Graphical abstract

Variation of mean contact pressure p_m and dislocation length λ as a function of indentation depth h for small and large bicrystal samples with $\Sigma 3 (111)$ GB and their related single crystal samples during nanoindentation.

single crystal large sample, $h \approx 11.5$ nm

bi-crystal large sample, $h \approx 11.5$ nm

Dislocation nucleation and evolution for large single crystal and bi-crystal samples at higher indentation depths: GB changes the pattern of dislocation structure in a way that the dislocations are piled up near the GB which increases the hardness.

Effects of grain boundary on the sources of size effects

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Abstract

The present paper investigates the effects of grain boundary (GB) on the sources of size effects. Up to now, several studies have been conducted to address the role of GBs in size effects from the atomistic point of view. However, a study which addresses the effects of GB on different governing mechanisms of size effects as the sample length scale varies has not been presented yet. Here, samples with different length scales are studied to capture the role of GB in size effects as the grain size changes. The response of single and bi-crystal Ni thin films with two different sizes are studied during nanoindentation experiment using large scale atomistic simulation. Various symmetric and asymmetric tilt GBs are incorporated to study the effects of GB geometry on the response of samples during nanoindentation. The sources of size effects are analyzed in each sample using the atomistic information obtained from the simulations. The results show that the size effects mechanism influenced by GBs changes from dislocation nucleation and source exhaustion to the forest hardening mechanism as the grain size increases. In the case of small bi-crystal samples, dislocation nucleation and source exhaustion govern the size effects. The GB contributes to the dislocation nucleation beneath the indenter which reduces the strength of sample by providing required dislocations to sustain the imposed plastic deformation. Also, the GB itself is the source of defects which can affect the sample strength depending on the indentation depth at which the dislocation is nucleated from the grain boundary. Increasing the indentation depth, some of the dislocations are blocked by the GB. However, there is no noticeable additional hardness due to the dislocations blockage by GB. Furthermore, the results show that the coherent twin boundaries shows the best performance during the nanoindentation. In the case of large bi-crystal samples, the GB does not influence the size effects at lower indentation depths where the source exhaustion is the controlling mechanism of size effects. However, at higher indentation depths, the dislocation interaction with GB contributes to the forest hardening mechanism and induces some hardening. It is observed that the dislocations are firstly absorbed by the GB. Increasing the indentation depth, some dislocations start dissociating into the next grain. However,

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it is observed that more dislocations are nucleated in the upper grain. The results show that the main role of GB at larger length scale is to change the pattern of dislocation structure in a way that the dislocations are piled up near the GB which increases the hardness.

Keywords: Grain boundary; Size effects; Molecular dynamics; Dislocation; Microstructures

1. Introduction

The mechanical properties of crystalline metals are usually governed by grain size and grain boundary (GB) properties [1-3]. The GB effects on the strength depend on several factors including the grain size, GBs geometry, mechanical and crystallographical structure of metal, strain rate, and temperature [1-3]. These factors define the deformation mechanisms in crystalline metals. Several numerical and experimental studies have been conducted to unravel the role of GBs in crystalline metals [1-3]. In the case of large grains, i.e. grain sizes of more than 1 μm , the strength increases as the grain size decreases which is commonly described by the Hall-Petch relation. The enhancement in strength is justified using the dislocation pile-up mechanism [1-3]. By decreasing the grain size, the pile-up model and consequently the Hall-Petch relationship break down at some grain size which is of the order of nanometers [4]. Other mechanisms such as GB sliding, GB rotation, and GB dislocation creation and annihilation have been proposed to describe the grain size dependency of strength in nanocrystalline metals [1-3].

Molecular dynamics (MD) simulation is a powerful tool [5-8] to model the effects of grain size and GB properties on the strength of nanocrystalline metals from the atomistic point of view. Van Swygenhoven et al. [9,10] incorporated the atomistic simulation and showed that below a critical grain size, the GB sliding is a dominant mechanism using molecular dynamics. In the cases of grains larger than 10-12 nm, they observed some intra-grain deformation mechanism such as partial dislocations [9,10]. Yamakov and coworkers [11-14] also conducted several atomistic simulation to study the effect of grain size on the deformation mechanisms of nanocrystalline fcc metals. It was observed that as the grain size decreases, the deformation process changes from a dislocation-based to a grain-boundary-based mechanism [11-14]. Derlet et al. [15] summarized the limitation of MD in the case of nanocrystalline metals simulation. They stated that the atomic deformation mechanisms can be successfully extracted from the MD simulation [15]. However, when it comes to the extrapolation of the obtained MD results to the experimental observations, one should consider the temperature, size, and rate at which the simulation is conducted [15].

The interaction of the dislocations with GBs has been investigated using atomistic simulation to understand the underlying deformation process. De Koning et al. [16] performed 2D

