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# Full length article

# Understanding and quantifying electron beam effects during *in situ* TEM nanomechanical tensile testing on metal thin films



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# ABSTRACT

Transmission electron microscopy (TEM) imaging relies on high energy electrons for atomic scale resolution, however, the electrons themselves interact with and may alter the material being imaged. Using an *in situ* TEM MEMS-based nanomechanical testing technique, the effect of the electron beam (e-beam) on the deformation behavior of nanocrystalline Al and ultrafine-grained Au is investigated and quantified. We show that the e-beam enhances plastic deformation, leading to an increase in plastic strain rate and a decrease in true activation volume  $V^*$  in Al (28 to  $21b^3$ , with *b* being the Burgers vector length). The e-beam has a much weaker effect on Au. The e-beam effect is not caused by knock-on damage, but rather an effective temperature increase due to additional atomic fluctuations provided by the e-beam. The effective temperature increase is larger for Al than Au. This e-beam effect does not change the deformation mechanisms, but instead accelerates the stress-driven, thermally activated plastic deformation. These experiments provide insight into the effects of the e-beam on plastic deformation in different metals and underscore the importance of understanding and quantifying these effects for proper interpretation of measured mechanical properties during *in situ* TEM experiments.

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# 1. Introduction

Transmission electron microscopy (TEM) is a widely used tool for investigating microstructures with nanometer scale resolution. This technique benefits from the ionizing radiation of a highenergy beam of electrons that produce a plethora of signals yielding both chemical and crystallographic information [1]. Because of this, TEM is a powerful tool used to study a wide range of materials, including metals, ceramics, glasses, and semiconductors. Due to the size constraints of an electron transparent sample with an ideal thickness less than 100 nm, TEM has been particularly useful in studying both the microstructure and defects in materials with sub-micron sized features, such as nanocrystalline (nc) and ultrafine grained (ufg) metal thin films. These materials exhibit unique properties when compared to their coarse-grained counterparts, most of which can be directly attributed to the high-volume fraction of grain boundaries (GB) that influence deformation behavior [2]. Much of the current knowledge of the active deformation mechanisms in these materials has been acquired via in situ TEM deformation experiments. Such identified deformation mechanisms

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https://doi.org/10.1016/j.actamat.2021.117441 1359-6454/© 2021 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved. include dislocation emission and absorption at GBs [3–6], GB migration [7–10], and grain rotation [11–14].

The resolving power of the high-energy electron beam (e-beam) comes at a price, however, with the electrons themselves interacting with and possibly altering the material being imaged. The result of these interactions varies with material type, but common effects of electrons include radiolysis, e-beam induced specimen heating, or defect generation due to the displacement of either bulk atoms or sputtering of surface atoms [1,15]. Radiolysis and specimen heating are typically considered to have minimal impact on metal specimens due to the nature of the delocalized electrons in the atomic bonding and, at least for most metals, their high thermal conductivity [1]. This leaves knock-on damage and sputtering as the most likely primary sources of e-beam induced damage in metals. The severity of this type of damage is dependent on both the maximum transferable energy of the incident electrons and the threshold displacement energy of the specimen, both of which are related to the atomic bonding [1]. Additionally, the local bonding is disrupted at defects and GBs which leads to lower atomic bonding energies. As such, it is likely that atoms near dislocations and GBs are more easily displaced, although the exact displacement energies for these environments are not well-defined [15]. Similar sub-threshold displacement events have been reported for hydro-



gen impurities in copper and vanadium, with electron irradiation damage increasing with hydrogen content [16].

When incident electrons displace atoms in a material, this leads to the formation of defects (interstitials and vacancies) which can assist diffusional processes [15,17]. There have been a variety of e-beam-induced effects on defect behavior during in situ TEM testing reported in the literature. For example, Wang et al. reported that e-beam exposure led to the thickening of the native oxide layer on iron nanoparticles due to enhanced mass transport facilitated by e-beam induced defects [18]. Similarly, Muntifering et al. demonstrated void formation in regions of e-beam exposure during in situ annealing of self-ion irradiated nc Ni [19]. They hypothesized that e-beam exposure stimulated oxide growth in localized regions which limited the ability of vacancies to escape through the free surface thus resulting in void formation. The e-beam has also been found to selectively suppress crack growth in nc copper (Cu) thin films on a polyimide (PI) substrate by increasing the adhesion between the Cu and PI as a result of enhanced migration of Cu into the PI substrate. This led to increased interfacial area between the Cu and PI as well as oxide formation of the migrated Cu atoms with oxygen atoms present in the PI substrate, both of which strengthened the adhesion and resulted in tensile strains surpassing 30%, whereas e-beam free regions formed cracks before strains of 10% [20].

