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Ion-Irradiation Effect on Strain Rate Sensitivity of Nanoindentation Hardness of W Single Crystal

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The local strain rate (LSR) dependence of nanoindentation (NI) hardness was investigated by using standard constant strain rate (CSR) test method and strain rate jump (SRJ) test method for W single crystal with the surface orientation of {001} before and after 6.4 MeV Fe^{3+} irradiations (nominal damage level of 0.1, 1 and 2 dpa, 573 K). The effect of ion-irradiation on the LSR sensitivity of NI-hardness at room temperature (RT) was evaluated by changing LSR between 0.3 s^{-1} and 0.01 s^{-1} or 0.03 s^{-1} and 0.001 s^{-1}. Under these experimental conditions, ion-irradiation increases NI-hardness and slightly decreases LSR sensitivity of NI-hardness for all damage levels. The effect is more pronounced with increasing damage level. The LSR sensitivity values are ranging between 0.015 and 0.04 in SRJ tests, and between 0.0425 and 0.06 in CSR tests, indicating that the deformation of bcc W{001} at RT is controlled by a high lattice friction stress. The decrease in LSR sensitivity by ion-irradiation could be attributed to the increase in the athermal stress caused by ion-irradiation induced defect structures, which is reflected to a decrease in the activation volume of dislocation motion in ion-irradiated W{001}. [doi:10.2320/matertrans.ML201603]

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1. Introduction

Tungsten (W) is the promising material for plasma facing components in ITER and DEMO fusion reactors[1–4]. During operation, W will be exposed to neutron irradiation which will cause irradiation hardening and embrittlement[4–8]. Off-normal plasma operation caused by plasma disruption and edge localized mode (ELM)[9] would degrade the mechanical properties of W suffering so called recrystallization embrittlement through the high heat loading, and the rapid change in thermal stress/strain may cause cracking. Since the strain rate sensitivity (SRS) is large in bcc W, the effect of irradiation on SRS is an important issue to understand material response to off-normal plasma operation that may induce irradiation embrittlement accompanied by hardening. Furthermore, the assessment of the SRS before and after irradiation offers one the important information about the characteristics of irradiation-induced obstacles to dislocation motion.

Fukumoto *et al.*[10] performed tensile tests of neutron (563 K, 0.003 dpa to 0.064 dpa) irradiated vanadium alloys, V-4Cr-4Ti-0.1Si, and the results of tensile testing at room temperature (RT) showed that the SRS was lower and higher in the specimen irradiated to 0.008 dpa and 0.064 dpa, respectively[10]. The early works of tensile tests of Wronski *et al.*[11] on neutron (2×10^{22} n/m^2; $E_n > 1$ MeV) irradiated molybdenum (Mo) showed that irradiation caused a mitigation of strain rate dependence of yield stress. They interpreted this in terms of an irradiation-induced increase in the effective activation volume for yielding[11]. Tanaka *et al.*[12] conducted tensile tests (223 K to 525 K; 2.8×10^{-6} s^{-1} to 2.8×10^{-2} s^{-1}) for neutron irradiated Mo (6.3×10^{21} n/m^2 to 5×10^{23} n/m^2; $E_n > 1$ MeV) and showed that the irradiation decreased the SRS of yield stress, which became more pronounced with increasing neutron fluence. They concluded that the athermal component of the yield stress was increased by neutron irradiation and this trend was more pronounced with increasing neutron fluence[12]. The tensile test results (223 K–373 K, 10^{-5} s^{-1} to 10^{-2} s^{-1}) of Li *et al.*[13] of neutron irradiated Mo (~353 K, 7.2×10^{-5} dpa to 0.28 dpa) showed that generally, irradiation mitigated strain rate dependence of the yield stress in comparison to unirradiated Mo. With increasing the damage level, the strain rate dependence of the yield stress was mitigated up to about 0.003 dpa. Beyond this damage level, the strain rate dependence of the yield stress was kept nearly constant[13]. No effect on the strain rate dependence of the plastic instability stress was observed[13]. Post-irradiation isochronal annealing (annealing temperature from 300 K to around 850 K; deformation temperature at 290 K) experiments by Aono *et al.*[14] for neutron irradiated ($T < 363$ K; $1.1. \times 10^{21}$ n/m^2 and 1×10^{22} n/m^2) high-purity iron single crystals revealed that the change in the SRS depended on the isochronal annealing temperature: the annealing between 300 K and 500 K caused an increase in SRS of yield stress, while between 500 K and 800 K, it led to a decrease in SRS, and above 800 K the SRS seemed to show no change compared to unirradiated condition[14].

