

Unified Model for Size-Dependent to Size-Independent Transition in Yield Strength of Crystalline Metallic Materials

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(Received 4 January 2020; revised manuscript received 14 May 2020; accepted 18 May 2020; published 9 June 2020)

Size-dependent yield strength is a common feature observed in miniaturized crystalline metallic samples, and plenty of studies have been conducted in experiments and numerical simulations to explore the underlying mechanism. However, the transition in yield strength from bulklike to size-affected behavior has received less attention. Here a unified theoretical model is proposed to probe the yield strength of crystalline metallic materials with sample size from nanoscale to macroscale. We show that the transition in yield strength versus size can be fully explained by the competition between the stresses required for dislocation source activation and dislocation motion, which is regulated by dislocation density, irradiation defect, grain boundary, and so on. Based on various grain boundary densities, the extended Hall-Petch relation, incorporated into the unified model, captures the reverse size effect for polycrystalline samples. The proposed model predictions agree well with reported experimental measurements of various specimens, including the prestrained nickel, irradiated copper, ultrafine grain tungsten, and so on.

DOI: 10.1103/PhysRevLett.124.235501

Understanding mechanical properties of crystalline metallic materials at the small length scale is important for designing and fabricating mechanically reliable nanodevices and nanostructures [1]. Size-dependent yielding, as a well-known phenomenon, is widely observed in the uniaxial tension or compression tests on micropillars [2–8]. Compared with bulk samples, higher stress is required in miniaturized samples during plastic deformation [5–7, 9–13]. In the past few decades, abundant studies demonstrated that the yield strength obeys a power law relation with respect to the specimen size in microscale regime and can be understood in terms of the dislocation source-limited mechanism [12–18]. Namely, the dislocation generation becomes harder at the small length scale [16,17], and escape of dislocations near the free surfaces causes the dislocation starvation or exhaustion, so a higher stress is needed to activate new dislocation sources [4,18–20]. The critical resolved shear stress (CRSS) τ_c of miniaturized materials, characterizing the yield strength σ_y , is generally adopted as the following simple linear form in most existing models [15,17,21–24]:

$$\tau_c = \tau_m + \tau_s. \quad (1)$$

Here τ_m denotes the stress required for dislocation motion, which is usually regarded as the bulk CRSS, and τ_s represents the contribution of dislocation source hardening. For crystalline materials, τ_s can be described by a power law $\tau_s = K\mu b d^{-m}$ [15], where μ is the material shear modulus, b is the magnitude of Burgers vector, d is the grain size, K and m are the material parameters associated

with stacking fault energy [6,25], temperature [7,15], dislocation source density [19], and so on. Although the size dependence of yield strength or CRSS at nanoscale or microscale has been captured by Eq. (1) [15,17,22,23], the transition from a size-dependent to size-independent yield strength is veiled [18,26], especially in prestrained nickel (Ni) [27], irradiated copper (Cu) [9], high strength Ni alloy [28], and tungsten (W) [11].

Besides the size-dependent yield strength depicted by the thick (red) line in Fig. 1, in the microtesting of one-dimensional polycrystalline materials such as nanowires and micropillars, the reverse size effect that the yield strength increases with increasing sample diameter has

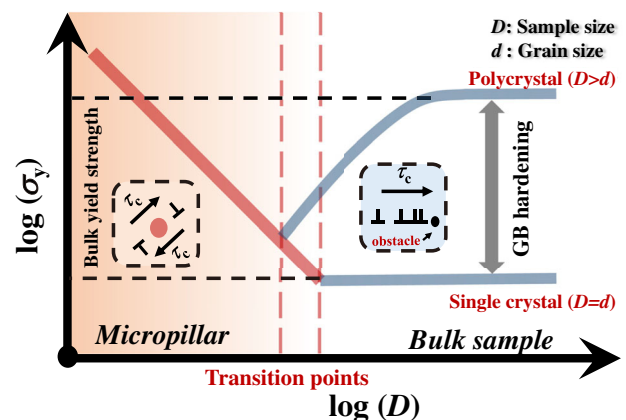


FIG. 1. Schematic of yield strength σ_y as a function of the sample size D .

been reported [11,29,30] as indicated by the thin (gray) line in Fig. 1.