Another report states that the e-beam interaction with the interface between small-volume Al-4Cu and the native oxide layer during in situ deformation creates local regions of disorder which promotes additional dislocation nucleation sites at the interface, thus aiding plastic flow [21]. The researchers reported a decrease in apparent activation volume from  $\sim 12$  to  $\sim 4 b^3$  when subject to beam-on conditions. They attributed this decrease in activation volume to a switch in deformation mechanisms from internal dislocation multiplication to interface-dominated dislocation nucleation processes during beam-off and on conditions, respectively. There have been alternative reports of e-beam induced stress relaxation in nc and ufg Al and Au caused by increased dislocation activity activated by the e-beam [22]. It was found that the effects were more significant at TEM accelerating voltages of 120 kV over that of 200 kV, suggesting that knock-on damage was not to blame. Instead, the researchers claim that the observed e-beam effect could be a result of the depinning of dislocations by phonons generated by inelastic scattering of electrons near lattice defects. Similar findings of phonon-assisted dislocation glide have been reported in semi-conductor materials [23]. Under e-beam irradiation, Faress et al. reported that the velocity of dislocation glide in ZnS increases linearly with TEM e-beam intensity. This phenomenon is attributed to the so-called 'phonon-kick mechanism' which involves the emission of phonons from nonradiative electron-hole recombination processes at dislocation sites that lead to the activated process for dislocation migration. Other dislocation depinning mechanisms have been reported in zirconium alloys during in situ ion irradiation [24]. Under an applied stress, dislocation glide occurred during segments of ion beam exposure, whereas dislocations became pinned by irradiation defects when the ion beam was switched off. The depinning phenomena was attributed to either the local climb of dislocations through absorption of point defects or by the direct effect of the displacement cascade on the pinning point.

There is clearly not a unified understanding of the e-beam influence on material behavior during *in situ* TEM testing and characterization. In the case of *in situ* deformation, this understanding is crucial to accurately interpret experimental results as the beam effect may manifest itself by either altering the governing deformation mechanisms or by changing the rate of the active mechanisms, both of which would lead to different measured mechanical properties. In this work, we quantify the effect of the e-beam during *in situ* tensile testing on both Au and Al thin films under beam-on and -off conditions. The experiments reveal a change in mechanical behavior, activation volume, and plastic strain rate during ebeam exposure, suggesting that there is a non-negligible influence on the plastic deformation of both materials. This is the first direct quantification of an e-beam effect on true activation volume [6].

#### 2. Experimental

This study investigates the influence of the e-beam on the deformation of two different metal thin films: 200 nm thick nc Al and 100 nm thick ufg Au. Both specimens were fabricated following similar procedures involving optical lithography, electron beam evaporation of high purity Au or Al onto a Si substrate and a liftoff technique to reveal dog-bone shaped specimens with gauge dimensions of width 1.5  $\mu$ m and length 20  $\mu$ m (more details on the fabrication process can be found in Ref. [25]). The nc Al specimens have an average grain size of 90 nm and a random out-ofplane texture, while the Au specimens have an average grain size of 150 nm and exhibit <111> out-of-plane texturing. Bright-field TEM images of undeformed specimens for Al and Au are shown in Fig. 1(a) and (b), respectively.

The MEMS device used to perform the tensile tests in this study has been previously used to conduct both ex situ and in situ TEM nanomechanical testing on similar specimens [6,10,25-29]. Shown in Fig. 1(c), the device consists of a thermal actuator (TA), two interdigitated capacitive sensors ( $CS_1$  and  $CS_2$ ), a load sensor (LS) beam and a specimen gap (SG). The specimens are placed across the SG (as shown in Fig. 1(d)) using a micromanipulator and clamped using UV-curable epoxy glue. This process introduces a small amount of elastic pre-stress and strain in the specimen due to the shrinkage of the glue upon curing, which can be accounted for using the approach described in [6]. Applying a voltage across the TA causes a displacement in the actuator  $(X_A)$ which shifts the central shuttle of the device. This in turn shifts the beams in the first capacitive sensor  $(CS_1)$  which is connected in series via the specimen to the second capacitive sensor  $(CS_2)$ . The change in capacitance of  $CS_1$  and  $CS_2$  is used to determine the displacement of the actuator  $X_A$  and load sensor  $X_{LS}$ , respectively. Both capacitive sensors are calibrated individually and have typical noise levels of 0.1–0.2 fF which translates to 1–2 MPa in stress [6]. These displacements are further used to independently determine the applied force  $(F = K_{LS}X_{LS})$  and displacement  $(X_S = X_A - X_{LS})$  of the specimen, where  $K_{LS}$  is the LS stiffness (either 100 or 480 N  $m^{-1}$  in this study). Stress and strain are then determined by dividing the force and displacement by the original cross-section area and the gauge length, respectively. As the freestanding gauge length varies depending on sample manipulation, the gauge length is measured in the SEM for each specimen. This definition of strain is based on the cross-head displacement which leads to inaccurate elastic strain measurements due to finite deformation of the specimen within the fillet region in contact with the glue [26]. As a result, the slope of the linear region of the stress-strain data is not an accurate measurement of the Young's modulus. Despite this, previous work [26] has shown that the plastic strains ( $\varepsilon_p$ ) are measured accurately with this technique and are determined using the following equation,

$$\varepsilon_p = \varepsilon - \sigma / E \tag{1}$$

where  $\varepsilon$  and  $\sigma$  are the total strain and stress measured by the MEMS device and *E* is the measured apparent Young's modulus.