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4 and 3D MD simulations to study the various mechanisms of dislocation absorption, transmission,
5 and reflection by incorporating both symmetric and asymmetric $\Sigma 11$ tilt GBs. Hasnaoui et al. [17]
6 simulated the nanoindentation experiment using MD simulation to study the interaction between
7 dislocations and GBs. It was observed that the GBs can act as a sink for dislocations, reflect or emit
8 dislocations [17]. Jang and Farkas [18] incorporated MD simulation and studied a bi-crystal nickel
9 thin film with $\Sigma 5 (2\ 1\ 0) [0\ 0\ 1]$ GB during the nanoindentation. It was shown that the GB induced
10 some resistance to the indentation due to the stacking fault expansion [18]. Kulkarni et al. [19]
11 compared the response of coherent twin boundary (CTB) with that of $\Sigma 9 (2\ 2\ 1)$ tilt GB during
12 nanoindentation using atomistic simulation. They stated that unlike the $\Sigma 9 (2\ 2\ 1)$ GB, the CTB
13 does not considerably reduce the strength of the sample. However, unlike Jang and Farkas [19],
14 they did not observe noticeable enhancement in strength. Tsuru et al. [20] studied the effects of
15 coincidence site lattice (CSL) grain-boundaries on the incipient plasticity using MD. They changed
16 the distance of indenter with respect to the GB and monitored the incipient plasticity. They
17 concluded that the GBs with lower energies have higher mean pressures at the onset of plasticity.
18 Stukowski et al. [21] compared the uniaxial response of nanotwinned Cu and Pd nanocrystalline
19 samples with those without twin boundaries using large scale atomistic simulation. They showed
20 that the effects of twin boundaries depend on the unstable stacking fault and twin boundary
21 migration energies, and it can increase or decrease the strength of the material [21]. Sangid et al.
22 [22] measured the energy barriers of CSL grain boundaries of slip-GB interactions using MD. It was
23 observed that the energy barrier has an inverse relationship with GB energy in a way that the GB
24 with lower energy has the higher energy barrier [22]. Zhang et al. [23] incorporated the atomistic
25 simulation to investigate the response of symmetric and asymmetric $\Sigma 5$ GBs during uniaxial
26 tension experiment. They studied the effect of inclination angles on the mechanical response of
27 copper bi-crystal [23]. Sainath and Choudhary [24] simulated the Cu nanopillars with and without
28 twin boundaries under uniaxial tension using MD. It was shown that the GB changes the
29 deformation mechanism and increase the strength of the simulated nanopillar [24].

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49 The interaction of dislocations with each other and with GBs governs the size effects in bulk
50 metals which is termed as the forest hardening [25,26]. Decreasing the sample size to the order of
51 nanometers, however, the forest hardening model is not the governing mechanism. The size effects
52 in nanoscale metallic samples are described using three models of source exhaustion hardening,
53 weakest link theory, and source truncation [26-29]. Dislocation starvation, mechanical annealing,
54 and source shut down reduce the number of sources in nanoscale samples in a way that the
55 reduced mobile dislocation density is insufficient to handle the imposed deformation.
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Consequently, the applied stress should be increased to sustain the plastic deformation. The induced hardening due to the lack of sources is termed exhaustion hardening [30,31]. In the case of metallic sample of confined volumes, since the double-ended dislocation sources are close to the free surfaces, they transform into the single-ended sources. Consequently, the strength is increased due to the reduction in characteristic length of dislocation sources which is commonly termed as source truncation [32,33]. The last mechanism is the weakest link theory in which the strength of the weakest slip plane increases by reducing the sample size which enhances the strength [34,35]. Tucker et al. [36] investigated the effects of GBs on the size effects of nanowires under uniaxial compression using MD. It was shown that both GB and free surfaces can be the favorable dislocation nucleation sites [36]. Recently, Yaghoobi and Voyiadjis [37] studied the sources of size effects in nanosize single crystal Ni thin films during nanoindentation using large scale atomistic simulation. They showed that the dislocation nucleation and source exhaustion are mainly responsible for size effects during the nanoindentation of Ni samples of confined volumes at lower indentation depths [37].

In the current study, the effects of GBs on the sources of size effects are investigated. The nanoindentation response of single and bi-crystal Ni thin films are investigated by incorporating large scale atomistic simulation. Here, samples with two different sizes and various symmetric and asymmetric tilt GBs are studied to capture the role of GB in size effects as the grain size changes. Furthermore, the sources of size effects are analyzed in each sample using the microstructural information obtained from the simulations. In order to study the size effects controlling mechanisms, the total dislocation length of each sample is studied during the nanoindentation. The obtained results are then compared to the variation of hardness versus the indentation depth to unravel the contribution of GBs to the sources of size effects as the sample length scale changes. The dislocation visualization is also incorporated to study the dislocation nucleation and evolution patterns for bi-crystal and single crystal samples of different sizes.

2. Simulation details and methodology

To investigate the effects of grain size on the sources of size effects, two different sizes of Ni thin films with the dimensions of $24 \text{ nm} \times 24 \text{ nm} \times 12 \text{ nm}$ (S1) and $120 \text{ nm} \times 120 \text{ nm} \times 60 \text{ nm}$ (S2) are generated and simulated using the classical molecular dynamics. The total number of atoms in the samples are around 650,000 for S1 and 79,000,000 for S2. The periodic boundary conditions are applied to the surrounding surfaces. The free boundary conditions are selected at the bottom of the sample. The selected boundary conditions ensure that the dislocations do not

bounce back from the sample bottom. To prevent the whole domain from translational movement, at each step, the total force is divided by the number of atoms and then applied in the opposite direction to all atoms [38-41]. Voyiadjis and his coworkers [37,40,41] has previously shown that the chosen boundary conditions type can successfully capture the nanoindentation response and microstructural information such as dislocation structure and length. The velocity Verlet algorithm with a time step of 2 fs is selected. The parallel MD code LAMMPS is used [42]. Seven different types of GB including four symmetric tilt boundaries of $\Sigma 3 (1\ 1\ 1) [1\ \bar{1}\ 0]$ ($\theta = 109.5^\circ$), $\Sigma 11 (1\ 1\ 3) [1\ \bar{1}\ 0]$ ($\theta = 50.5^\circ$), $\Sigma 3 (1\ 1\ 2) [1\ \bar{1}\ 0]$ ($\theta = 70.5^\circ$), and $\Sigma 11 (3\ 3\ 2) [1\ \bar{1}\ 0]$ ($\theta = 129.5^\circ$), and three assymetric tilt boundaries of $\Sigma 11 (2\ 2\ 5)/(4\ 4\ 1)$ ($\varphi = 54.74^\circ$), $\Sigma 3 (1\ 1\ 2)/(\bar{5}\ \bar{5}\ 2)$ ($\varphi = 19.47^\circ$), and $\Sigma 3 (1\ 1\ 4)/(1\ 1\ 0)$ ($\varphi = 35.26^\circ$) are selected to study the effects of GB properties on the size effects, where θ and φ are the interface misorientation and inclination angles, respectively. The GBs are located at a third of the thickness from the top surface. For each bi-crystal sample, one single crystal sample is also generated which has the atomic structure similar to that of the upper grain. Two spherical indenters with the radius of $R_1 = 10$ nm and $R_2 = 15$ nm are selected to perform indentation on S1 and S2 samples, respectively. The CSL grain boundaries are created by a symmetric tilt rotation θ of opposing lattice regions around a misorientation axis $[1\ \bar{1}\ 0]$. Asymmetric tilt GBs are obtained from symmetric tilt boundaries of the same misorientation angle by incorporating the inclination angle φ , which is the deviation of the GB plane from the selected symmetrical position. The final configuration is obtained using molecular statics [42]. Energy minimization is attained using the conjugate gradient algorithm [42]. In order to reach the minimum energy configuration of GBs, several in-plane rigid body translations are conducted in which the top and bottom grains are rigidly translated with respect to each other. Next, the samples are relaxed for 100 ps with the temperature increasing from 1 K to 300 K. Finally, they are relaxed for 100 ps at 300 K. The GBs equilibrium structures are presented in Fig. 1 using the Centrosymmetry parameter [43]. The software OVITO [44] is used to visualize the equilibrium structures. In Fig. 1, the vertical direction of the upper grain is normal to the upper free surface of each sample.