In this work, we propose a unified model to probe the yield strength of crystalline metallic materials in a wide sample size range at a fundamental level. For single crystals, a novel yielding mechanism is presented that the yield strength depends on the competition between stresses required for dislocation motion and dislocation source activation. This competition model is fundamentally different from the model in previous works where the source activation stress is considered as a part of superposition form. Moreover, we extend the classical Hall–Petch relation to the microsize polycrystals based on the evolution of grain boundary (GB) density. We then propose a model to describe the yield strength of single crystals and polycrystals analytically.

It is well known that the hardening effects of bulk materials arise from different aspects of material microstructures. For example, the lattice friction, dislocation forest, GB, and irradiation defect are generally regarded as the obstacles for dislocation motion, and they together contribute to the stress τ_m required for dislocation motion following the superposition principle [23,31]. Different from the bulk materials, the size effect of miniaturized samples is controlled by dislocation source-limited mechanism. At the small length scale, the limited dislocation source activation leads to a lack of mobile dislocations, which further causes the increasing yield strength, i.e., source hardening effect τ_s . At $\tau_s \gg \tau_m$, the yielding model in superposition form [Eq. (1)] is valid to model the size-dependent yield strength [15,17,21–23]. However, in the cases where τ_s and τ_m are at the same order, Eq. (1) has difficulty in explaining the experimental results [9,11,27,28], which are depicted as the transition behaviors in Fig. 1.

Focusing on physical mechanisms of size-dependent yield strength of crystalline metallic materials, here we hypothesize that the yield strength of material characterized by the CRSS τ_c is dominated by the competition between the activation of dislocation source and motion of dislocations as given in Eq. (2)

$$\tau_c = \max(\tau_m, \tau_s) = \max(\tau_f + \tau_{\text{dis}}, \tau_s) \quad (2)$$

with τ_m depending on the lattice friction stress τ_f and dislocation forest hardening stress τ_{dis} . Here τ_{dis} can be represented as $\tau_{\text{dis}} = \alpha\mu b\sqrt{\rho_{\text{dis}}}$, where ρ_{dis} denotes the dislocation density and α is the material constant. Equation (2) points out that the plastic deforming in crystalline metallic materials needs to satisfy the following requirements. First, the stress on dislocations is sufficient to overcome the obstacles on the slip plane, and second, dislocation source could continuously emit dislocation. For pure single crystals, Eq. (2) can be rewritten as

$$\tau_c = \max(\tau_f + \alpha\mu b\sqrt{\rho_{\text{dis}}}, K\mu b d^{-m}), \quad (3)$$

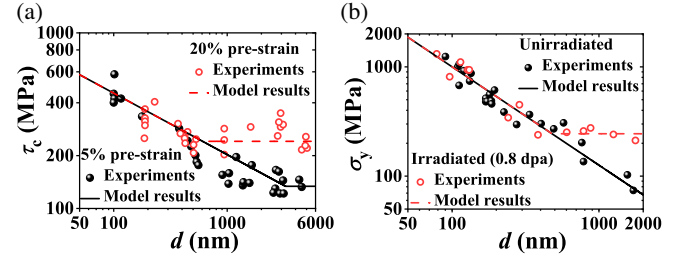


FIG. 2. CRSS τ_c /Yield strength σ_y , as a function of pillar diameter for (a) Ni single crystal at different prestrains and (b) irradiated and unirradiated Cu single crystals. Symbols represent experimental data from Refs. [9] and [27].

where $\tau_m = \tau_f + \alpha\mu b\sqrt{\rho_{\text{dis}}}$ is adopted to be independent of the sample size [15,17]. Note that the proposed model is not applicable for the dislocation-free whisker owing to the absence of dislocation source [6,12,15].

Equation (3) is verified by microcompression tests on micropillars of prestrained Ni single crystal [27]. The size-dependent CRSS of Ni micropillars of different diameters d has been measured at prestrains of 5% and 20%. A larger prestrain corresponds to a higher dislocation density [27]. In Fig. 2(a), we compare measured τ_c of nickel micropillars of different diameters d at 5% and 20% prestrains with our theoretical prediction (model parameters listed in Table I). Here we ignore the lattice friction stress τ_f of face-centered cubic materials at room temperature [23]. It is shown that Eq. (3) with $\rho_{\text{dis}} = 2 \times 10^{14} \text{ m}^{-2}$ and $6.5 \times 10^{14} \text{ m}^{-2}$ adopted at 5% and 20% prestrains, respectively, agrees well with experimental results with measured ρ_{dis} of $3.7 \times 10^{14} \text{ m}^{-2}$ and $4.5 \times 10^{14} \text{ m}^{-2}$ [27]. The CRSS of both samples exhibits significant size effect at relatively small sizes. As the sample size d exceeds certain values (around 500 nm and 3000 nm at 5% and 20% prestrains, respectively), CRSS becomes size independent because τ_s is less than τ_m . The length scale of the transition of CRSS from size dependent to size independent decreases as the dislocation density increases, which is consistent with the dislocation dynamics simulations [26].