The MEMS device is used to perform both monotonic and stress-relaxation experiments (*in situ* and *ex situ*). By performing consecutive stress-relaxations segments, the true activation volume



**Fig. 1.** Bright-field TEM images of undeformed (a) nc Al and (b) ufg Au specimens taken at same magnification. (c) SEM image of the MEMS device used to conduct *in situ* TEM experiments. It consists of a thermal actuator (TA), two capacitive sensors (CS<sub>1</sub> and CS<sub>2</sub>), load sensing (LS) beams and a specimen gap (SG). (d) SEM image of an Au specimen clamped to the MEMS device. The specimen is placed on two droplets of epoxy glue as shown. The Al specimens have the same dog-bone type geometry and in-plane dimensions.

*V*<sup>\*</sup> is determined using the equation below [30]

$$V^* = \sqrt{3}kT \frac{\ln\left(\dot{\varepsilon}_{i2}/\dot{\varepsilon}_{f1}\right)}{\Delta\sigma_{12}} \tag{2}$$

where *k* is Boltzmann's constant, *T* is temperature,  $\Delta \sigma_{12}$  is the stress increase during the elastic reloading,  $\dot{\varepsilon}_{i2}$  is the initial plastic strain rate of the second relaxation and  $\dot{\varepsilon}_{f1}$  is the final plastic strain rate of the first relaxation segment. The plastic strain rate  $\dot{\varepsilon}_p$  is determined by

$$\dot{\varepsilon}_p = -\dot{\sigma} / M \tag{3}$$

where  $\dot{\sigma}$  is stress rate obtained by fitting the stress relaxation data with logarithmic fit and *M* is the machine-specimen stiffness [6,26,29,31]. By combining Eqs. (2) and (3), the true activation volume *V*<sup>\*</sup> can be obtained from

$$V^* = \sqrt{3}kT \frac{\ln\left(\dot{\sigma}_{i2}/\dot{\sigma}_{f1}\right)}{\Delta\sigma_{12}} \tag{4}$$

Using Eq. (4) eliminates the dependency of  $V^*$  on the strain rate and instead implies that the accuracy of  $V^*$  depends on the stress rate, which is independent of gauge length and is more accurately determined using this technique. The accuracy of the determined stress rate depends on the signal-to-noise ratio (SNR) of the measurement. Previous work has shown that a SNR > 5 requires logarithmic fits with  $R^2 > 0.9$ , which is used as a criterion for accurate  $V^*$  measurements [6]. This technique is also capable of measuring apparent activation volume [6], however, we focus on true activation volume  $V^*$  measurements in this work. It is the more relevant metric that is unaffected by the change of dislocation density during measurements. It directly represents the dislocation velocity dependence on stress, and thus the strain rate evolution in Eq. (2) is only related to dislocation velocity.

The *in situ* tensile tests were conducted in an FEI Tecnai F30 TEM operating at an accelerating voltage of either 300 kV (current  $\sim 11$  nA) or 80 kV (current  $\sim 4$  nA) using a Hummingbird electrical biasing holder. During each experiment, the beam condition was controlled and defined as either 'beam-on' or 'beam-off'. Under the beam-on condition, the e-beam was used to illuminate a portion of the specimen, while under the beam-off condition, the specimen was moved so the e-beam was exposed to vacuum only. For both the Al and Au specimens, monotonic tensile tests were conducted in which the beam condition was switched from beam-on to -off every 30 s for both 80 and 300 kV TEM accelerating voltages. Additional monotonic tensile tests were performed on the Al specimens with the beam condition constant throughout (either beam-on or

beam-off). A stress-relaxation experiment was also completed on an Al specimen while changing beam-on/off condition for the loading and paused segments in order to measure the corresponding true activation volume. The experiments conducted *ex situ* follow a similar experimental procedure to *in situ*, albeit slight differences in the calibration of  $CS_2$  [26]. More specifically, the *ex situ* setup does not allow direct calibration of  $CS_2$ , but instead relies on the assumption that  $CS_1$  and  $CS_2$  are identical. By comparing *in situ* and *ex situ* calibration constants, we approximated the error bars for *ex situ* measurements of  $V^*$  to be about 30% of the calculated values, resulting from the lower accuracy in stress measurements (i.e. accuracy in calibration of  $CS_2$ ) (see Ref. [32] for more details).

## 3. Results

The specimens used in this study have previously been tested under *in situ* TEM testing conditions. For the ufg Au thin films, *in situ* TEM observations have shown that deformation during relaxation is dominated by localized intergranular and transgranular dislocation motion [31]. The emission and subsequent transgranular glide of both partial and full dislocations are observed, with GBs serving as primary sources for dislocation nucleation [6]. The deformation behavior of the nc Al specimens under monotonic loading is characterized by dislocation glide and stress-assisted GB migration leading to extensive grain growth within the necked region [10]. In this study, we document the behavior of these two specimen types (ufg Au and nc Al) under beam-on and beam-off conditions to investigate how the e-beam influences the deformation and mechanical properties of the separate materials.