S.M. Ohr *et al.*[15] carried out strain rate change tests and stress relaxation tests (RT, 6.56×10^{-5} s^{-1} and 6.56×10^{-4} s^{-1}) on unirradiated and neutron (1.2×10^{20} n/m^2; $E_n > 1$ MeV) irradiated Ferrovac-E iron and concluded that the irradiation caused no change in activation volume suggesting that irradiation did not induce further short range obstacles to dislocation motion. They also reported an irradiation-induced increase in the athermal stress or long range internal stress by approximately 33 MPa, which accounted for nearly all the increase in the yield stress at RT due to irradiation[15]. They further on reported that the effective stress was also increased by irradiation when tested at low temperatures[15]. McRickard *et al.*[16] carried out stress relaxation tests at a low strain rate (<0.01 s^{-1}) for neutron irradiated (2×10^{22} n/m^2; $E_n >$ 1 MeV) Ferrovac iron, and came to the same trend with them for activation volume and athermal stress. Early works[17,18] on neutron (539 K; 0.5, 4, and 6.5×10^{23} n/m^2; $E_n > 1$ MeV)

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irradiated ASTM A533-B steel showed that for all the testing temperatures, increasing fluence led to an increase in yield stress and to a decrease in strain rate sensitivity.

As for W, early works of Steichen[19] reported tensile property changes after neutron irradiation ($0.5 \times 10^{26}\,\mathrm{n/m^2}$ at 644 K and $0.9 \times 10^{26}\,\mathrm{n/m^2}$ at 655 K) over a wide range of tensile test temperatures and strain rates (295 K to 1200 K, $3 \times 10^{-4}\,\mathrm{s^{-1}}$ to $1\,\mathrm{s^{-1}}$), summarized by a rate-temperature parameter[20] and set in relation with the yield stress[20]. Under these conditions, W showed a pronounced strain rate and temperature dependence. However, no individual strain rate sensitivity values were assessed by the author[19] for neutron irradiated W.

There is no research on ion-irradiation effects on the strain rate sensitivity of W, although ion irradiation experiments have been considered to be convenient to study radiation effects on materials. Since several MeV of ion-irradiation causes a damaged layer within the range of a few microns, nanoindentation (NI) hardness test method is applied to evaluate the ion-irradiation hardening. In NI, a well-recognized deduction of SRS of NI-hardness is defined[21] in analogy to the SRS exponent formulation for uniaxial tensile and compression tests[22]. Maier *et al.*[23] conducted a strain rate jump (SRJ) test of NI-hardness to assess the localized strain rate (LSR) sensitivity, where a SRS parameter m was derived by conducting several SRJ set-ups at different indentation depths. It is noticed that SRS is now represented by LSR sensitivity in NI-hardness tests since the deformation area is limited just beneath the indenter. The original method[23] has been developed for nanocrystalline and ultrafine-grained materials exhibiting no indentation size effect (ISE)[24], which is a phenomenon that NI hardness decreases with indentation depth, but attempts have been made to apply the method to single crystal materials[25] exhibiting strong ISE[24] in order to evaluate the influence of the ISE[24] on the LSR sensitivity measurements[25]. The SRJ method was recently applied to ion-irradiated single crystal of Fe-15Cr-20Ni austenitic steel, which had an fcc structure, by Kasada *et al.*[26]. Their results showed that ion-irradiation slightly decreased SRS of NI-hardness and the SRS parameters were similar with those obtained by Sun *et al.*[27] by means of SRJ tensile test method on ultra-fine grained and coarse grained austenitic Fe-14Cr-16Ni alloy tested at 293 K[27].

LSR sensitivity of ion-irradiated bcc metal, which generally had rather higher SRS than fcc metals, is worth to be studied. Furthermore, it is considered that the feasibility check of the validity of SRS measurement by NI-hardness technique for the ion-irradiated materials is necessary for the assessment of correlation potential between ion- and neutron-irradiation effects. Thus, the objective of this study is to investigate the effect of ion-irradiation on the LSR sensitivity of NI-hardness of W single crystal for understanding characteristics of ion-irradiation induced defects as obstacles to the dislocation motion in W.

2. Experimental

2.1 Material and sample preparation

The material used in this study was a pure (99.97 mass%) W single crystal with {001} surface orientation which allowed us to evaluate the SRS without considering the effect of grain boundaries. The exact surface orientation of the specimen was measured by electron back scatter diffraction patterns (EBSD) method. Four W{001} specimens were mechanically polished by SiC abrasive papers. Then the surfaces were polished with diamond paste of which the particle size was 0.25 μm. Finally, electrochemical polishing (20 V, 5 min., 1%NaOH solution) was carried out to remove the deformation layer induced by mechanical polishing.

2.2 Ion irradiation

Three W{001} specimens were irradiated with 6.4 MeV Fe^{3+} ions at 573 K to nominal displacement damages of 0.1, 1 and 2 dpa using a tandetron accelerator in Kyoto University[28]. The target depth profile of the displacement damage and ion distribution was calculated using the SRIM (the stopping and range of ions in matter) package[29] and is given in Fig. 1, where the nominal dpa is defined as the displacement damage at the depth of 600 nm. The average threshold energy is chosen as 90 eV according to ASTM standards[30] for the SRIM[29] calculation.