Finally, the relaxed samples are indented along the normal to the upper free surface with the velocity of 10 m/s, which has been widely used in previous simulations [37,40,41,45,46,47], at 300 K. The simulation is performed using NPT ensemble [42]. The nickel embedded-atom method (EAM) potential developed by Mishin et al. [48] is incorporated to simulate the atomic interaction of Ni-Ni [49]. A repulsive potential is selected here to describe the interaction between the indenter and Ni atoms, which is described as follows [42]:

$$E^{\text{ind}}(r) = \varepsilon(r - r_c)^2 \quad r < r_c \quad (1)$$

where ε is the specified force constant which is chosen as $1 \text{ eV}/\text{\AA}^2$, r is the distance from particle to the indenter surface, and r_c is the cutoff distance which is selected as 0.3 nm. To calculate the contact area A , first, atoms located in direct contact with the indenter are identified. Next, a triangular 2D-mesh is created from the projections of those atoms onto the indentation surface. The contact area is the summation of all triangle areas. The mean contact pressure p_m can be obtained by deviding the indentation load P by contact area A . In the case of the spherical tip, true indentation depth h , which is different from the displacement of the indenter tip d , can be obtained from geometrical relations [40]. Here, the indentation depth of a spherical tip is calculated by incorporating the contact area as follows:

$$h = R - \sqrt{R^2 - a_c^2} \quad (2)$$

where $a_c = \sqrt{A/\pi}$ is the contact radius and R is the spherical tip radius.

The Crystal Analysis Tool developed by Stukowski and his co-workers [50-53] is incorporated to visualize the dislocations and provide additional information such as dislocation length and Burgers vector. One can obtain all the required dislocation properties from the MD simulation output using the Crystal Analysis Tool which, to the best of authors' knowledge, is the only open source post processing tool providing such information. First, atomic arrangements and structures are identified. Next, a pattern matching algorithm groups atoms into so-called clusters. A Delaunay mesh is generated that connects all atoms and the corresponding elastic deformation gradient tensor is computed for the constructed mesh. If a dislocation intersects a tessellation element, the elastic deformation gradient is multi-valued. In the next step, trial circuits are built on the interface mesh to define the dislocation lines. Finally, the code provides a one-dimensional line representation of the dislocation segments. The software OVITO [44] and Paraview [54] are used to visualize the dislocations and process the microstructural information such as dislocation length and density. As described by Stukowski et al. [21], GBs may be detected as small angle GBs. However, the immobile GB dislocations should be excluded from the total length of lattice dislocations. Accordingly, the dislocations located in a 1 nm wide region on each side of the GB planes are not considered.

3. Results and Discussions

Fig. 2 presents the variation of the mean contact pressure ($p_m = P/A$), which is equivalent to the hardness H in the plastic region, as a function of indentation depth h for S1 samples. In the

cases of S1 samples, the results show that the GB mostly reduces the strength. However, in the case of $\Sigma 3 (111) [1\bar{1}0]$, i.e. coherent twin boundary (CTB), the GB slightly increases the strength at some ranges of indentation depth. Furthermore, the CTB has the least reduction in the strength compared to those of the other GBs. Same results has been reported by Kulkarni et al. [19] during nanoindentation using MD simulation. In order to unravel the governing mechanisms of strength reduction in S1 samples, the variations of dislocation length λ and mean contact pressure p_m versus the indentation depth h should be studied. As an example, Fig. 3 illustrates the variation of p_m and λ versus the h in the case of $\Sigma 3 (111) [1\bar{1}0]$ GB. The size effects can be studied in five different regions of indentation depths:

- *Region I:* Here, the only defect existing in bi-crystal sample is the $\Sigma 3 (111) [1\bar{1}0]$ GB. In the case of single crystal, the sample is defect-free. The response in this region is elastic for both bi-crystal and single crystal samples. There is no difference between the single crystal and bi-crystal samples responses in this region.
- *Region II:* In the case of bi-crystal sample, the initial dislocation nucleation occurred beneath the indenter at lower indentation depth compared to the single crystal sample, which was comprehensively studied by Tsuru et al. [20]. The size effects in the bi-crystal sample is initially controlled by dislocation nucleation and source exhaustion. Here, the resulting mobile dislocation density is increased by increasing the total dislocation length. Consequently, the required applied stress to sustain the plastic deformation decreases. In other words, the dislocations created in the bicrystal sample reduce the strength. In the case of single crystal, the sample is still defect-free and the response remains elastic.
- *Region III:* In the case of single crystal sample, the first dislocation is nucleated beneath the indenter which induces a large drop in hardness. Again, dislocation nucleation and source exhaustion mechanisms initially govern the size effects. In the case of the bi-crystal sample, the dislocation length remains nearly constant. According to the source exhaustion mechanism, the applied stress should increase to sustain the plastic flow which increases the strength. The dislocations are stopped by the GB from moving downward. However, it does not lead to any additional strength since the number of dislocations blocked by the GB is not noticeable. The fact that the dislocation blockage mechanism is available in the next regions but does not lead to any significant size effects supports this argument.
- *Region IV:* According to the source exhaustion mechanism, increasing the mobile dislocation density reduces the strength which justifies the strength reduction in both single crystal and bi-crystal samples. In *Regions III* and *IV*, the dominance of source

exhaustion mechanism decreases by increasing the total dislocation length in the case of single crystal sample. Consequently, the slope of hardness reduction decreases as the total dislocation length increases which is obvious in *Region IV*. The same trend can be observed for the bi-crystal sample in *Regions IV* and *V*. The GB still blocks the dislocations moving downward. Eventually, the dislocations start dissociating into the next grain.

- *Region V*: Increasing the indentation depth, the dislocation interaction with each other becomes important which activates the forest hardening mechanism. Also, the available dislocation length is enough to sustain the applied plastic flow. Consequently, the source exhaustion mechanism is not active anymore. As described by Swadener et al. [55] and Yaghoobi and Voyiadjis [37], in this region, the hardness should become nearly constant. The GB still blocks some part of dislocations. However, it does not influence the size effects, and single crystal and bi-crystal samples reach the same hardness.

Fig. 4 presents the dislocation structure in different regions for both single crystal and bi-crystal samples in the case of $\Sigma 3$ (1 1 1) $[1 \bar{1} 0]$ GB. Fig. 4 (a) shows that the single crystal sample in *Region II* is defect-free. The dislocation structure of the bi-crystal sample in *Region II* ($h \approx 0.6$ nm) is depicted in Fig. 4 (b) in which the dislocations are nucleated beneath the indenter. Fig. 4 (b) shows that the dislocation multiplication mechanism is cross-slip which increases the number of pinning points. The dislocations which are pinned at their ends are elongated and provide the required mobile dislocation to sustain the plastic flow. Fig. 4 (c) illustrates the cross-slip and dislocation elongation in the case of single crystal sample in *Region III* ($h \approx 0.9$ nm). The dislocations blockage by GB are depicted in Fig. 4(d). Figs. 4 (e) and (f) demonstrates the dislocation structure of single crystal and bi-crystal samples, respectively, in *Region IV*. First dislocation which is emitted into the next grain is a Shockley partial dislocation with the Burgers vector of $\frac{1}{6}[\bar{1} \bar{2} \bar{1}]$ which is shown in Fig. 4 (f). Figs 4 (g) and (h) show the dislocation network beneath the indenter in single crystal and bi-crystal samples, respectively, in which there is enough dislocation content to sustain the applied plastic flow, and the dislocation interaction with each other becomes important (*Region V*).

The variation of p_m and λ versus the indentation depth for all bi-crystal and their related single crystal samples, except the sample with $\Sigma 3$ (1 1 1) $[1 \bar{1} 0]$ GB, are presented in Fig. 5. The results show that, in contrast to the $\Sigma 3$ (1 1 1) $[1 \bar{1} 0]$ bicrystal and related single crystal samples, the sudden drop in strength does not simultaneously occur with the nucleation of the first dislocation. In the case of single crystal samples, Fig. 5 shows that the large drop in strength occurs when a sudden jump happens in the total dislocation length. In the case of bi-crystal samples, since

the dislocations interact with the GB, the nature of sudden strength reduction becomes complicated. Moreover, Fig. 5 shows that the drop in hardness occurs at lower indentation depths in the cases of bi-crystal samples compared to that observed in the single crystal samples. At the moment of sudden drop in hardness of bi-crystal samples, they have larger total dislocation lengths compared to those of the single crystal samples which is reasonable based on the source exhaustion mechanism. Also, the grain boundary itself can be a source of dislocation nucleation in the initial phases of indentation. Although all GBs become the source of defects, it happens at different stages of indentation which leads to the different effects. As an example, Fig. 6 shows the initial dislocation nucleation in bi-crystal samples with GBs of $\Sigma 3 (112)/(\bar{5}\bar{5}2)$ and $\Sigma 11 (225)/(441)$. The results show that the initial dislocations are nucleated from the GB which are the Shockley partial dislocations with the Burgers vectors of $\frac{1}{6}[11\bar{2}]$ and $\frac{1}{6}[112]$ in the cases of $\Sigma 3 (112)/(\bar{5}\bar{5}2)$ and $\Sigma 11 (225)/(441)$ GBs, respectively. In other words, the initial dislocation nucleation does not occur beneath the indenter. Since the dislocation nucleation from the $\Sigma 3 (112)/(\bar{5}\bar{5}2)$ and $\Sigma 11 (225)/(441)$ GBs occurs at the very initial phases of defect nucleation, it severely affects the strength and eliminates the local hardening occurred in the other bi-crystal samples after the sudden drop in hardness, as shown in Figs. 5 (d) and (e). One can unravel the variation of hardness versus the indentation depth for all samples using the microstructural information including the total dislocation length and dislocation visualization. Generally, the results show that the size effects is initially controlled by the source exhaustion mechanism which is affected by GB in the cases of S1 samples. Eventually, the total dislocation length required to sustain the plastic flow is provided, and the source exhaustion mechanism becomes inactive. On the other hand, the GB does not have any considerable effect on the forest hardening mechanism. In other words, the interaction between dislocation and GB does not induce any noticeable hardening at this length scale for any of the simulated S1 samples.