#### 3.1. Electron beam effects in nc Al during in situ deformation

In order to study the potential impact of the e-beam on the deformation of nc Al thin films, monotonic tensile tests were first conducted in either a complete beam-on or beam-off condition at different strain-rates. Fig. 2(a) shows the influence of the e-beam on the mechanical properties of the Al thin films tested under varying beam condition and strain rates (strain rates for each test are given in Fig. 2 caption). For samples tested at similar strain rates of  $1.2 - 2.6 \times 10^{-4} \text{ s}^{-1}$  (specimen #1, #3, and #5), the e-beam drastically increases the ductility of the samples, with the specimen #5 (beam-on condition) failing at 19% strain, while specimens #1 and #3 (beam-off condition) failed at strains of 3.7% and 6%, respectively. The specimens also showed variations in apparent



**Fig. 2.** Behavior of nc Al under monotonic testing for 5 different specimens tested under different beam condition and strain rates. (a) Stress-strain curves showing different tensile behavior of nc Al specimens tested under total beam-off condition (diamonds) and beam-on condition (circles) at varying strain rates. Discontinuities in the data correspond to moments in which the loading was paused to capture the deformation in more detail, leading to stress relaxation. The strain rates for each specimen are: (#1)  $1.2 \times 10^{-4} \text{ s}^{-1}$ , (#2)  $6 \times 10^{-4} \text{ s}^{-1}$ , (#3)  $1.8 \times 10^{-4} \text{ s}^{-1}$ , (#4)  $1.5 \times 10^{-5} \text{ s}^{-1}$ , (#5)  $2.7 \times 10^{-4} \text{ s}^{-1}$ . (b-g) Bright-field TEM micrographs of the fracture surfaces with the white numbers assigning the micrograph to the corresponding curve on (a). (e) Bright-field and (f) dark-field TEM micrographs of a fracture surface for a specimen tested in beam-off condition with a strain rate of  $1.5 \times 10^{-5} \text{ s}^{-1}$  which exhibit large grains within the necked region, closely resembling that of (g). (b-g) are all taken at the same magnification.

yield stress, though this may have been due to early crack initiation during deformation leading to a stress decrease. For all the curves, the offset from the zero-point origin is to account for prestress caused by the shrinkage of the glue upon curing [6]. Specimen #2 was tested under beam-on condition at a slightly faster strain rate of  $6 \times 10^{-4} \text{ s}^{-1}$ . The specimen deformed uniformly until failure quickly occurred, similar to that of specimen #1 and #3. Specimen #4 was deformed under beam-off condition with a strain rate ~10 times slower of  $1.5 \times 10^{-5} \text{ s}^{-1}$ . Under this condition, significant ductility was achieved with failure occurring at 12% strain.

*Post mortem* analysis of the fracture surfaces revealed different types of microstructures at failure depending on e-beam ex-

posure and strain rate (Fig. 2(b–g)). In agreement with the mechanical testing data, the fracture surfaces of specimens #1 and #3 (beam-off,  $\dot{\varepsilon} \sim 1.2 - 1.8 \times 10^{-4} \text{ s}^{-1}$ ) showed features characteristic of brittle failure (Fig. 2(b) and (d)). Specifically, there is no evidence of a necked region or grain growth in the surrounding area. In contrast, the fracture surfaces for specimen #5 (beam-on,  $\dot{\varepsilon} = 2.7 \times 10^{-4} \text{ s}^{-1}$ ) shows that a necked region developed alongside substantial grain growth within that region during the beamon condition (Fig. 2(g)). Grains in the necked region were measured to be upwards of 250 nm in size, almost three times larger than the initial average grain size. This grain size was determined by manually tracing the grains and calculating the diameter of a circle with area equivalent to that of the largest grains. This grain growth is consistent with other studies that reveal stress-assisted grain growth in nc metals [7,11,33–35]. Specimen #2, tested under beam-on condition with a slightly faster strain rate  $\dot{\varepsilon} = 6 \times 10^{-4}$  $s^{-1}$ , shows only a slight neck with minimal grain growth near the fractured surface. Interestingly, the mechanical behavior and microstructure evolution for specimen #4 (beam-off,  $\dot{\varepsilon} = 1.5 \times 10^{-5}$  $s^{-1}$ ) closely resembled that of the sample deformed under the beam-on condition at  $\dot{\varepsilon} = 2.7 \times 10^{-4} \text{ s}^{-1}$  and exhibited extended plasticity with a failure strain of 12%. Fig. 2(e) is a bright-field TEM micrograph of a fractured surface from such an experiment with Fig. 2(f) highlighting certain large grains in dark-field mode. In agreement with the measured ductility, the TEM micrographs show necking behavior accompanied by significant grain growth. These results indicate that the e-beam may be promoting slow strain rate conditions, without changing the deformation mechanisms.

To understand the instantaneous e-beam effect on the mechanical behavior, monotonic tensile tests were conducted at similar strain rates ( $\dot{\varepsilon} = 1 - 2.7 \times 10^{-4} \text{ s}^{-1}$ ) on nc Al specimens while alternating between beam-on to beam-off conditions every 30 s for both TEM accelerating voltages (300 and 80 kV), as shown in Fig. 3. The first beam-on (gray data) then -off (green data) segments correspond to the elastic loading of the specimen. The second beam-on segment deviates from the linearity of the previous two segments, indicating that yielding of the material has occurred under the beam-on condition. The apparent yield point for the 300 kV experiment smaller than that previously reported in Fig. 2(a) (127 MPa versus 380 MPa), however this could be an artifact of the small-scale of the specimens, which tends to amplify minor variations in the material starting geometry, alignment, or clamping that may lead to different local stress states. Based off analysis by Kang and Saif, given the specimen geometry and assuming a maximum misalignment of  $\theta = 5^{\circ}$ , the majority of the specimen is expected to be under uniaxial tension [36]. During the next beam-off segment, the stress increases in the same linear manner as the first beam-off segment, likely indicating that the stress level is not sufficient to activate deformation mechanisms in this beam-off condition or that plastic deformation occurs at a slower rate. The next 30 s with the beam-on is marked by a decrease in stress during e-beam exposure, followed by an increase in stress when the e-beam is removed again. This trend is mirrored by the next six beam-on segments which all exhibit substantial stress decrease while exposed to the e-beam, resulting in the 'saw-tooth' like behavior seen in Fig. 3(a).