2.3 Nanoindentation tests

Nanoindentation tests on the unirradiated and ion-irradiated specimens were performed using a NanoIndenter G200 of Agilent Technologies Inc., operated with a Berkovich diamond tip. The azimuthal orientation of the indenter has been set randomly with respect to the local crystal orientation, as Yao[31] experimentally and numerically pointed out for W single crystals of {001}, {011} and {111} surface orientation, that the azimuthal orientation of the indenter has a negligible effect on the load–displacement curves.

Several definitions of the term "strain rate" exist in NI test. According to Lucas *et al.*[32], indentation strain rate $\dot{\varepsilon}$ can be estimated by the eq. (1) considering the NI-hardness rate $H \approx 0$, consequently it is in good approximation to a half of the loading-rate $\dot{P}$ divided by the load P, $\dot{P}/P$:

$$\dot{\varepsilon} = \dot{h}/h = (\dot{P}/P - \dot{H}/H)/2 \approx (\dot{P}/P)/2 \quad (1)$$

where $\dot{h}/h$ is the ratio of the indenter displacement velocity $\dot{h}$ and the contact depth h[21] and $\dot{H}/H$ is the ratio of NI-hardness increasing rate $\dot{H}$ divided by the NI-hardness H. A directly

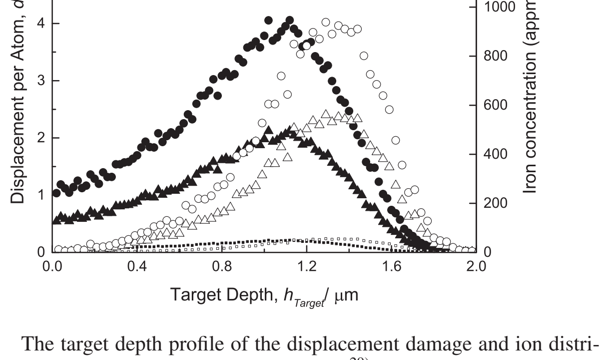

Fig. 1 The target depth profile of the displacement damage and ion distribution calculated using the SRIM package[29] (0.1 dpa, 1 dpa and 2 dpa W{001}).

proportional relationship between $\dot{h}/h$ and $\dot{P}/P$ was also shown by Cheng *et al.*[33]. Since in NI-hardness test, the strain rate is of a very localized area, we call it as local strain rate (LSR) hereinafter for the term $\dot{P}/P$, because in standard constant strain rate (CSR) test method[32] as well as SRJ test method, the ratio $\dot{P}/P$ is stable while the indentation strain rate $\dot{\varepsilon}$ that is obtained from $\dot{h}/h$ is rather unstable. In both testing methods, a continuously stiffness measurement (CSM) condition[34] was superimposed to the loading path and set to a frequency 45 Hz and an oscillation amplitude 1 nm. The testing temperatures were 299 ± 2 K.

2.3.1 Standard CSR test method

Standard CSR tests were performed at two different LSR of 0.3 s^{-1} and 0.01 s^{-1}. The LSR sensitivity was estimated from the NI-hardness values at the several depths similar to those of SRJ tests, ranging from 300 to 900 nm as shown in Fig. 4, by performing two indentation tests at two different LSR up to the maximum indentation depth of 2000 nm. At each LSR, 16 tests were carried out, whereas the 10 test results giving the smallest error bar were used for the analysis. Averaged NI-hardness at each LSR was used to estimate the LSR sensitivity.

2.3.2 SRJ test method

The SRJ method[23,25] has been applied to obtain LSR sensitivity of NI-hardness at various indentation depths. The indentation depths for initial unloading have been chosen at 8 different indentation depths up to 800 nm. The experimental set-ups are summarized in Table 1. Each of these 8 SRJ test set-ups consisted of 4–5 testing points and the tests were carried out with two different combinations of LSR changes: 0.3 s^{-1} and 0.01 s^{-1} or 0.03 s^{-1} and 0.001 s^{-1}.

2.3.3 Analytical method

The indentation strain rate change, $d\dot{\varepsilon}$, at a certain indentation depth causes the change in the NI-hardness dH, giving the LSR sensitivity, m, of NI-hardness at the indentation depth h[21,36].

$$m = d(lnH)/(d(ln\dot{\varepsilon})) \tag{2}$$

Obtaining LSR sensitivity by CSR tests with eq. (2) requires the results of two independent tests at different $\dot{\varepsilon}$ or $\dot{P}/P$. In the following work, our results and the term LSR are referred to the ratio $\dot{P}/P$ not to $\dot{\varepsilon}$, keeping in mind the proportional relationship between them[32,33]. The advantages of this method will be visible in Fig. 2 later on.