The transition in yield strength from size dependent to size independent can also be observed in the microcompression tests of irradiated single crystal Cu [9]. As shown in Fig. 2(b), the irradiated Cu micropillars present a transition behavior at submicroscale (around 500 nm), whereas the unirradiated ones of the same size still display size effect. In previous studies, the resistance of irradiation defects to dislocation motion has been described by $\beta\sqrt{N_{\text{def}}d_{\text{def}}}$, where β is the hardening coefficient, and N_{def} and d_{def} are, respectively, the average density and size of the irradiation defect [23,33,34]. Therefore, the CRSS τ_c^{irr} of the irradiated single crystals is proposed as

$$\tau_c^{\text{irr}} = \max(\tau_f + \alpha\mu b\sqrt{\rho_{\text{dis}}} + \beta\sqrt{N_{\text{def}}d_{\text{def}}}, K\mu b d^{-m}). \quad (4)$$

TABLE I. Parameters for model prediction of Ni and Cu single crystals.

Materials	μ (GPa)	b (nm)	K (m^{m-1})	m	α	β (N/m)	N_{def} (10^{23} m^{-3})	d_{def} (nm)
Ni	76 ^a	0.249 ^a	8.5×10^4	0.35	0.5 ^a
Cu	48.3 ^a	0.255 ^a	17	0.9	0.5 ^a	0.224	4.5 ^b	2.5 ^b

Parameters with superscripts a and b are adopted from Refs. [32] and [23], respectively.

The dislocation density ρ_{dis} here is adopted as $2 \times 10^{12} \text{ m}^{-2}$ [23]. Because the yield stress is measured under the loading direction [100] in experiments, we have $\sigma_y^{\text{irr}} = \tau_c^{\text{irr}}/R$ with $R = 0.408$ as the corresponding Schmid factor. Figure 2(b) shows that Eq. (4) agrees well with experimental results (model parameters listed in Table I). In addition to the transition behavior of yield strength in both irradiated and unirradiated Cu, the absence of irradiation hardening effect in irradiated Cu at $d < 500 \text{ nm}$ validates Eq. (4). At $\tau_s > \tau_m$, the yield strength is dominated by τ_s , so unirradiated and irradiated samples could exhibit the same yield strength.

From the case studied above, one can see that the transition point (Fig. 1) in miniaturized single crystals decreases as the stress required for dislocation motion increases. Besides the prestrained Ni and irradiated Cu single crystals, Eq. (2) is also valid for high strength Ni-based alloy, in which a high constant yield strength is observed for diameters ranging from 200 nm to 4 μm [28] (see Fig. S1 in Supplemental Material [35]). This model enriches our understanding of the appearance or disappearance of the size effect for Ni-based alloy micropillars [28,36–38].

Once the sample size exceeds the transition point (Fig. 1), the miniaturized polycrystalline materials ($d < D$) generally display a reverse size effect such that yield strength decreases with a decreasing sample size [29,30]. To explain the reverse size effect, Yang *et al.* [29]

suggested that the yield strength of the region near the free surface is smaller than that of the interior region and proposed a mixture model to characterize the yield strength of miniaturized polycrystalline copper wires, which agreed well with experiments [30]. However, there exist several artificial fitting parameters in the model that further inspire one to consider whether there is a better theoretical model.

