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Research Article

Effects of Free Surface and Heterogeneous Residual Internal Stress on Stress-Driven Grain Growth in Nanocrystalline Metals

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By reevaluating the experimental study of Zhang et al. (2005), here we demonstrate that the extent of grain growth, previously proposed to be solely driven by external stress, may have been significantly overestimated. A new physical mechanism, termed as free surface assisted stress-driven grain growth (or self-mechanical annealing), is proposed and discussed in detail. Representing the cooperative effect of free surface and heterogeneous residual internal stress, the proposed mechanism is considered more favorable than the traditional pure stress-driven mechanism for interpreting the abnormal grain growth widely observed in deforming nanocrystalline metals at room temperature.

Whilst grain boundaries (GBs) in polycrystalline metals are traditionally described as a mechanically static and immovable microstructural obstacle to dislocation motion, numerous experimental studies have provided convincing evidence that GBs are not as static as traditionally assumed in nanocrystalline (NC) metals [1–6]. These studies indicated further that the unusual mobility of GBs under applied stress may eventually lead to rapid grain growth in NC metals. Also, surprisingly, the grain growth was found to be faster at cryogenic temperature than at room temperature (RT), suggesting that the grain coarsening process was driven primarily by stress but not diffusion [3, 5, 6]. As the classical models for grain growth failed to explain the observed grain structure instability in NC metals, identifying the underlying mechanism of the unique grain growth process has become a hot research topic.

Before clarifying the underlying mechanism, however, existing inconsistency concerning the mechanical properties of NC metals needs to be addressed in advance. Contrary to prevalent experimental reports of grain growth in NC metals [1, 2, 4–9] by *in situ* or post mortem transmission electron microscopy (TEM) analysis, bulk NC metals exhibiting remarkable grain structure stability have also been reported [10, 11]. For typical instance, whilst significant stress-assisted grain growth was observed in an 800 nm grained Al thin film by means of *in situ* TEM straining experiments [2], however, it has never been observed in bulk nanostructured face-centered cubic (FCC) metals having similar grain size. As grain growth is commonly observed by TEM observation, this inconsistency may arise from surface effects inherent to TEM experiments. Given that the thin foils are less than a few micrometers in thickness, it remains unclear whether the observed GB and dislocation structures are representative of material structure during deformation, or just artifacts from thinning [12]. For *in situ* testing of ultrathin TEM foils, the remarkable grain instability may be enhanced by significant diffusion from nearby free surfaces, leading possible wrong interpretations of the coarsening extent. *Ex situ* TEM observations, on the other hand, may be influenced by stress relaxation during specimen preparation (e.g., sample thinning to submicron scale). Accordingly, identifying the dominating mechanism of stress-driven grain growth of NC metals at RT in NC metals and predicting quantitatively how fast and how far the stress-driven grain growth could

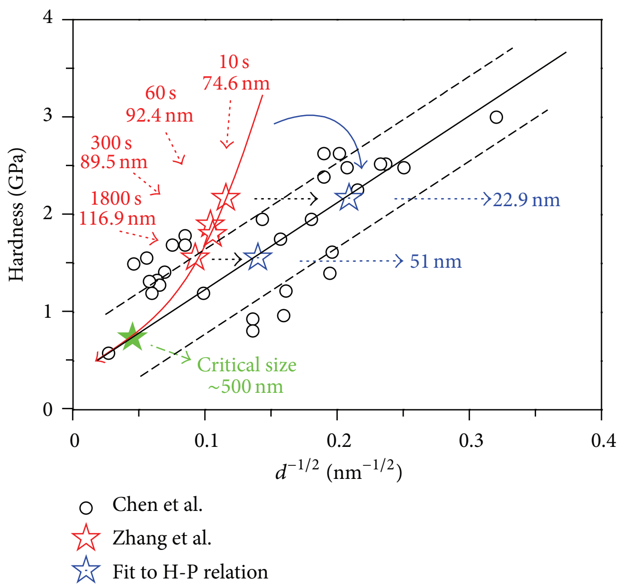

FIGURE 1: Grain size dependent hardness of Cu. The circles and the generated trend line of Hall-Petch relation are taken from the work of Chen et al. [13], and the red stars are data reproduced from the work of Zhang et al. [5].

be are of great scientific interest. The aim of this letter is to suggest new physical mechanism governing grain structure instability of NC metals that may significantly increase the extent of grain growth, by considering the cooperative effects of a free surface and heterogeneous residual stresses.