An activation volume V^*[37–41] accounting for the thermally activated deformation mechanism can be assessed as reported in the other works[25,42–45] from the indentation strain rate change in logarithmic expression $d(ln\dot{\varepsilon})$ and corresponding NI-hardness change dH at a certain indentation depth h,

$$V^* = 3\sqrt{3}kT(d(ln\dot{\varepsilon}))/(dH) \tag{3}$$

where k is the Boltzmann constant and T the absolute temperature. The factors 3 and $\sqrt{3}$ stand for the constraint factor between hardness and flow stress and for the factor between flow stress and shear stress, respectively.

2.3.4 Bulk equivalent LSR sensitivity and bulk equivalent activation volume

Nix and Gao[24] interpreted ISE based on the concept of geometrically necessary dislocation, and they defined a bulk equivalent hardness as the H at $h = \infty$, which indicated that the H was equal to H_0 at infinite depth, in the following equation:

$$H/H_0 = (1 + (h^*/h))^{1/2} \tag{4}$$

where H is the NI-hardness at a certain indentation depth h, H_0 is the hardness at the infinite depth and h^* is the so-called characteristic length, which depends on the shape of the indenter, the shear modulus and H_0[24]. In the original work of

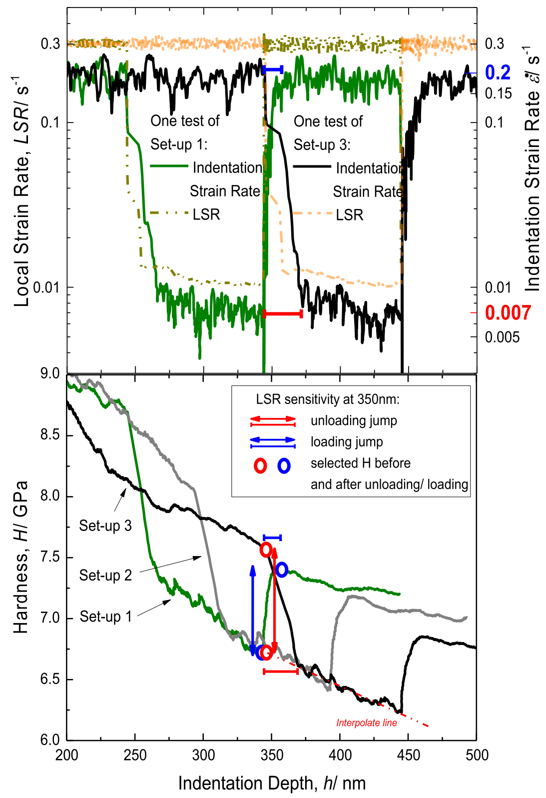

Fig. 2 SRJ test method (unloading and loading): Correlation between LSR- and indentation strain rate jumps and NI-hardness evolution. Methods to calculate LSR sensitivity by Maier *et al.* (red lines) and Kasada *et al.* (blue lines).

Table 1 The set-ups of SRJ test method, varying in depth of initial unloading and LSR. The difference of indentation depth between unloading and loading jump per set-up was kept constant over all set-ups to 100 nm.

Set-up	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
0.3–0.01 [s^{-1}]	T	T	T	T	T	T	T	T								
0.03–0.001 [s^{-1}]									T	T	T	T	T	T	T	T
h unload [nm]	250	300	350	450	550	600	650	800	250	300	350	450	550	600	650	800
h load [nm]	350	400	450	550	650	700	750	900	350	400	450	550	650	700	750	900

Nix and Gao[24]), h^* is the slope of the H/H_0 versus $1/h$ plot. To obtain the bulk equivalent LSR sensitivity of NI-hardness of the ion-irradiated W{001}, we applied the extended method of Nix and Gao[24]) by Kasada *et al.*[46]) considering SSE[35]), where the bulk equivalent NI-hardness of ion irradiated region, $H_{irr\ bulkequ.}$, can be obtained by the least square fitting of the NI-hardness data up to the so called critical indentation depth, h_c[46]), which is denoted as the indentation depth, below which the underlying substrate has a minor effect on the hardness of the thin film[35]). H_0 and $H_{irr\ bulkequ}$ were obtained for the LSR change, 0.3 s^{-1}–0.01 s^{-1} by SRJ tests and CSR tests.

The bulk equivalent activation volume has been calculated according to eq. (3). In this case, dH stands for the change of the bulk equivalent NI-hardness between two LSR.