In this study, the reverse size effect is attributed to the increases of the proportion of GBs, which could increase the yield strength by hindering dislocation motion. For bulk polycrystalline materials, the classical Hall–Petch relation [39,40] is widely applied to describe the GB hardening effect. Here we propose an analytical model based on the Hall–Petch relation to investigate the size-dependent yield strength of miniaturized polycrystalline materials. The classical Hall–Petch relation has the general form [39,40]

$$\tau_c = \tau_0 + k\sqrt{\frac{1}{d}}, \quad (5)$$

where k is material constant, d is the characteristic grain size, and $\tau_0 = \tau_f + \tau_{\text{dis}}$ is a combination of intrinsic lattice resistance and dislocation hardening stress for pure metallic materials. It is noted that $1/d$ is proportional to GB density (GB area per unit volume) [32]. Thus, Eq. (5) can be rewritten as [31,41]

$$\tau_c = \tau_0 + k^* \sqrt{S_V}, \quad (6)$$

where $S_V \propto 1/d$ is defined as GB density. Note that the value of GB density in miniaturized samples is also associated with sample size. The inset in Fig. 3 provides a schematic illustration of the polycrystalline materials in which the geometric shape of each grain is set to be a cube. Then, the corresponding GB density can be determined by

$$S_V = \sum_{n=1}^3 \left(\frac{1}{d_n} - \frac{1}{D_n} \right). \quad (7)$$

Using the harmonic mean of grain sizes $3/\sum_{n=1}^3 1/d_n$ and harmonic mean of sample sizes $3/\sum_{n=1}^3 1/D_n$ to effectively represent the grain size d and sample size D , Eq. (7) can be written as $S_V = 3(1/d - 1/D)$. To cover the variety of grain shapes, we introduce a grain shape coefficient ξ and rewrite Eq. (7) as

$$S_V = \xi \left(\frac{1}{d} - \frac{1}{D} \right) \quad (8)$$

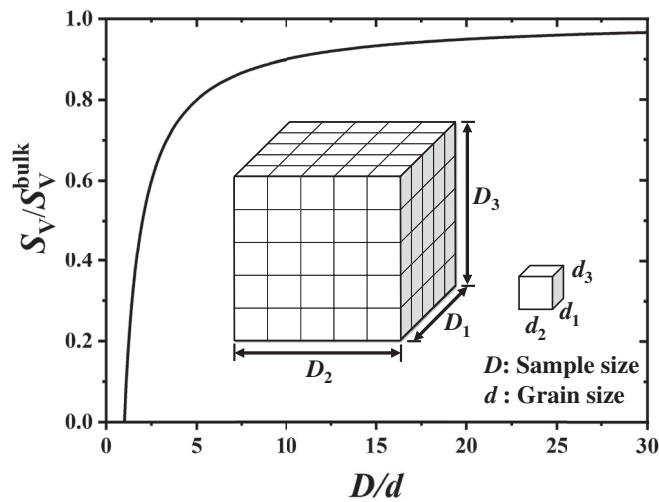


FIG. 3. Normalized GB density S_V/S_V^{bulk} as a function of D/d . Embedded graph shows a simplified illustration of polycrystalline materials.

with $\xi \approx 2$ for columnar grains and $\xi \approx 3$ for equiaxed grains. The normalized GB density can then be expressed as $S_V/S_V^{\text{bulk}} = 1 - d/D$ with $S_V^{\text{bulk}} = \xi/d$. Figure 3 shows S_V/S_V^{bulk} as a function of D/d . The GB density increases rapidly at $D/d < 5$ and gradually saturates at $D/d > 15$.

Substituting Eq. (8) into Eq. (6), we have

$$\tau_c = \tau_0 + k\sqrt{\frac{1}{d} - \frac{1}{D}}. \quad (9)$$

Here the material parameter $k(=\xi k^*)$ is associated with the grain shape and material properties. The modified Hall–Petch relation includes not only the grain size d but also the sample size D . At $d = D$, Eq. (9) reduces to $\tau_c = \tau_0$ without GB effect. At $d \ll D$, Eq. (9) reduces to the classical Hall–Petch relation in Eq. (5).

Note that only the homogeneous materials are discussed here. For the gradient-structured polycrystals, GB density is a function of spatial location [42]. Similar to the classical Hall–Petch relation, a restriction of Eq. (9) is that it cannot be applied to samples with grain size on the order of 10 nm whose plastic deformation is mediated by GB motions such as GB migration, GB sliding, and grain rotation [43–45].

