Semi-log plot of isothermal time interval versus inverse isochronal annealing temperature used to determine activation energy using the Meechan–Brinkman method. Results are shown for stress-relieved and recrystallized LCAC molybdenum irradiated at 270 °C to a nominal fluence of 10.5×10^{24} n/m^2 ($E > 0.1$ MeV)/0.5 dpa and at 605 °C to nominal fluences of 16.2×10^{24} and 27.0×10^{24} n/m^2 ($E > 0.1$ MeV)/0.9 and 1.4 dpa.

Fig. 7 shows the Arrhenius plot of isothermal annealing times and inverse isochronal annealing temperature at which the fraction of recovery is equivalent, which is used to determine activation energies for annealing recovery using the Meechan–Brinkman method. Data points were not used when RAH was observed and $f > 1$, and for the 605 °C irradiated LCAC only the results at annealing times > 2–3 h were used in the kinetic analysis to avoid the effects of RAH. The activation energy values are shown in Table 6 to be within the range of values reported in literature for self-diffusion in molybdenum within the error of the measurement. The relatively large scatter in hardness values results in relatively poor-fits for the Arrhenius plot in Fig. 7, but the activation energies provide a measure of the kinetic process that is controlling the recovery. Since little recovery was observed for the 605 °C irradiated recrystallized specimen at the shorter fluence of 0.9 dpa, and radiation hardening has been shown to not be saturated for this fluence [5], activation energy values for recovery were not determined for this condition. These results indicate that the recovery of 270 °C and 605 °C irradiated LCAC molybdenum is controlled by solid-state diffusion of vacancies that likely coarsen the dislocation loops and voids to reduce the hardening. The activation energy values for 605 °C irradiated LCAC molybdenum were generally higher than for the 270 °C irradiations, which could indicate that the recovery mechanism is slightly different. Such difference in activation energy may result from the influence of RAH on the recovery of defects for 605 °C irradiated LCAC, but further investigation is needed to resolve these differences. Activation energy values slightly lower (3.3–3.5 eV) than the values for molybdenum self-diffusion and the results determined in this study have been reported [26,42], but other activation energies for recovery reported in literature are much lower than determined in this work, see Table 6. The literature studies were generally performed at lower neutron fluences, and development of the same microstructure and irradiation defect density may not have been achieved. Differences in irradiation temperature and neutron fluence that affect the starting microstructure, and differences in the isothermal annealing temperatures that can affect the recovery process, likely explain the differences in activation energy between this work

Table 6
Summary of activation energy determined from the 700 °C isothermal anneal using the Meechan–Brinkman analysis compared with literature data

Irradiation temperature	Neutron fluence [$\times 10^{24}$ n/m^2, $E > 0.1$ MeV]	Isothermal annealing temperature (°C)	Activation energy ± standard deviation [eV]
LCAC molybdenum			
270 °C – SR	10.5	700	4.35 ± 0.28
270 °C – RX	10.5		4.07 ± 1.10
605 °C – SR	16.2		4.88 ± 0.77
605 °C – RX[a]	16.2		N/A
605 °C – SR	27.0		4.83 ± 0.62
605 °C – RX	27.0		4.63 ± 1.36
Literature data [2][b]			
N/A – [26]	N/A	805–897	Stage A – 1.95 Stage B – 3.30
70 °C – [42]	0.98[c]	800	3.5
70 °C – [44]	0.11–15.0[c]	500–700	1.70 ± 0.15
600 °C – [30]	11	947–1097	2.0
40 °C – [23]	0.4[c]	134–167	1.20
Molybdenum self-diffusion[b]			
[26]	N/A	N/A	4.30
[50]	N/A	N/A	4.0
[51]	N/A	N/A	4.20

N/A means this information is not available.

[a] True recovery was not resolved for this specimen.

[b] The reference number is indicated for irradiation temperature, and self-diffusion data.