3. Results and Discussion

3.1 Effect of unloading/loading in SRJ tests

Figure 2 shows an example of unloading and loading SRJ test for unirradiated W{001}, where the NI-hardness changes with the change in the indentation strain rate, $\dot{\varepsilon} = \dot{h}/h$, and LSR = $\dot{P}/P$ over the indentation depth. At a reduction of LSR and consequently a reduction of $\dot{\varepsilon}$, a transient decrease in NI-hardness, namely relevant unloading, was observed until the NI-hardness reached to the level corresponding to the lower LSR. Generally, compared to LSR, the $\dot{\varepsilon}$ fluctuated and delayed in reaching its target values, see the red and blue line in the upper image of Fig. 2. Moreover, $\dot{\varepsilon}$ values were not always in agreement with eq. (1) in the shallow indentation depth. The upper image of Fig. 2 shows that $\dot{\varepsilon}$ was around 2/3 of LSR, highlighted by the red and blue digits for the upper and lower $\dot{\varepsilon}$. However, because the ratio was constant for all tested LSR as well as in theory, we used LSR as a measure of strain rate as mentioned before. The LSR sensitivity was obtained by both the unloading and loading jump tests for comparison. Note that a set-up of unloading and loading needs about 100 nm of indentation depth, consequently, in this study, two set-ups of each unloading and loading jump were conducted at the same indentation depth to directly compare the results for the same indentation depth, as shown in the bottom of Fig. 2. The delay in reaching the target indentation strain rate $\dot{\varepsilon}$ is much smaller for the loading jump compared to the unloading jump, as can be seen in the both images in Fig. 2.

Maier *et al.*[25]) evaluated the LSR sensitivity by performing the unloading jump tests with taking account of the indentation depth difference caused by the delay in reaching the target $\dot{\varepsilon}$, where the NI-hardness after unloading jump was the interpolated value at the same depth with before unloading, as shown by red lines in the bottom of Fig. 2. The LSR sensitivity by the loading jump tests was also evaluated by Kasada *et al.*[26]) with the argument that an increase in strain rate would deliver more reliable SRS results, because the indentation tip may keep more continuous contact with the testing material during loading than during unloading. They defined the NI-hardness after loading jump by the plateau NI-hardness value, see the blue lines in the bottom image of Fig. 2. In their method[26]), however, since the NI-hardness values before and after loading jump are not those at the same indentation depth, it should be treated in analogy to the unloading method[25]), although the delay in reaching the target value of indentation strain rate $\dot{\varepsilon}$ is very small in the case of loading jump. In this study, we used the hardness value that most matches with the position where the target indentation strain rate was reached first time, instead of the plateau hardness value. The hardness values before and after the SRJ for unloading and loading jump that have been obtained by the two methods are marked in Fig. 2 by red and blue dots, respectively.

3.2 Irradiation effect on *m* value

The LSR sensitivity, m value, evaluated by SRJ tests of unloading and loading for unirradiated and irradiated W{001} is given in Fig. 3(a) and Fig. 3(b), respectively. Both methods, unloading and loading, led to the same conclusion, although the m values obtained by the loading tests are smaller than those by the unloading tests. Generally, the Fe^{3+} irradiation caused a slight decrease in m value and this trend is enhanced by increasing the damage level in both the cases of SRJ tests from 0.3 s^{-1} to 0.01 s^{-1} and from 0.03 s^{-1} to 0.001 s^{-1}. The m values for the higher LSR regime are larger and the effect of decreasing m by irradiation seems to be more evolved in the unloading tests. The SSE[35]) of SRS vanished at 800 nm, ex-

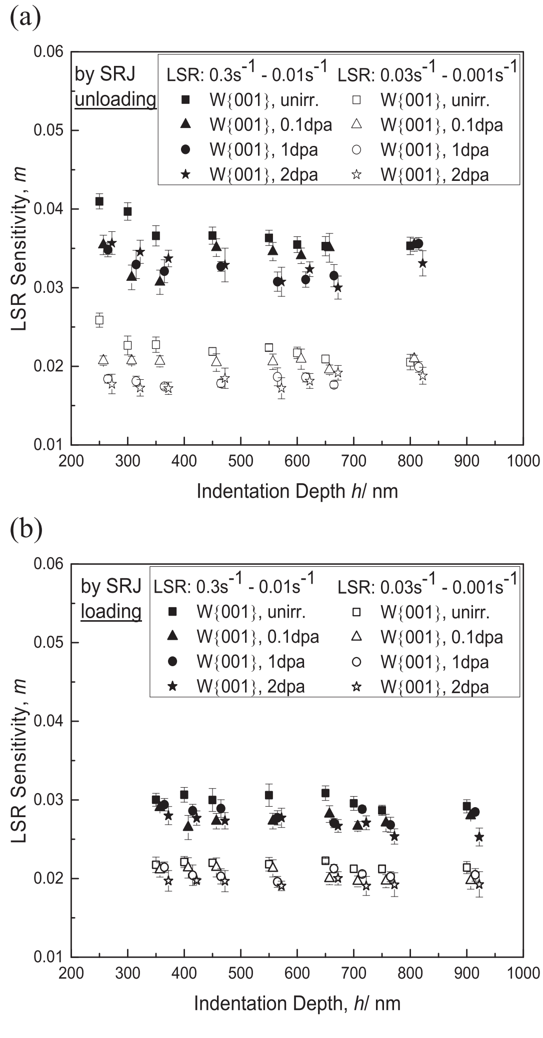

Fig. 3 Averaged LSR sensitivity m over indentation depth h for unirradiated and ion irradiated W{001} using (a) SRJ unloading test method, (b) SRJ loading test method. (Overlapped data points are shifted slightly from the actual indentation depth for eye-friendly plotting).