Equation (9) is verified with experiments on Cu [29,46,47] and austenitic stainless steel [5,30] polycrystals. To normalize the experimental data for different materials [29,30], we analyze the size effect using the normalized yield strength $\sigma_y/\sigma_y^{\text{bulk}}$ as

$$\frac{\sigma_y}{\sigma_y^{\text{bulk}}} = \frac{M\left(\tau_0 + k\sqrt{\frac{1}{d} - \frac{1}{D}}\right)}{M\left(\tau_0 + k\sqrt{\frac{1}{d}}\right)} = \frac{\tau_0 + k\sqrt{\frac{1}{d} - \frac{1}{D}}}{\tau_0 + k\sqrt{\frac{1}{d}}}. \quad (10)$$

Here σ_y^{bulk} denotes the bulk yield strength, and M is the Taylor factor for the cubic polycrystalline materials [48]. For polycrystal materials including limited number of grains, the Taylor factor depends on not only the grain orientations but also the loading direction and is not a constant. As it is quite difficult to determine the value of M for miniaturized samples, here $M \approx 3$ is taken as in most of the literature [48] to simplify the calculation.

$\sigma_y/\sigma_y^{\text{bulk}}$ is plotted in Fig. 4 as a function of D/d for Cu ($d = 3 \mu\text{m}$ and $\tau_0 = 10 \text{ MPa}$) and austenitic stainless steel ($d = 0.6 \mu\text{m}$ and $\tau_0 = 200 \text{ MPa}$) polycrystal samples. In the experiments, the grain size of Cu polycrystals is from $3 \mu\text{m}$ to $150 \mu\text{m}$ [29,46,47]; the grain size of austenitic stainless steels [5,30] falls in a range from $0.6 \mu\text{m}$ to $90 \mu\text{m}$. As τ_0 depends on the initial dislocation densities, here to explore the role of the microstructure in evolution of $\sigma_y/\sigma_y^{\text{bulk}}$, different values of d and τ_0 are adopted in our model predictions (see Fig. S2 in Supplemental Material [35]). It is shown that, for both Cu and austenitic stainless steels, the reverse size effect becomes slightly less obvious as d and τ_0 increase, which is attributed to the reduction in the contribution of GB hardening in yield strength.

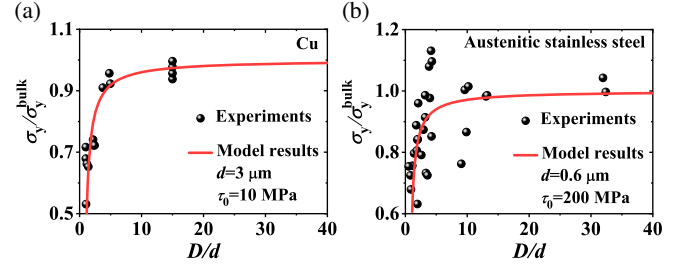


FIG. 4. Size dependence of normalized yield stress $\sigma_y/\sigma_y^{\text{bulk}}$ for (a) Cu [29] and (b) austenitic stainless steel [30] polycrystal samples. The material constant k is taken as $0.06 \text{ MPa} \cdot \sqrt{\text{m}}$ and $0.2 \text{ MPa} \cdot \sqrt{\text{m}}$ for Cu and austenitic stainless steel, respectively.

Our analytical predictions given by Eq. (10) agree well with experimental results, indicating that the proposed model could effectively describe the size dependence of yield strength for polycrystalline samples. At $D/d < 5$, $\sigma_y/\sigma_y^{\text{bulk}}$ increases rapidly with increasing D/d and the reverse size effect is strong, consistent with the trend in Fig. 3. The yield strength increases with GB density. At $D/d > 15$, $\sigma_y/\sigma_y^{\text{bulk}}$ is close to 1 as expected, attributed to the saturation of GB density.

GB hardening effect arises from the resistance of GB to dislocation motion. Substituting Eqs. (3) and (9) into Eq. (2), a modified yield model can then be given as

$$\tau_c = \max\left(\tau_f + \alpha\mu b\sqrt{\rho_{\text{dis}}} + k\sqrt{\frac{1}{d} - \frac{1}{D}}, K\mu b d^{-m}\right). \quad (11)$$

Eq. (11) is more general and could describe the yield strength for single crystal and polycrystalline materials with sample size ranging from nano-length to macro-length scale.