Fig. 7 presents the variation of the mean contact pressure ($p_m = P/A$), which is equivalent to the hardness H in the plastic region, as a function of indentation depth h for the S2 samples. The results show that the nanoindentation responses of the bi-crystal and their related single crystal samples are nearly similar at the lower indentation depths. However, the bi-crystal samples show larger hardness at higher indentation depths. The variation of p_m and λ versus h should be investigated to understand the mechanisms leading to the observed behavior. As an example, Fig. 8 illustrates the variation of p_m and λ versus h for $\Sigma 3 (111) [1\bar{1}0]$ bi-crystal and its related single crystal S2 samples. The results show that the size effects can be divided in three regions as follows:

- *Region I*: The response in this region is elastic for both bi-crystal and single crystal samples. Besides the GB, there are no other defects in both bi-crystal and single crystal samples.
- *Region II*: The initial dislocation nucleation occurred beneath the indenter. A large drop in hardness occurs immediately after the first dislocation nucleation for both bi-crystal and single crystal samples. The size effects in bi-crystal and single crystal samples are controlled by dislocation nucleation and source exhaustion. As the total dislocation density increases, the resulting mobile dislocation density also increases which leads to the lower hardness according to the source exhaustion mechanism. In *Region II*, the difference between the total dislocation length of the bi-crystal and single crystal samples is small which leads to the nearly similar hardness for the bi-crystal and single crystal samples according to the source exhaustion mechanism. Increasing the indentation depth, the GB starts blocking the movement of dislocations. However, the source exhaustion mechanism is still the controlling mechanism of size effects and the hardness decreases as the total dislocation length increases. The slope of hardness reduction decreases by increasing the indentation depth which means that the dominance of source exhaustion mechanism decreases.
- *Region III*: As the total dislocation length increases, the source exhaustion mechanism becomes inactive in this region due to the fact that enough dislocation length is available to sustain the applied plastic flow. On the other hand, the forest hardening mechanism gradually becomes activated as the total dislocation length increases. The total dislocation length of the bi-crystal sample starts deviating from that of the single crystal sample, and it has larger values. Furthermore, the total length of dislocation blocked by the GB becomes noticeable. In *Region III*, the hardness in the bi-crystal samples becomes larger than that of the single crystal sample.

Fig. 9 illustrates the dislocation nucleation and evolution for $\Sigma 3$ (1 1 1) $[\bar{1} \bar{1} 0]$ bi-crystal and its related single crystal S2 samples. Figs. 9 (a) and (b) show that the initial dislocation nucleation process is homogeneous which is similar for both single crystal and bi-crystal samples. The initial dislocation is a Shockley partial dislocation with the Burgers vector of $\frac{1}{6}[\bar{2} \bar{1} 1]$. Figs. 9 (c) and (d) show that the dislocations are multiplied using the cross slip mechanism, and the dislocations pinned at their ends are elongated to sustain the applied plastic flow. Again, the dislocation structure for both single crystal and bi-crystal looks similar at this stage. Increasing the indentation depth, the dislocation loops are emitted by cross-slipping and pinching off of screw dislocations. In the case of the single crystal sample, Fig. 9 (e) shows that the dislocation loop is moving downward and reaches the sample bottom. In the case of the bi-crystal sample, however,

Fig. 9 (f) shows that the dislocation loops are blocked by the GB. Eventually, the dislocations are emitted to the lower grain. The first emitted dislocation into the next grain is a Shockley partial dislocation with the Burgers vector of $\frac{1}{6}[1\bar{2}1]$ which is depicted in Fig. 9 (h). The dislocation structure of the single crystal and bi-crystal samples at higher indentation depths ($h \approx 11.5$ nm) are presented in Figs. 9 (i) and (j), respectively. The results show that in the case of bi-crystal, although part of the dislocation are emitted to the next grain, most of the dislocations are located in the upper grain which are blocked by the GB. In the case of single crystal sample, however, there is no obstacle for the dislocation movement and they are freely moving downward.

Fig. 10 shows the variation of p_m and λ versus h for all bi-crystal and their related single crystal samples except the sample with $\Sigma 3(111)[1\bar{1}0]$ GB. The mechanisms described for $\Sigma 3(111)[1\bar{1}0]$ GB are applicable to all cases except the samples with the GBs of $\Sigma 11(332)$ and $\Sigma 11(225)/(441)$ in which the total dislocation lengths of bicrystal samples are nearly similar to those of the related single crystal samples. However, the hardness of bi-crystal samples are larger at higher indentation depths. In the case of source hardening mechanism, the total dislocation length of the thin film is an appropriate representative parameter. However, in the case of forest hardening mechanism, the dislocation density around the indenter is a controlling factor and not the total dislocation length. Here, the plastic zone is assumed to be confined in the upper grain. In other words, the dislocations which are effective in the forest hardening are the ones located in the plastic zone which can be approximately considered as upper grain. Hence, in order to study the size effects due to the forest hardening, the total dislocation length located in the upper grain λ_{upper} of the bi-crystal sample is more appropriate. In the case of single crystal samples, the dislocations located in the upper one third of the thickness, which is similar to the location of the upper grain, are considered. Fig. 11 shows the variation of p_m and λ_{upper} versus h for all bi-crystal and their related single crystal samples. It shows that, the λ_{upper} is larger for all bi-crystal samples at higher indentation depths compared to those of the single crystal samples which leads to the larger hardness according to the forest hardening mechanism. Although some of the dislocations start dissociating to the next grain, the results show that more dislocations are nucleated in the upper grain. Figs. 10 and 11 show that the GBs main role is to change the pattern of dislocation structure in a way that the dislocations are piled up near the GB which increases the hardness.