cept for the 2 dpa condition, which is considered to be due to a lack of contribution of irradiated area to the strain rate sensitivity for the specimen irradiated to 0.1 and 1 dpa, but a remaining irradiation effect at around 900 nm in the specimen irradiated to 2 dpa.

The m values obtained for unirradiated W{001} in this study are reasonably above and below the m values obtained by Maier *et al.*[25] (LSR change from 0.05 s^{-1} to 0.005 s^{-1}) for the higher and lower LSR regime in the current SRJ test, respectively. However, our SRJ unloading results, which are rather scattered, suggest that the ISE[24] on the LSR sensitivity cannot be neglected in this study.

The obtained m values are ranging from approximately 0.015 to 0.04 for SRJ tests and from approximately 0.0425 to 0.06 for CSR tests, as shown in Fig. 4 before and after irradiation. The absolute m values indicate a strong LSR dependence of NI-hardness, reflecting the high lattice friction in bcc W at RT[25] where the deformation is controlled by the thermally activated motion of screw dislocations. The decrease in the LSR sensitivity of NI-hardness by irradiation can be explained in terms of an increase in the athermal stress caused by irradiation-induced obstacles against dislocation motion. Ongoing microstructure observations revealed that dislocation loops of interstitial type were formed as obstacles to the dislocation motion and the trend of further decrease in m values with the displacement damage suggests the number density of obstacles increase with displacement damage. The estimated m values obtained from the tensile test data (for the tests: SRS between 3×10^{-2} s^{-1} and 3×10^{-4} s^{-1} for temperatures 700 K and 922 K) of neutron (0.5×10^{26} n/m^2 at 644 K and 0.9×10^{26} n/m^2 at 655 K) irradiated W by Steichen[19] (the neutron fluence corresponds to roughly 1.1 dpa[47,48]) are around 0.035, which agrees with the m values obtained in this research for ion-irradiated W{001} by NI test method. Larger m values in CSR tests than those in SRJ tests might be attributed to the difference in the test method as follows. Since in the NI-hardness measurement, the deformation area is limited to the localized area beneath the indentation, the LSR change could not be immediately reflected on the enough amount of dislocation motion in comparison to tensile test method. Especially an abrupt LSR change during ongoing indentation in loading as well as unloading SRJ tests, the stress relaxation is significant. Stress relaxation causes an apparent reduction of machine stiffness which could reduce the LSR in NI tests and SRJ tests. Therefore, the m values are smaller in SRJ tests than in CSR tests.

3.3 Irradiation effect on V^*

The activation volume, which is normalized by unit volume b^3 where b is the Burgers vector of W with 0.2741 nm, was obtained according to eq. (3) and the results are shown in Fig. 5(a) and (b) for unloading and loading jump test method, respectively, indicating that the activation volume seems to be slightly decreased by ion-irradiation. At each indentation depth, the activation volume of the irradiated W{001} is smaller than that of the unirradiated W{001}, and it tended to linearly increase with increasing indentation depth irrespective of irradiation condition or SRJ calculation method. Since the m values are decreased by irradiation, it is considered that the effective stress is reduced while long range athermal stress is increased. It is also considered that the irradiation-in-

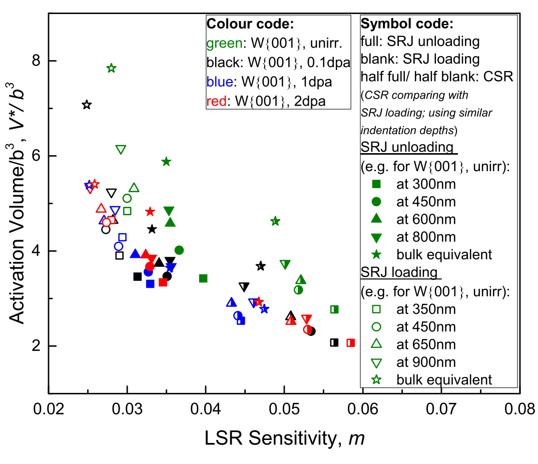

Fig. 4 Averaged SRS (LSR sensitivity and bulk equivalent LSR sensitivity) vs. averaged activation volume V^* (local activation volume and bulk equivalent activation volume), normalized by unit volume b^3 where b is Burgers vector of W, for unirradiated and ion-irradiated W{001} at various indentation depths h.