To rationalize the above hypothesis, we begin by reevaluating the work of Zhang et al. [5], which, as a milestone in the research field of *stress-driven grain growth*, is amongst the most cited studies thus far. In this study, a number of remarkable observations were made by nanoindentation and post mortem TEM analysis, including the following: (1) whilst the indentation hardness continually decreased with time for NC Cu, it did not change significantly for coarse grain Cu under the same circumstances; (2) the mechanical coarsening process, which was thought to be responsible for the decreasing hardness, was extremely fast; for example, the grain size increased more than five times within a dwell time of only 10 s in indentation creep test; (3) grain growth was faster at −190°C than at RT, which provides a crucial evidence that the grains could grow purely under the action of stress without any diffusional processes involved. However, the hardness measured for a specific grain size significantly deviates from other studies as shown in Figure 1. This indicates that the amount of grain growth as reported by Zhang et al. [5] was overestimated by ignoring several key parameters, for example, surface effects, intrinsically involved in preparing TEM samples.

In order to rationalize this assumption, a comparison between the test data presented by Zhang et al. [5] and the Hall-Petch (H-P) relation was made in Figure 1, with circles and the trend lines representing the H-P relation of pure Cu [13]. The results of Figure 1 demonstrate clearly that, based on the grain sizes calculated from Figure 2 in [5] (also see Figure 13 in [14] for better resolution), the hardness derived by Zhang et al. deviates significantly from the classical H-P relation for Cu. For example, the grain size of 74.6 nm observed after 10 s dwell time is more than three times larger than that obtained by fitting to H-P relation, that is, 22.9 nm as shown in Figure 1. After 1800 s dwell time, the grain size of 116.9 nm [5, 14] observed in post modern TEM is still more than twice larger than that fitting to the H-P relation. Note that the hardness (shown as red star in Figure 1) derived from indentation test should reflect the original grain size, that is, before the Cu sample was processed into a TEM foil. Consequently, the comparison shown in Figure 1 indicates that the grain sizes determined form postmortem TEM are significantly larger than those corresponding to indentation hardness. This observation suggests further that the grain size may only increase ~3 nm and ~41 nm after 10 s and 1800 s dwell time, respectively, in comparison with the initial average grain size of 20 nm for inert gas condensation (IGC) Cu reported by Zhang et al. [5]. Given that the grains directly underneath the indenter should exhibit a gradient distribution along the direction away from the indenter tip and that all the grains involved in the deformed region contribute to the indention hardness, the extent of grain growth must be larger than 3 nm, although this is still significantly overestimated. In other words, the majority of the extent of grain growth (specifically, several tens of nanometers for the test sample with 10 s dwell time) observed in post mortem TEM may be induced by mechanisms other than pure *stress-driven* process. In other words, additional mechanisms may exist and play a crucial role in initiating and promoting the grain growth in NC metals.

Upon deformation, yielding should occur first in relatively larger grains, with smaller grains accommodating the deformation elastically. This result in inhomogeneous stress distribution and significant residual internal stress built up in NC metals [15]. Even though the inhomogeneous stress distribution as well as the residual internal stress could be somewhat relaxed during unloading, the majority of the residual internal stress would remain after unloading for the deformed region is restricted by the surrounding undeformed material. In some certain circumstances, the residual internal stress remaining after unloading could affect the plastic deformation of NC metals in an unexpected way as addressed below.