It is important to note that this stress decrease is during monotonic loading (applied displacement from the MEMS thermal actuator,  $X_A$ , increases) and not during a stress-relaxation experiment (in which  $X_A$  is kept constant). A decrease in stress during monotonic loading indicates that thermally activated plasticity leads to specimen elongation at a faster rate than the applied displacement. This means that when the specimen is exposed to the ebeam, specimen elongation initially occurs at a faster rate than the applied displacement, leading to a decrease in stress even during monotonic loading.

Fig. 3(c) displays the accumulation of plastic strain (calculated using Eq. (1)) throughout the duration of the experiment, with the different beam-on/off segments colored accordingly. This figure reveals that the plastic strain rate is roughly 3 times higher during beam-on segments than when the beam is off. This experiment was completed at a TEM accelerating voltage of 300 kV, however, the same 'saw-tooth' trend was observed even when the voltage was lowered to 80 kV, as shown in Fig. 3(c) and (d). This indicates that even at the much lower accelerating voltage of 80 kV, the e-beam effect is still observed through an increase in plastic strain rate (increased by a factor of 1.95). Fig. 3(e) and (f) shows

Table 1

True activation volume  $V^*$  measurements for three different relaxation segments with varying e-beam condition during repeated stressrelaxation experiment for nc Al.

	$\varepsilon_p$ (%)	$V^*$ $(b^3)$
Beam-on	8.4	19
Beam-	8.6	28
off	8.8	34

the initial and final microstructure (after unloading) of the specimen tested at 300 kV, respectively. Since Fig. 3(f) is taken after unloading has occurred, there is a certain amount of specimen buckling due to plastic elongation which causes some distortion in the specimen. Despite this, it is still clear that there is localized reduction in width (necking) near the center of the gauge, directly corresponding to the region exposed to the e-beam during the beam-on segments.

In addition to monitoring the e-beam effect on monotonic behavior, a multiple stress-relaxation experiment was completed to measure the change in  $V^*$  between beam-on and beam-off conditions. During the experiment, repeated stress-relaxation segments were completed while the beam condition varied between beamon and -off and  $V^*$  was measured. The results of the experiment are summarized in Table 1, which shows  $V^*$  measurements for three consecutive relaxations segments starting at the given plastic strain values. These results show that at similar deformation (plastic strain) levels,  $V^*$  increases from 19 to 28/34  $b^3$  when the condition is changed to beam-off. This is the first time a change in  $V^*$  due to e-beam presence has been reported. A detailed description of this experiment and TEM snapshots of the deformation are provided in the Supplementary Information (SI) document.

Fig. 4 shows  $V^*$  measurements for relaxation segments of different Al specimens from beam-on and beam-off conditions. The solid green squares specifically correspond to tests done *ex situ*. Because the experiments were conducted *ex situ*, there is a certain amount of error in the  $V^*$  measurements because the device cannot be fully calibrated. This type of error is not present for *in situ* experiments as  $CS_2$  can be exactly calibrated prior to the test [32]. The beam-on and -off  $V^*$  measurements from Table 1 are indicated by the unfilled data points. The average  $V^*$  for all beam-on condition is 21.7  $b^3$ , which is consistent with the measured  $V^*$  of 19  $b^3$ in Table 1. The average  $V^*$  for all beam-off conditions is 28.6  $b^3$  is once again comparable to the values of 28 and 34  $b^3$  measured for the last beam-off relaxations in Table 1. Overall, this figure shows that there is a consistent and repeatable decrease in measured true activation volume when the specimen is exposed to the e-beam.

#### 3.2. Electron beam effects in ufg Au during in situ deformation

Additional experiments were conducted on ufg Au thin films in order to investigate how the e-beam effect varies across different materials. Specifically, the potential interaction between the e-beam and oxide layer (a possible e-beam effect for the nc Al films) can be directly investigated as Au films do not have a native oxide layer. Fig. 5 shows the response of two ufg Au specimens under monotonic loading with alternating beam-on and off conditions every 30 s (similar to that done on Al in Fig. 3). Fig. 5(a) and (c) correspond to a specimen tested under a TEM accelerating voltage of 300 kV whereas Fig. 5(b) and (d) are from a specimen tested under a lower TEM accelerating voltage of 80 kV. An accelerating voltage of 80 kV was chosen as this beam energy is well below that required for knock-on and sputtering damage in Au. The strain rates for the experiments are  $1.4 \times 10^{-4}$ (300 kV) and  $1.1 \times 10^{-4}$  s<sup>-1</sup> (80 kV). For both accelerating volt-



**Fig. 3.** Monotonic deformation of nc Al under alternating beam-off and -on conditions for TEM accelerating voltages of 300 and 80 kV. Stress-strain behavior during a monotonic test with alternating beam-on (gray) and -off (green) conditions every 30 s for experiments conducted during (a) 300 kV and (b) 80 kV TEM accelerating voltage. Significant stress-relaxation is observed during beam-on segments. Accumulated plastic strain throughout the experiment showing an increase in plastic strain rate during beam-on conditions for (c) 300 kV and (d) 80 kV accelerating volages. (e,f) Bright-field TEM images showing the (e) initial microstructure and (f) final microstructure of the specimen. Note that (f) is taken after unloading, so there is specimen buckling near the bottom of the specimen. The black box in (e) outlines the region of the specimen exposed to the e-beam during beam-on conditions (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.).