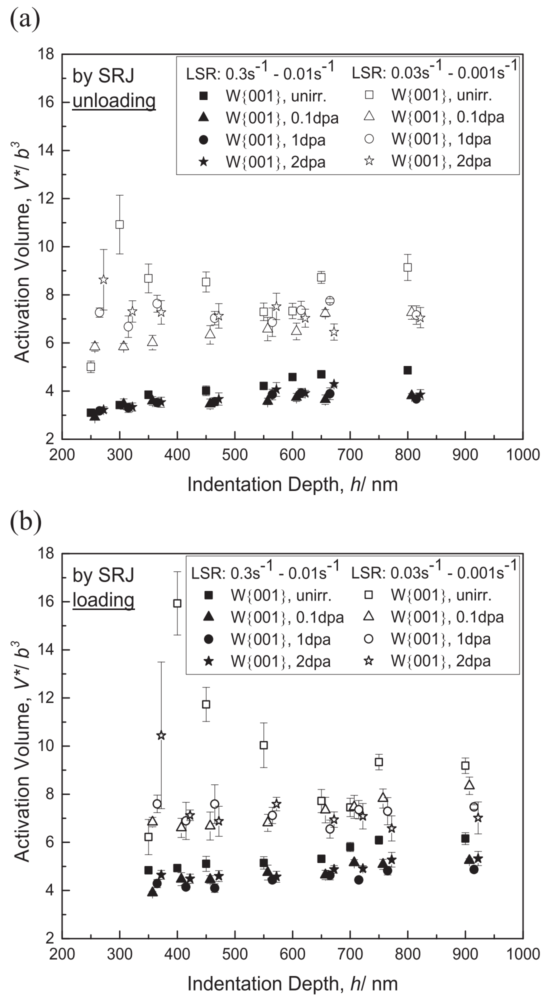

Fig. 5 Averaged local activation volume V,* normalized by unit volume b^3 where b is Burgers vector of W, over indentation depth for unirradiated and ion-irradiated W{001} using (a) SRJ unloading test method, (b) SRJ loading test method. (Overlapped data points are shifted slightly from the actual indentation depth for eye-friendly plotting).

duced reduction of activation volume could be interpreted in terms of long range stress field suppressing the kink pair motion caused by the irradiation-induced obstacles. Set-up 10, 11 and 12 in unirradiated W{001} serves higher activation volumes than the trend line, of which the reason could be attributed to the ISE[24] or measurement artifacts in low LSR tests in general. Unfortunately, Maier *et al.*[25] do not show comparable data at indentation depths between their data points of 200 nm and 400 nm. At lower LSR, the small NI-hardness changes by SRJ were difficult to estimate, and especially in the shallow indentation depth, the NI-hardness profile varied quite significantly with each individual test. This is reflected in the fluctuation of V^*, but not visible in the LSR sensitivity. Thus, the discussion in this work is focused on the SRJ tests at higher LSR regime 0.3 s^{-1} to 0.01 s^{-1}.

3.4 SRS of bulk equivalent NI-hardness

The SRS of bulk equivalent NI-hardness was estimated on the basis of the Nix and Gao model[24] applied to the ion-irradiated materials by Kasada *et al.*[46]. Note that H^2 versus $1/h$ profiles were used to obtain the unknown H_0 and $H_{irr\,bulkequ}$. In Fig. 6, the Nix and Gao[24] plots for tests at two different LSR levels are shown to estimate bulk equivalent NI-hardness in the case of irradiated W{001} to 0.1 dpa evaluated by SRJ[23,25] test method - unloading and loading - and CSR test method.

The bulk equivalent NI-hardness values were calculated according to eq. (4), from which the m values were obtained according to eq. (2). The obtained bulk equivalent NI-hardness for irradiated W{001} to 0.1 dpa by the SRJ loading/unloading test method at LSR of 0.01 s^{-1} and 0.3 s^{-1} is 5.57 GPa/6.53 GPa and 6.06GPa/7.31 GPa, compared to 5.44 GPa and 6.39 GPa by CSR test method. This results in the m values of 0.025/0.033 by SRJ loading/unloading test method and of 0.047 by CSR test method. The obtained bulk equivalent hardness and obtained bulk equivalent SRS for