4. Conclusions

The present work investigates the coupling effects of sample length scale and grain boundary on the sources of size effects using large scale atomistic simulation. Two different sizes of

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4 Ni thin films are incorporated to study the effects of sample length scale. Seven different types of
5 symmetric and asymmetric tilt boundaries are selected to investigate the effects of GB geometry on
6 the size effects. In the case of single crystal samples, the size effects are initially governed by
7 dislocation nucleation and source exhaustion mechanisms. Therefore, increasing the total content
8 of dislocations leads to the hardness reduction because more dislocations are available to sustain
9 the plastic flow. However, the dominancy of source exhaustion hardening decreases as the
10 indentation depth and consequently the total dislocation length increase. Hence, the slope of
11 hardness reduction decreases as the indentation depths increases. Eventually, the source
12 exhaustion mechanism becomes inactive when enough dislocation content is available to sustain
13 the applied plastic flow. On the other hand, the forest hardening mechanism, which is originated
14 from dislocation interactions with each other, becomes more dominant as the total dislocation
15 length increases. Finally, at higher indentation depths, the forest hardening is the only governing
16 mechanism.

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18 The effects of GB on the sources of size effects depend on the sample length scale. GB does
19 not change the general pattern of size effects; however, it can contribute to some specific
20 mechanisms depending on the sample size. The size effects is initially controlled by source
21 exhaustion mechanism during the nanoindentation which is influenced by GB in the cases of small
22 samples. The initial strength drop occurs earlier in small bi-crystal samples compared to their
23 related single crystal samples. It may be originated from the fact the total dislocation lengths of bi-
24 crystal samples are larger than those of the related single crystals at the moment of strength drop
25 which ease the sudden hardness reduction according to the source exhaustion mechanism. Also, the
26 grain boundary itself is a source of dislocation nucleation. However, the effects of a GB, as a
27 nucleation source, on the hardness of a thin film depend on the depth at which the dislocations start
28 nucleating from GB. If the dislocation nucleation occurs at the very initial phases of indentation, it
29 can severely affect the strength. The forest hardening mechanism becomes dominant at larger
30 indentation depths. In the cases of small samples, however, the responses of bi-crystal and single-
31 crystal samples are similar in this region. It shows that the GB does not change the nanoindentation
32 response at larger indentation depths when the source exhaustion mechanism becomes inactive
33 and forest hardening is dominant. It is worth mentioning that the dislocations movement are
34 blocked by GBs during nanoindentation in small bi-crystal samples. However, the density of piled-
35 up dislocation is not enough to change the nanoindentation response. In the cases of small samples,
36 the best performance between all GBs is demonstrated by coherent twin boundary which has the

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4 least strength reduction. Also, it shows small enhancement in hardness at some ranges of
5 indentation depth.
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8 In the cases of large samples, the total dislocation content is much greater than that of the
9 small samples. Hence, the interaction of dislocations and GBs plays a key role. The initial
10 nanoindentation responses of bi-crystal and single crystal samples are similar in the cases of large
11 samples. It is due to the fact that the total dislocation length of the bi-crystal sample is close to that
12 of the single crystal sample. It means that the GB does not have any noticeable effect on the source
13 exhaustion mechanism. The dislocations moving downward are blocked by the GB. At higher
14 indentation depths, the strength of bi-crystal samples becomes larger than that of the single crystal
15 samples. The total dislocation lengths are large at higher indentation depths, and consequently the
16 density of dislocations piled-up behind the GB becomes noticeable. Hence, the increase in bicrystal
17 samples can be justified according to the forest hardening mechanism. Moreover, the results show
18 that the total dislocation length in the upper grain is a better representative factor to investigate the
19 strength size effects. In other words, according to the forest hardening mechanism, the dislocations
20 in the plastic zone which are closer to the indenter are the effective ones. Hence, in the cases of
21 large samples, the most important role of GBs is to change the pattern of dislocation structure by
22 blocking their movement which increases the resulting hardness at higher indentation depths.
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45 46 **References**

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Figure Captions

Fig. 1. The equilibrium structure of the symmetric and assymetric tilt grain boundaries of (a) $\Sigma 3 (1\ 1\ 1) [1\ \bar{1}\ 0]$ ($\theta = 109.5^\circ$), (b) $\Sigma 11 (1\ 1\ 3) [1\ \bar{1}\ 0]$ ($\theta = 50.5^\circ$), (c) $\Sigma 3 (1\ 1\ 2) [1\ \bar{1}\ 0]$ ($\theta = 70.5^\circ$), (d) $\Sigma 11 (3\ 3\ 2) [1\ \bar{1}\ 0]$ ($\theta = 129.5^\circ$), (e) $\Sigma 11 (2\ 2\ 5)/(4\ 4\ 1)$ ($\varphi = 54.74^\circ$), (f) $\Sigma 3 (1\ 1\ 2)/(\bar{5}\ \bar{5}\ 2)$ ($\varphi = 19.47^\circ$), and (g) $\Sigma 3 (1\ 1\ 4)/(1\ 1\ 0)$ ($\varphi = 35.26^\circ$), along $[1\ \bar{1}\ 0]$ axis.

Fig. 2. Variation of mean contact pressure p_m as a function of indentation depth h for S1 single crystal and their related bi-crystal samples with the grain boundaries of (a) $\Sigma 3 (1\ 1\ 1)$ (b) $\Sigma 11 (1\ 1\ 3)$ (c) $\Sigma 3 (1\ 1\ 2)$ (d) $\Sigma 11 (3\ 3\ 2)$ (e) $\Sigma 11 (2\ 2\ 5)/(4\ 4\ 1)$ (f) $\Sigma 3 (1\ 1\ 2)/(\bar{5}\ \bar{5}\ 2)$ (g) $\Sigma 3 (1\ 1\ 4)/(1\ 1\ 0)$.

Fig. 3. Variation of mean contact pressure p_m and dislocation length λ as a function of indentation depth h for S1 bicrystal sample with $\Sigma 3 (1\ 1\ 1)$ GB and its related single crystal sample.