**Fig. 4.** True activation volume  $V^*$  measurements for nc Al specimens tested under beam-off (green squares) and beam-on (gray circles) conditions. The open square data points correspond to the activation volume measurements shown in Table 1 and Supplementary Information Fig. S1(c) and (e) (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.).

ages, the stress-strain response resembles the 'saw-tooth' like behavior previously seen in Al (Fig. 3), indicating that some stress decrease occurs during e-beam exposure for the Au specimens. For 300 kV, the average stress decrease during the beam-on segments is 18 MPa, with a maximum of 23 MPa. The average stress decrease for the 80 kV experiment was 16 MPa, although there were only two beam-on segments inducing stress-relaxation before specimen failure at  $\varepsilon$  = 3.7%. When considering the accumulation of plastic strain (shown in Fig. 5(c) and (d)), the plastic strain rate for beamon is  $\sim$ 1.2 times larger than that of beam-off for 300 kV and  $\sim$ 1.1 times larger for 80 kV. This is a sizeable decrease in the effect seen in Al, where the e-beam induced a three times increase in plastic strain rate. Although the effects discussed above are minimal and less drastic than those seen in Al, these results suggest that the effect of the e-beam cannot be ignored in Au, even at accelerating voltages as low as 80 kV.

Fig. 6 shows the deformation during the *in situ* test conducted using the 300 kV accelerating voltage. Fig. 6(a) and (b) are the initial and final microstructures, with the black box on both designating the region of the specimen that was repeatedly illuminated by the e-beam during the beam-on segments. Although failure of the specimen occurred outside this region, Fig. 6(b) does provide evidence that the region exposed to the e-beam experiences more necking in comparison to the regions not exposed to the e-beam (average local width within the box is 1.52  $\mu$ m compared to 1.65  $\mu$ m outside this region).

Similar to the Al specimens, V\* measurements were obtained for different Au specimens and relaxation segments for both beam-



**Fig. 5.** Stress-strain response during monotonic testing of ufg Al specimen with alternative beam-on and beam-off conditions with a TEM e-beam accelerating voltage of (a) 300 kV and (b) 80 kV. Accumulated plastic strain during the experiments conducted at TEM accelerating voltages of (c) 300 kV and (d) 80 kV. The strain rates for (a) and (b) are  $1.4 \times 10^{-4}$  and  $1.1 \times 10^{-4}$  s<sup>-1</sup>, respectively.



**Fig. 6.** TEM micrographs of the microstructural evolution of a ufg Au specimen tested under monotonic loading with alternating beam-on and beam-off conditions every 30 s. (a) Initial microstructure with black box indicating region of specimen that was repeatedly exposed to the e-beam. (b) Final microstructure after failure. The region within the black box exhibits localized reduction in width corresponding to region that was exposed to e-beam.



**Fig. 7.** True activation volume  $V^*$  measurements from different ufg Au specimens and relaxation segments for *ex situ*/beam-off (red squares) and *in situ*/beam-on (gray circles) conditions (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.).

on (*in situ*) and beam-off (*ex situ*) conditions and are shown in Fig. 7. Unlike the Al specimens, however, there is a minimal change in average activation volume  $V^*$  from beam-on (10.9  $\pm$  5.5  $b^3$ ) to beam-off (10.3  $\pm$  3  $b^3$ ) conditions. Given the scatter in the data points, these averages can be considered essentially the same. This indicates that the e-beam effect on true activation volume for Au may be negligible or too small to be measured with the precision of the current technique. This is also in agreement with only a slight increase in plastic strain rate during e-beam exposure as shown in Fig. 5.

# 4. Discussion

The above experimental results show that the e-beam influences the behavior of both nc Al and ufg Au specimens during *in situ* TEM straining. The e-beam effect is more apparent in the nc Al specimens, with the beam accelerating the plastic strain rate by a factor of three and reducing the true activation volume  $V^*$  by 25%. For the ufg Au specimens, the e-beam increases the plastic strain rate by a factor of 1.2 and the true activation volume essentially remains unchanged. In both materials, necking was observed to occur in the region illuminated by the e-beam, indicating localized deformation.