In a study on the plastic deformation in free-standing NC metallic thin films, Rajagopalan et al. found that a substantial fraction of the plastic deformation could be recovered after unloading and subsequent annealing for a 50 nm grained Al film but not for 180 nm grained Au film [16]. This recovery process was proposed to be a thermally activated and time dependent event. Two mechanisms have been proposed to interpret the plastic deformation recovery: thermally activated dislocation jumps over grain boundary obstacles [16, 17] and heterogeneous grain boundary diffusion and sliding [15, 18]. Despite the fact that these two microscopic processes are quite different, both mechanisms considered the heterogeneity of the stress distribution; that is, large internal residual stresses built-up during plastic deformation play a dominant role in the recovery phenomena [15–18]. It

Table 1: Experimental data on deformation induced grain growth in NC metals and alloys.

Material (synthesis)	d_{initial} (nm)	Deformation mode	d_{final} (nm)	Mechanism	Reference
Al (MST)	20	*In situ* TEM indentation	>100	GBM, GR, GC	[22]
Ni (ED)	45	Compression creep	75	GBS, GR	[23]
Al (MST)	40–90	Tension	>100	Stress coupled GBM	[4]
Co-P (ED)	12	Tension	25	Stress-driven GBM, GC	[24]
Ni-Fe (ED)	23	Tension	250	Stress-driven GB process	[25]
Ni (ED)	30	HPT	130	Stress assisted GR	[8]
Ni (ED)	15	Multi-indentation	<200	GB processes	[26]
Cu (IGC)	36	Compression	90	Stress-driven mechanism	[27]
Ni (ED)	15	*In situ* TEM tension	>50	GR	[28]
Ni (ED)	37	Rolling	110	GB-dislocation interaction	[29]
Ni (ED) Ni-Fe (ED) Ni-Mn (ED)	42 25 115	Fatigue in tension	>500	Diffusional shear stress-driven growth	[30]
Ni-Fe (ED)	20	Rolling	50	GR	[31]
Pt (MST)	20	Tension	33	Stress-driven GB process	[32]
Ni-Fe (ED)	21	HPT	50	GR	[33]

MST: magnetron sputtering technique; IGC: inert gas condensation; ED: electrodeposition; HPT: high pressure torsion; GBM: GB migration; GR: grain rotation; GC: grain coalescence.

is assumed that the heterogeneity of the stress distribution provides the driving force for the reverse plastic deformation, which is then triggered by an annealing treatment at a higher temperature [16]. In a similar way, peeling restricting regions in preparing the TEM sample may trigger the redistribution of the heterogeneous stress remaining after indentation deformation and thus might cause additional grain growth.

Built upon existing studies as discussed above, we hypothesize that the mechanism(s) that corresponds to the additional grain coarsening observed by post mortem TEM is closely related to stress relaxation (i.e., reducing the heterogeneity of stress distribution) during TEM sample preparation, namely, free surface assisted stress-driven grain growth. To this end, two factors are crucial: significant residual internal stresses and free surface effect. Although the precise microstructural processes that can attribute to the additional extent of grain growth are yet clear, numerous potential deformation mechanisms may be activated in the additional coarsening process. As only a few grains are sitting atop each other and due to the proximity of the free surface in the TEM foil, surface effects could enhance both GB- and dislocation-mediated deformation processes significantly [19–21]. Table 1 summarizes existing experimental data [4, 8, 22–33] concerning abnormal grain growth in NC metals and alloys at 300 K (except those in [23] obtained at 373 K); the corresponding synthesizing method, initial grain size d_{initial}, testing method, final grain size d_{final}, and proposed dominant deformation mechanisms are also listed. It can be seen that the majority of the proposed grain coarsening processes involve GB motions under high stress (i.e., the so-called stress-driven or stress assisted GB activities), for example, GB sliding, GB rotation, GB migration, GB diffusion and even dislocation motions, and so forth. Apparently, no matter which one of these candidate mechanisms dominates the coarsening process, enlarging grain size requires a decrease in the volume fraction of GBs; that is, parts of the GBs present after unloading should be eliminated upon the thinning process.