Fig. 4. Dislocation nucleation and evolution: (a) *Region II*, single crystal sample; (b) *Region II*, bi-crystal sample; (c) *Region III*, single crystal sample; (d) *Region III*, bi-crystal sample; (e) *Region IV*, single crystal sample; (f) *Region IV*, bi-crystal sample; (g) *Region V*, single crystal sample; (h) *Region V*, bi-crystal sample.

Fig. 5. Variation of mean contact pressure p_m and dislocation length λ as a function of indentation depth h for S1 bicrystal and their related single crystal samples with grain boundaries of : (a) $\Sigma 11 (1\ 1\ 3)$ (b) $\Sigma 3 (1\ 1\ 2)$ (c) $\Sigma 11 (3\ 3\ 2)$ (d) $\Sigma 11 (2\ 2\ 5)/(4\ 4\ 1)$ (e) $\Sigma 3 (1\ 1\ 2)/(\bar{5}\ \bar{5}\ 2)$ (f) $\Sigma 3 (1\ 1\ 4)/(1\ 1\ 0)$.

Fig. 6. Dislocation nucleation from the GB: (a) bi-crystal sample with $\Sigma 3 (1\ 1\ 2)/(\bar{5}\ \bar{5}\ 2)$ GB, $h \approx 0.37$ nm; (b) bi-crystal sample with $\Sigma 11 (2\ 2\ 5)/(4\ 4\ 1)$ GB, $h \approx 0.43$ nm.

Fig. 7. Variation of mean contact pressure p_m as a function of indentation depth h for S2 single crystal and their related bi-crystal samples with the grain boundaries of (a) $\Sigma 3 (1\ 1\ 1)$ (b)

$\Sigma 11 (1 1 3)$ (c) $\Sigma 3 (1 1 2)$ (d) $\Sigma 11 (3 3 2)$ (e) $\Sigma 11 (2 2 5)/(4 4 1)$ (f) $\Sigma 3 (1 1 2)/(\bar{5} \bar{5} 2)$ (g) $\Sigma 3 (1 1 4)/(1 1 0)$.

Fig. 8. Variation of mean contact pressure p_m and dislocation length λ as a function of indentation depth h for S2 bicrystal sample with $\Sigma 3 (1 1 1)$ GB and its related single crystal sample.

Fig. 9. Dislocation nucleation and evolution: (a) single crystal sample, $h \approx 0.88$ nm; (b) bi-crystal sample, $h \approx 0.88$ nm; (c) single crystal sample, $h \approx 1.15$ nm; (d) bi-crystal sample, $h \approx 1.15$ nm; (e) single crystal sample, $h \approx 1.44$ nm; (f) bi-crystal sample, $h \approx 1.44$ nm; (g) single crystal sample, $h \approx 2.03$ nm; (h) bi-crystal sample, $h \approx 2.03$ nm; (i) single crystal sample, $h \approx 11.5$ nm; (j) bi-crystal sample, $h \approx 11.5$ nm.

Fig. 10. Variation of mean contact pressure p_m and dislocation length λ as a function of indentation depth h for S2 bicrystal and their related single crystal samples with grain boundaries of : (a) $\Sigma 11 (1 1 3)$ (b) $\Sigma 3 (1 1 2)$ (c) $\Sigma 11 (3 3 2)$ (d) $\Sigma 11 (2 2 5)/(4 4 1)$ (e) $\Sigma 3 (1 1 2)/(\bar{5} \bar{5} 2)$ (f) $\Sigma 3 (1 1 4)/(1 1 0)$.

Fig. 11. Variation of mean contact pressure p_m and total dislocation length located in the upper grain λ_{upper} as a function of indentation depth h for S2 bicrystal and their related single crystal samples with grain boundaries of (a) $\Sigma 3 (1 1 1)$ (b) $\Sigma 11 (1 1 3)$ (c) $\Sigma 3 (1 1 2)$ (d) $\Sigma 11 (3 3 2)$ (e) $\Sigma 11 (2 2 5)/(4 4 1)$ (f) $\Sigma 3 (1 1 2)/(\bar{5} \bar{5} 2)$ (g) $\Sigma 3 (1 1 4)/(1 1 0)$.

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Fig. 1. The equilibrium structure of the symmetric and asymmetric tilt grain boundaries of (a) $\Sigma 3$ (1 1 1) $[1 \bar{1} 0]$ ($\theta = 109.5^\circ$), (b) $\Sigma 11$ (1 1 3) $[1 \bar{1} 0]$ ($\theta = 50.5^\circ$), (c) $\Sigma 3$ (1 1 2) $[1 \bar{1} 0]$ ($\theta = 70.5^\circ$), (d) $\Sigma 11$ (3 3 2) $[1 \bar{1} 0]$ ($\theta = 129.5^\circ$), (e) $\Sigma 11$ (2 2 5)/(4 4 1) ($\varphi = 54.74^\circ$), (f) $\Sigma 3$ (1 1 2)/ $(\bar{5} \bar{5} 2)$ ($\varphi = 19.47^\circ$), and (g) $\Sigma 3$ (1 1 4)/(1 1 0) ($\varphi = 35.26^\circ$), along $[1 \bar{1} 0]$ axis.

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Fig. 2. Variation of mean contact pressure p_m as a function of indentation depth h for S1 single crystal and their related bi-crystal samples with the grain boundaries of (a) $\Sigma 3 (1 1 1)$ (b) $\Sigma 11 (1 1 3)$ (c) $\Sigma 3 (1 1 2)$ (d) $\Sigma 11 (3 3 2)$ (e) $\Sigma 11 (2 2 5)/(4 4 1)$ (f) $\Sigma 3 (1 1 2)/(\bar{5} \bar{5} 2)$ (g) $\Sigma 3 (1 1 4)/(1 1 0)$.