Knock-on displacement is typically considered as the most prevalent form of e-beam damage in metals. However, the experiments completed at a TEM accelerating voltage of 80 kV show the same e-beam induced stress-relaxation during monotonic loading (Fig. 5) as the experiments completed at 300 kV in both nc Al and ufg Au. At a beam accelerating voltage of 80 kV, the maximum transferable energy to Al is less than 8.93 eV, which is lower than the energy required for knock-on damage (16 eV), however, larger than the energy required for sputtering ( $\sim$ 4–8 eV) [1]. For Au, the maximum transferable energy of an 80 kV e-beam is less than 1.22 eV, which is much smaller than the required energy for bulk knock-on displacement (36 eV) or sputtering ( $\sim$ 9–18 eV) [1]. Since an e-beam effect is observed for Au tested under an 80 kV accelerating voltage, the effect cannot be explained by either bulk knock-on displacement or sputtering of the surface atoms.

Specimen heating is another common effect of e-beam interactions. The maximum expected temperature increase due to e-beam heating is calculated using the following equation for 1-dimension heat transfer,

$$\Delta T = \frac{l}{2\kappa we} \frac{\Delta E}{d} \left[ 2c - x_0 \right]$$
(5)

In Eq. (5), I is the beam current, w is the width of the specimen gauge,  $\kappa$  is the thermal conductivity of the TEM sample, *e* is electron charge, *c* is the distance to the heat sink,  $x_0$  is the beam radius, and  $\Delta E/d$  is the total energy loss per electron in a sample with thickness d. This equation is taken from Ref. [37] and modified for the 1-dimension heat transfer case, since there is no radial heat dissipation within the gauge area (see SI for full derivation). The following values were used to determine the maximum temperature change: thermal conductivity  $\kappa = 320$  (Au), 237 (Al) W/mK, w = 1700 nm,  $c = 10 \ \mu$ m,  $x_0 = 1200$  nm,  $\Delta E/d = 3.74$  (Au), 0.84 (Al) eV/nm at 80 kV and  $\Delta E/d = 2.33$  (Au), 0.50 (Al) eV/nm at 300 kV. For Al, the maximum expected temperature increase is 0.08 K at 80 kV and 0.13 K at 300 kV. The temperature increase in the Au specimens is 0.25 K at 80 kV and 0.44 K at 300 kV. Considering that there is a thin native oxide layer on Al, the calculations were also completed for alumina ( $\kappa = 30$  W/mK), which resulted in an expected temperature increase of 0.24 K at 80 kV and 0.39 K at 300 kV. For each material, the estimated temperature increase is higher at a higher accelerating voltage, which is counter to what is generally expected. In this case, the beam current *I* is different and large enough for the higher accelerating voltage (11 nA at 300 kV versus4 nA at 80 kV) which leads to a higher estimated temperature increase using Eq. (5). Based on these values, no significant heating is expected during e-beam exposure. This is also confirmed by considering that no e-beam effect is seen during the elastic portion of deformation (Figs. 3 and 5). If the specimens were heating during e-beam exposure, the increase in thermal strain would be detected before yielding occurs, which is not currently the case. Any temperature rise due to the e-beam is considered negligible.

Another possible explanation for this phenomenon is that the ebeam induces additional fluctuations of atomic motion in the two metals which effectively accelerate the stress-driven, thermallyactivated processes during their plastic deformation. Using the classical transition state theory, the plastic shear rate given by an underlying rate-controlling process is expressed as

$$\dot{\gamma}_p = \dot{\gamma}_0 \exp\left(-\frac{\Delta G}{kT}\right) \tag{6}$$

where  $\dot{\gamma}_0$  is a pre-exponential factor and  $\Delta G$  is the activation free energy of the rate-controlling process. Substituting Eq. (6) into Eq. (2), we obtain the activation volume under the beam-off condition,

$$V_{\rm OFF}^* = \sqrt{3} \frac{\Delta G_2 - \Delta G_1}{\sigma_1 - \sigma_2} = -\frac{\partial \Delta G}{\partial \tau} = V^*$$
(7)

where  $\Delta G_2$  is activation energy corresponding to the initial plastic strain rate  $\dot{\varepsilon}_{i2}$  (at stress  $\sigma_2$ ) of the second relaxation, and  $\Delta G_1$  is activation energy corresponding to the final plastic strain rate  $\dot{\varepsilon}_{f1}$  (at stress  $\sigma_1$ ) of the first relaxation segment. Under the beam-on condition, the thermal activation process is promoted by an additional effective thermal energy  $E_{\text{beam}}$  due to the beam effect

$$\dot{\gamma}_p = \dot{\gamma}_0 \exp\left(-\frac{\Delta G}{kT + E_{\text{beam}}}\right) \tag{8}$$

Similar to the activation volume under the beam-off condition in Eq. (7), we combine Eqs. (2) and (8) to obtain the activation volume under the beam-on condition,

$$V_{\rm ON}^* = \frac{1}{1 + E_{\rm beam}/kT} V^*$$
(9)