[c] The neutron fluence for these earlier studies is in terms of $E > 1$ MeV. A rough estimate for fluence in terms of $E > 0.1$ MeV can be obtained by multiplying the fluence ($E > 1$ MeV) times a factor of 2.

and other studies in the literature. This work represents the first effort to anneal molybdenum that has been irradiated to high fluences at temperatures close to Stage V recovery, and is consistent with the expectation that diffusion of vacancies is controlling recovery at Stage V temperatures.

5. Summary

The changes in hardness after irradiation of LCAC molybdenum were similar to trends observed in tensile strength in these same specimens [5]. The highest degree of hardening was observed for those samples irradiated at 270 °C. Moderate hardening and little or no hardening were observed for samples irradiated at 605 °C and 935–1100 °C, respectively. The increase in the electrical resistivity of LCAC after irradiation was similar to the trends observed for the hardness tests with respect to irradiation temperature. However, the maximum change in hardness after irradiation (113%) was much greater than the % change in electrical resistivity after irradiation (12% maximum). The increase in the electrical resistivity of molybdenum following irradiation results from the creation of transmutation products and the creation of point defects such as vacancies and interstitials, defect clusters, dislocation loops, and voids, while the change in hardness after irradiation results from the formation of defects that have a strong influence on dislocation mobility, such as loops and voids [13,26,37,39]. The defects that produce the largest increase in electrical resistivity are recovered at Stage III annealing temperatures (≈160 °C), which are less than the irradiation temperatures (270–1100 °C) so that recovery of these defects would occur during the irradiations [26,42–46]. The defects produced by irradiation at 270 °C and 605 °C are expected to be a high number density of small dislocation loops and/or voids that are shown to have a relatively small effect on properties that are controlled by electronic conduction mechanisms such as electrical resistivity, but have a strong effect on dislocation mobility. Irradiation at 935–1100 °C, where the mobility of point defects is very high, resulted in small changes in both hardness and electrical resistivity, which is consistent with the expectation of extensive recovery during the irradiations with the formation of a low number density of coarse voids [13,26]. No recovery was observed for 1 h isochronal anneals of LCAC molybdenum irradiated at 935 °C and 1100 °C since little hardening and little change in electrical resistivity was produced due to the formation of a low number density of coarse voids.

The recovery of the hardness of 270 °C and 605 °C irradiated LCAC begins at 600–900 °C, which is close to or slightly above the reported Stage V recovery temperature (≈600 °C) for molybdenum [21]. Recovery of electrical resistivity starts at slightly lower temperatures (500–600 °C) than Stage V recovery, which is consistent with defect annihilation processes involving smaller clusters. Recovery of radiation hardening is reported to result from the coarsening of dislocation loops and voids by the solid-state diffusion of vacancies at 600–900 °C with full recovery at isochronal annealing temperatures of 1000–1200 °C [12,13,22,26,30,39]. This is consistent with the results observed here, where full recovery of 270 °C- and 605 °C-irradiated molybdenum is observed at 980 °C and 1010–1130 °C, respectively. Irradiation of LCAC molybdenum at 270 °C likely produces the highest number density and smallest dislocation loop/void size, which results in the highest hardness/electrical resistivity values, and recovery generally occurs at lower temperatures due to the shorter diffusion distances involved. Irradiation of LCAC molybdenum at 605 °C likely produces a lower number density of coarser voids, and recovery occurs at higher temperatures where the mobility of vacancies is greater.

The Meechan–Brinkman method of analysis was used to show that the activation energy for recovery of 270 °C and 605 °C irradiated LCAC at an isothermal annealing temperature of 700 °C (4.07–4.88 eV ± 0.83 eV) was comparable to the activation energy for molybdenum self-diffusion (4.0–4.3 eV) within the error of the measurement. These results indicate that the recovery of 270 °C and 605 °C irradiated LCAC molybdenum is controlled by solid-state diffusion of vacancies that likely coarsen the dislocation loops and voids to reduce the hardening. This work represents the first effort to anneal molybdenum that has been irradiated to high fluences at a temperature close to Stage V recovery, and confirms the expected result that diffusion of vacancies is controlling recovery at Stage V annealing temperatures.

Acknowledgments

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