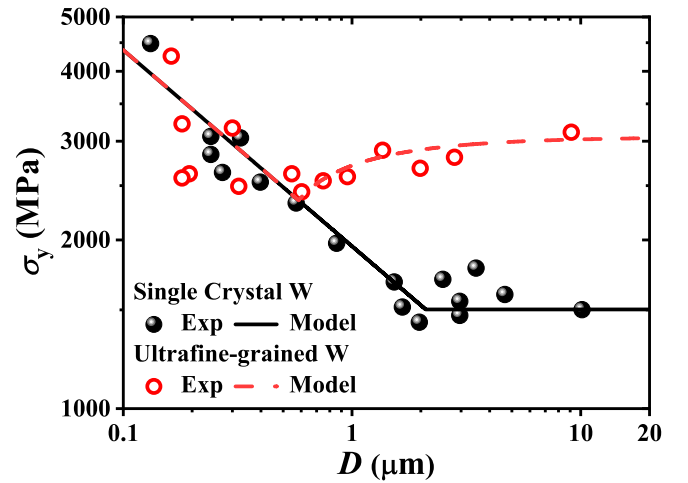


FIG. 5. Size-dependent yield stresses σ_y for the single crystal and ultrafine grain W. Symbols represent experimental data from Ref. [11].

TABLE II. Parameters for model prediction of W.

Materials	τ_f (MPa)	μ (GPa)	b (nm)	K (m^{m-1})	m	k ($\text{MPa}\sqrt{\text{m}}$)	α	ρ_{dis} (10^{14} m^{-2})
Single crystal W	370 ^a	153.7 ^a	0.274 ^a	1.5×10^5	0.35	...	0.45 ^b	1.65 ^c
Ultrafine grain W	370 ^a	153.7 ^a	0.274 ^a	1.2×10^5	0.35	0.28	0.45 ^b	1.65 ^c

Parameters with superscripts *a*, *b* and *c* are adopted from Refs. [15], [32], and [11], respectively.

The microcompression tests on single crystal and ultrafine grain W with grain size $d \approx 480$ nm are used to verify Eq. (11) [11]. Figure 5 displays σ_y as a function of D for single crystal and ultrafine grain W, and the experimental results show the same trend as illustrated in Fig. 1. The parameters used are listed in Table II. The Schmid factor is taken as $R = 0.408$ [11] and the Taylor factor is $M \approx 3$ for single crystal W and ultrafine grain W, respectively.

It is shown that Eq. (11) can accurately capture the experimental results and both samples exhibit the transition feature. The yield mechanism of various sample sizes is illustrated in Fig. 1. For single crystals, the transition point of yield strength from size dependent to size independent is at $D \approx 2 \mu\text{m}$, consistent with our prediction. At $D \geq 2 \mu\text{m}$, dislocation motion dominates the yield of materials. For the ultrafine grain W, the yield strength exhibits the size effect that σ_y decreases with D increasing at $D < 500$ nm, and the sample size is smaller than the initial grain size 480 nm. Therefore, it can be regarded as a single crystal, and its yield strength is the same as that of single crystals. As D exceeds 500 nm, the yield stress of ultrafine grain W displays the reverse size effect, indicating the increases of GB density. On the other hand, it also demonstrates the transition of the yield strength of ultrafine grain W from dislocation source controlled to dislocation motion controlled.

Equation (11) also suggests that the polycrystalline samples will not display the GB hardening effect at $\tau_m \leq \tau_s$. Such predictions have been observed in the Cu bi-crystal [49–51] and ultrafine grain Cu [52] (as detailed in Figs. S3 and S4 of Supplemental Material [35]).

In summary, we establish a unified model to characterize the yield strength for crystalline metallic materials with sample size from microscales to macroscales. Compared with existing models, the yielding of miniaturized metallic samples in this study needs to satisfy both dislocation motion and dislocation source activation. CRSS can then be determined by the competition between the stresses required for these two mechanisms, so that the transition of yield strength from size dependent to size independent can be captured. As the sample size becomes smaller than the length scale of transition, the yield strength is governed by dislocation source activation stress to exhibit the size dependence. After the source activation stress less than dislocation motion stress, single crystals display the size-independent yield strength, and polycrystals could show the reverse size effect of yield strength owing to increase in GB density. All of these experimental results are accurately captured by the theoretical model. It is revealed that the

transition of yield strength from size dependent to size independent is sensitive to microstructures, depending on the contribution of dislocation density, GB density, irradiation defect, dispersoids, and other microstructures to dislocation motion resistance.

The authors thank the National Natural Science Foundation of China (Grants No. 11632001, No. 11521202, No. 11988102, and No. U1830121), the National Science and Technology Major Project (Grant No. 2017-VI-0003-0073), and the Science Challenge Project (Grant No. TZ2018001) for their support.

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