Eq. (9) indicates that due to the presence of  $E_{\text{beam}}$ , the true activation volume during e-beam exposure  $V_{\rm ON}^*$  is smaller than the activation volume V\* under no beam conditions, which is consistent with the results reported in Fig. 4 for nc Al. Using the measured activation volume in Fig. 4, we estimate the  $E_{\text{beam}}$  value for the Al specimens as  $E_{\text{beam}-Al} = 8.2 \text{meV}$  under a 300 kV e-beam. Activation volume measurements were also completed under the 80 kV e-beam condition, but there was not a measurable change in the activation volume values. This is likely because any change in activation volume values due to a decrease in TEM accelerative voltage only is below the measurable limit of the testing platform. Hence, the accelerated plastic strain rate under the beam-on condition is equivalent to an effective temperature increase  $\Delta T = 96$ K for nc Al. This is not an actual increase in temperature, but results in the same increase in plastic strain rate as a temperature increase would. Unlike a physical temperature increase of the whole specimen, the e-beam effect influences the highly localized regions around defects where the atomic bonds are weaker. For the ufg Au specimens, the true activation volume remains essentially unchanged depending on the beam condition, likely indicating that  $E_{\text{beam}}$  is very small, leading to a negligible change in  $V_{\text{ON}}^*$  versus  $V^*$ . Interpreting the beam effect in terms of additional thermal energy differs from previous interpretations of irradiation causing a decrease in activation energy for dislocation glide [23]. However, this new interpretation is necessary to explain the change in  $V^*$  measured during e-beam exposure, as a decrease in activation energy would leave V\* unchanged.

During e-beam exposure, the additional energy  $E_{\text{beam}}$  accelerates plastic deformation by introducing additional atomic fluctuations. This leads to the observed increase in ductility (Fig. 2) and accelerated plastic strain rate (Fig. 3). The effect is magnified in nc Al because the  $E_{\text{beam}}$  term, which is likely related to atomic number, is larger than that of ufg Au, although an exact  $E_{\text{beam}}$  term could not be determined for Au since the effect on activation volume is too small to be measured. The results presented here are similar to previous reports of e-beam induced stress relaxation in Al and Au via increased dislocation activation and depinning [22]. It is possible that the additional atomic fluctuations provided by

the beam can lead to dislocation activation and depinning, which underlie the apparent increase of plastic strain rate. It is unlikely the depinning occurs due to a displacement cascade effect (as previously reported for ion irradiation [24]) since electron irradiation does not cause a displacement cascade. Instead, the e-beam effects the highly localized regions around defects. It is also unlikely that the observed effect is due to the local climb of dislocations aided by absorption of point defects, as minimal point defects are expected in the Au specimens (with higher knock-on and sputtering energies) but the e-beam influence is still observed. However, it is possible that the atoms near dislocations and GBs experience subthreshold displacements as the periodic lattice is disrupted and the energy required to displace these atoms is reduced [16]. This could contribute to the observed e-beam effect, as this could induce additional fluctuations in atomic motion and aid in dislocation glide and GB migration.

There is no evidence in the present study that the deformation mechanisms themselves change due to e-beam exposure; rather the mechanisms are accelerated with e-beam exposure. The post mortem fracture surface and surrounding microstructure of the nc Al specimen tested at a slow strain rate (Fig. 2(e) and (f)) beamoff condition resembles that of the beam-on condition at a faster strain rate (Fig. 2(d)). Both specimens exhibit a necked region with prevalent grain growth, which suggests that the deformation mechanisms may not have changed due to the influence of the e-beam, however, this cannot be proved exactly as the deformation cannot be documented in the beam-off condition. This indicates that the measured change in true activation volume, which is a parameter typically associated with a rate-controlling deformation mechanism, is not a result of a change in deformation mechanisms, but rather the addition of effective thermal energy. Other researchers have reported a similar decrease in apparent activation volume (12 to 4  $b^3$ ) under e-beam exposure for Al-4Cu alloys, however, they attributed the decrease to a change in deformation mechanisms from internal dislocation nucleation to surface dominated dislocation nucleation processes [21]. They claim that the ebeam causes local disorder between the native oxide layer and the Al-4Cu alloy which promotes dislocation nucleation at the surface. As the e-beam influence on mechanical properties was demonstrated in the Au thin films, oxide layer effect alone cannot explain the observed behavior. In addition, the slow strain rate experiment on the nc Al specimens suggests that the underlying deformation mechanisms do not change in response to the e-beam. Instead, the experiments conducted in this study suggest that the change in true activation volume is due to the increase in atomic fluctuations by an additional effective thermal energy. This accelerates the thermally-activated deformation mechanism present in these materials, such as grain boundary migration, dislocation glide, and dislocation nucleation/emission from grain boundaries.

## 5. Conclusion

We investigated the effect of the e-beam on the deformation behavior of nc Al and ufg Au thin films using an *in situ* TEM nanomechanical testing technique that allows for stress-strain and true activation volume measurements. The effect of the e-beam was quantified as an increase in plastic strain rate in both materials, and a decrease in true activation volume from 28 to 21  $b^3$ in Al and a negligible change of true activation volume in Au. The e-beam effects were seen at TEM beam accelerating voltages of 80 and 300 kV, discounting knock-on damage as the source e-beam influence. Instead, the experiments suggest that the e-beam causes additional thermal activation that accelerates the stress-driven, thermally-activation plastic deformation. The additional thermal activation eases the barrier to plastic deformation and leads to the change in the mechanical properties across the beam-on and -off conditions, but does not change the active deformation mechanisms themselves. These results show that there is non-negligible e-beam effect in different materials that must be considered for accurate interpretation of *in situ* experiment results. The reported e-beam effects should be broadly applicable to other nc/ufg facecentered cubic metals and warrant further in-depth study in the future.

# **Declaration of Competing Interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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#### Supplementary materials

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