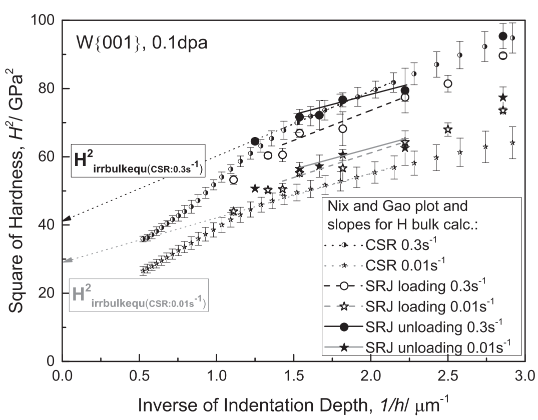

Fig. 6 Nix and Gao plot for SRJ tests (loading and unloading method) and CSR tests for 0.1 dpa W{001}.

unirradiated and 0.1 dpa irradiated W{001} are summarized in Table 2. Generally, the LSR sensitivity as well as bulk equivalent LSR sensitivity results show values of the same order of magnitude compared with the only available SRS data source for tensile tested neutron irradiated W[19].

In the original work on NI-SRJ tests[23] on nanocrystalline nickel, it was reported that by the tests at a LSR of 0.05 s^{-1}, the NI-hardness changes were almost the same between CSR tests and SRJ tests. However, decreasing the LSR led to a slight decrease in NI-hardness by SRJ tests, but to a remarkable decrease of NI hardness by CSR tests. This resulted in a higher LSR sensitivity of NI-hardness by CSR tests[23]. This could explain our experimental result at the LSR of 0.3 s^{-1} that the NI-hardness was in good agreement between SRJ unloading tests and CSR tests. However, the NI-hardness after SRJ loading jump did not reach the target value of NI-hardness by CSR tests again. This difference in NI-hardness according to different testing methods can be seen in Fig. 6.

Figure 4 also summarizes the relationship between LSR sensitivity m and activation volume V^* (average values) obtained for each irradiation condition for SRJ loading test method and CSR test method. Although the data is scattered among the plots obtained by the different methods, the effect of ion-irradiation could be confirmed to reduce both the m value and the V^*. This suggests that the ion-irradiation induced hardening is due to the increase in the obstacles to dislocation motion through the long range interaction with athermal motion of dislocations. As for the ion-irradiation induced obstacles, it is considered that the dislocation loops are the main defect structures, and the detailed microstructure examination study will be shown elsewhere. Furthermore Fig. 4 shows that the damage level does not have a significant effect on the LSR sensitivity, which is considered to be due to the fact that the ion-irradiation effects on the LSR sensitivity saturate at a low damage level of 0.1 dpa.

4. Conclusion

In this study, LSR dependence of NI-hardness was investigated by using CSR test method and SRJ test method for W{001} single crystal specimens before and after 6.4 MeV Fe^{3+} irradiations (nominal damage level of 0.1, 1 and 2 dpa, 573 K) to evaluate the effect of ion-irradiation on the LSR sensitivity of NI-hardness at RT. The obtained main conclusions are as follows:

(1) Ion-irradiation increased NI-hardness and slightly decreased LSR sensitivity of NI-hardness at all damage levels. The effect was more pronounced with increasing damage level. The m values were large ranging between 0.015 and 0.04 in SRJ tests, and between 0.0425 and 0.06 in CSR tests. This indicates that the deformation of bcc W{001} at RT was controlled by a high lattice friction stress both before and after

Table 2 Summary of bulk equivalent hardness and obtained bulk equivalent SRS for unirradiated/0.1 dpa irradiated W{001}.

Set-up	H at 0.3 s^{-1} [GPa] Unirr/0.1 dpa	H at 0.01 s^{-1} [GPa] Unirr/0.1 dpa	bulk equivalent SRS m [-] Unirr/0.1 dpa
SRJ loading	4.87/6.06	4.43/5.57	0.028/0.025
SRJ unloading	5.27/7.31	4.67/6.53	0.036/0.033
CSR	4.9/6.39	4.15/5.44	0.049/0.047

the ion-irradiation.

(2) The decrease in LSR sensitivity by ion-irradiation could be attributed to the increase in the athermal stress caused by ion-irradiation induced defect structures, which was reflected to a slight decrease, or even no change, in the activation volume in irradiated W{001}.

(3) The SSE[35] of SRS vanished at 800 nm, except for the 2 dpa condition, which is considered to be due to a lack of contribution of irradiated area to the strain rate sensitivity for the specimen irradiated to 0.1 and 1 dpa, but a remaining irradiation effect at around 900 nm in the specimen irradiated to 2 dpa.

(4) The bulk equivalent LSR sensitivity *m* and bulk equivalent activation volume *V** obtained from the bulk equivalent hardness showed reasonable values compared with the values of the averaged LSR sensitivity *m* over the indentation depth *h*.

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Appendix

List of abbreviations used in the text:
(in alphabetical order)

CSM	Continuously stiffness method
CSR	Constant strain rate
ISE	Indentation size effect
LSR	Local strain rate
SRJ	Strain rate jump
SRS	Strain rate sensitivity
SSE	Softer substrate effect

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