Fig. 3. Variation of mean contact pressure p_m and dislocation length λ as a function of indentation depth h for S1 bicrystal sample with $\Sigma 3 (1 1 1)$ GB and its related single crystal sample.

(a) single crystal sample, $h \approx 0.6$ nm

(b) bi-crystal sample, $h \approx 0.6$ nm

(c) single crystal sample, $h \approx 0.9$ nm

(d) bi-crystal sample, $h \approx 0.9$ nm

(e) single crystal sample, $h \approx 1.1$ nm

(f) bi-crystal sample, $h \approx 1.1$ nm

(g) single crystal sample, $h \approx 2.15$ nm

(h) bi-crystal sample, $h \approx 2.15$ nm

Fig. 4. Dislocation nucleation and evolution: (a) *Region II*, single crystal sample; (b) *Region II*, bi-crystal sample; (c) *Region III*, single crystal sample; (d) *Region III*, bi-crystal sample; (e) *Region IV*, single crystal sample; (f) *Region IV*, bi-crystal sample; (g) *Region V*, single crystal sample; (h) *Region V*, bi-crystal sample.

Fig. 5. Variation of mean contact pressure p_m and dislocation length λ as a function of indentation depth h for S1 bicrystal and their related single crystal samples with grain boundaries of : (a) $\Sigma 11 (1 1 3)$ (b) $\Sigma 3 (1 1 2)$ (c) $\Sigma 11 (3 3 2)$ (d) $\Sigma 11 (2 2 5)/(4 4 1)$ (e) $\Sigma 3 (1 1 2)/(\bar{5} \bar{5} 2)$ (f) $\Sigma 3 (1 1 4)/(1 1 0)$.

(a) bi-crystal sample with $\Sigma 3 (1 1 2)/(\bar{5} \bar{5} 2)$ GB, $h \approx 0.37$ nm

(b) bi-crystal sample with $\Sigma 11\ (2\ 2\ 5)/(4\ 4\ 1)$ GB, $h \approx 0.43$ nm

Fig. 6. Dislocation nucleation from the GB: (a) bi-crystal sample with $\Sigma 3\ (1\ 1\ 2)/(\bar{5}\ \bar{5}\ 2)$ GB, $h \approx 0.37$ nm; (b) bi-crystal sample with $\Sigma 11\ (2\ 2\ 5)/(4\ 4\ 1)$ GB, $h \approx 0.43$ nm.

(c)

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Fig. 7. Variation of mean contact pressure p_m as a function of indentation depth h for S2 single crystal and their related bi-crystal samples with the grain boundaries of (a) $\Sigma 3 (1 1 1)$ (b) $\Sigma 11 (1 1 3)$ (c) $\Sigma 3 (1 1 2)$ (d) $\Sigma 11 (3 3 2)$ (e) $\Sigma 11 (2 2 5)/(4 4 1)$ (f) $\Sigma 3 (1 1 2)/(\bar{5} \bar{5} 2)$ (g) $\Sigma 3 (1 1 4)/(1 1 0)$.

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Fig. 8. Variation of mean contact pressure p_m and dislocation length λ as a function of indentation depth h for S2 bicrystal sample with $\Sigma 3 (1 1 1)$ GB and its related single crystal sample.

Homogeneous dislocation nucleation

(a) single crystal sample, $h \approx 0.88$ nm

Homogeneous dislocation nucleation

(b) bi-crystal sample, $h \approx 0.88$ nm

Cross-slip of screw components and elongation of the dislocations pinned at their ends

(c) single crystal sample, $h \approx 1.15$ nm

Cross-slip of screw components and elongation of the dislocations pinned at their ends

(d) bi-crystal sample, $h \approx 1.15$ nm

Dislocation loops can freely move downward towards the sample bottom.

(e) single crystal sample, $h \approx 1.44$ nm

Dislocation loops movement are blocked by grain boundary.

(f) bi-crystal sample, $h \approx 1.44$ nm

(g) single crystal sample, $h \approx 2$ nm

(h) bi-crystal sample, $h \approx 2$ nm

(i) single crystal sample, $h \approx 11.5$ nm

(j) bi-crystal sample, $h \approx 11.5$ nm

Fig. 9. Dislocation nucleation and evolution: (a) single crystal sample, $h \approx 0.88$ nm; (b) bi-crystal sample, $h \approx 0.88$ nm; (c) single crystal sample, $h \approx 1.15$ nm; (d) bi-crystal sample, $h \approx 1.15$ nm; (e) single crystal sample, $h \approx 1.44$ nm; (f) bi-crystal sample, $h \approx 1.44$ nm; (g) single crystal sample, $h \approx 2.03$ nm; (h) bi-crystal sample, $h \approx 2.03$ nm; (i) single crystal sample, $h \approx 11.5$ nm; (j) bi-crystal sample, $h \approx 11.5$ nm.

Fig. 10. Variation of mean contact pressure p_m and dislocation length λ as a function of indentation depth h for S2 bicrystal and their related single crystal samples with grain boundaries of : (a) $\Sigma 11 (1 1 3)$ (b) $\Sigma 3 (1 1 2)$ (c) $\Sigma 11 (3 3 2)$ (d) $\Sigma 11 (2 2 5)/(4 4 1)$ (e) $\Sigma 3 (1 1 2)/(\bar{5} \bar{5} 2)$ (f) $\Sigma 3 (1 1 4)/(1 1 0)$.

(a)

(b)

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Fig. 11. Variation of mean contact pressure p_m and total dislocation length located in the upper grain λ_{upper} as a function of indentation depth h for S2 bicrystal and their related single crystal samples with grain boundaries of (a) $\Sigma 3 (111)$ (b) $\Sigma 11 (113)$ (c) $\Sigma 3 (112)$ (d) $\Sigma 11 (332)$ (e) $\Sigma 11 (225)/(441)$ (f) $\Sigma 3 (112)/(\bar{5}52)$ (g) $\Sigma 3 (114)/(110)$.