Concerning dislocation movement, Rajagopalan et al. suggested that dislocation propagation in the plastically deformed NC metals would occur upon unloading and is responsible for the time evolution of strain recovery. However, neither molecular dynamics (MD) simulations [34] nor experiments [35] have revealed that dislocation nucleation and propagation induces grain growth in NC metals. Therefore, whilst such dislocation movement may be active in the free surface assisted stress-driven grain growth process, dislocation propagation could hardly move GBs and hence only serve as an accessory deformation mechanism in the coarsening of nanoscale grains. Opposed to that, numerous studies concerning *in situ* mechanical testing indicated that the applied stress could indeed cause significant GB migration while the dimension of the sample is in submicron scale [2, 36]. Note that the critical dimension of these sub-micrometer sized test samples is quite similar to those of a typical TEM foil. Thus, it is very likely that the internal stress in a TEM sample can induce grain growth upon the thinning procedure. The only difference is that the driving force changes from the external stress to the relaxation of the high residual internal stress.

The mechanism which causes the grain growth in the subsurface region might involve numerous GB motions, for example, shear-coupled GB migration, GB rotation, GB diffusion and GB sliding, and so forth. Specifically, in NC Al thin films, Rupert et al. [1] showed that GBs could be moved by shear stress in a manner of coupled grain boundary migration. The shear-coupled GB migration, initially modeled in a bicrystal configuration and later confirmed afterward by experimental [1] and simulation [37] studies, has been proposed as an effective way for stress relaxation by generating permanent shear [38]. In a similar way, the shear-coupled GB migration could occur in a thinning TEM foil via relaxation of high residual internal stress. On the other hand, Bobylev and Ovid'ko recently proposed a new mechanism for GB rotation in NC metals, that is, stress-driven rotations of GBs in subsurface areas of NC solids [21]. Here, the free surface provides an effective sink for GBs in reducing their length. This mechanism may indeed occur in stress-driven grain growth, as evidence of grain rotation dominating the coarsening process was found in NC Cu film by post-TEM observations [39] (it is worth mentioning that the sample preparation and indentation procedures as reported in [39] and [5] were identical). However, this kind of GB rotation can only affect the outmost grains and its contribution to additional grain growth is not as large as shear-coupled GB migration. In addition, heterogeneous grain boundary diffusion and sliding as proposed by Wei et al. [15, 18], which are also driven by the heterogeneity of the stress distribution in NC metals with submicron scale thickness, could also play a key role in the additional grain growth.

Taking as a whole, we cannot rule out any of the three possible mechanisms discussed above as the dominant one in the free surface assisted stress-driven grain growth process. To this end, elaborate experiments are clearly needed, as experimentally separating the extent of grain growth between the two physical processes, that is, pure stress-driven grain growth and the free surface assisted stress-driven grain growth, is extremely challenging. Therefore, numerical simulations, for example, MD simulation, may be more suitable for this task.

Other than experimental observation of grain growth in post-TEM indentation (specifically, [5] in this study), it should be mentioned that the mechanism of free surface assisted stress-driven grain growth may occur in all thinning TEM samples which underwent mechanical stress driven grain growth as shown in Table 1 [8, 23–27, 29–31, 33] and hence can shed some light on the interpretation of grain growth in *in situ* mechanical testing where no thinning process is required after deformation [4, 22, 28, 32]. Moreover, there must be a critical grain size above which the additional extent of grain growth induced by the proposed mechanism is negligible. On the basis of the trend line following Zhang et al.'s data [5] shown in Figure 1, we speculate that the critical grain size may be as large as roughly 500 nm.

Further, the mechanism proposed in the present study could explain, to certain extent, why grain growth was faster at −190°C than that at RT in NC Cu. As the higher indentation hardness derived at −190°C corresponds to larger residual internal stress located inside the nanoscale grains, the free surface assisted stress-driven grain growth should be more pronounced than that at RT. Therefore, for samples having identical grain size before deformation, the additional extent of grain growth should be much larger at −190°C than that at RT.

In conclusion, by reevaluating the experimental observations reported by Zhang et al. [5], our analysis reveals that the extent of grain growth driven by pure mechanical stress may be significantly overestimated. A new physical mechanism of grain growth driven by both mechanical stress and free surface effect is proposed for NC metals. Whilst the dominant mechanism(s) is yet to be identified specifically, it is clear that several GB-mediated mechanisms may contribute to the additional grain growth. However, to what extent the free surface assisted stress-driven grain growth is related to the total grain growth observed under TEM remains a challenge.

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