Chapter 7

Grain Boundary Effects on Microstructural Stability of Nanocrystalline Metallic Materials

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Abstract

Grain boundaries play an important role in dictating the mechanical and physical properties of nanocrystalline (NC) materials because of the increased volume fraction of intercrystalline components as the grain size decreases. In general, grain boundaries have a high energy level and there exists a thermodynamic driving force to reduce the overall area of grain boundaries through grain coarsening, making NC material systems intrinsically unstable. Recent investigations also indicate that mechanical deformation can promote grain growth in NC material even at the cryogenic temperatures. In this chapter, first, the current investigation on the grain boundary structures of NC metallic materials is briefly reviewed and then the state-of-the-art of experimental results on the microstructural stability during deformation processes is discussed. Finally, several key issues for improving the microstructure stability of NC metallic materials and possible future work are discussed.

Keywords: nanocrystalline, grain boundary, microstructural stability, deformation mechanism, fracture

1. Introduction

A grain boundary (GB) in the homophase polycrystalline metal is an interface between two crystals of the same crystal structure [1, 2]. According to the dimensional scale of defects in polycrystalline materials, the GB is classified as a planar defect. GBs play an important role in the mechanical properties of polycrystalline metals [3, 4]. For bulk polycrystalline metals, the GB is considered to be stable during the plastic deformation processes and acts as a sink, source or obstacle to the dislocations [1]. With the decreasing microstructural length scale of polycrystalline materials, the volume fraction of atoms residing in or near the grain boundaries

increases. Experimental results indicate that GB width (δ) is approximately 0.5 nm for face-centered cubic (fcc) NC alloys and slightly larger than 1.0 nm for body-centered cubic (bcc) NC alloys [3, 5, 6]. Assuming the grains have the shape of spheres, the volume fractions of intercrystal regions and GB as a function of grain size (*d*) are shown in **Figure 1**. It can be seen from **Figure 1** that for nanostructured materials with grain size of 5 nm, nearly 50% of atoms will reside in or near the GB [7]. Therefore, nanostructured materials can be considered to compose of two parts: the core crystallites and a network of intercrystal regions (grain boundaries, triple junctions, etc.) [8]. In NC materials, grain boundary mediated processes, such as emission and absorption of dislocations by grain boundaries, grain rotation, and GB sliding will dominate the plastic deformation as the grain size is smaller than a certain critical value [9]. The properties of the nanostructure materials are thus determined not only by their reduced microstructural length scale, but also by the nature of their GB structures [10, 11]. Due to high volume fraction of the GB in NC materials, thermodynamic driving force exists to drive GB migration which results in the low stability of NC materials. The main objective of this chapter is to provide a comprehensive review of the experimental and simulation results on the microstructure instability of NC materials system under various deformation conditions.

This chapter is structured as follows: Section 2 describes the basic methods established in the field of GB structure to describe the GB structure. The section that follows addresses the GB

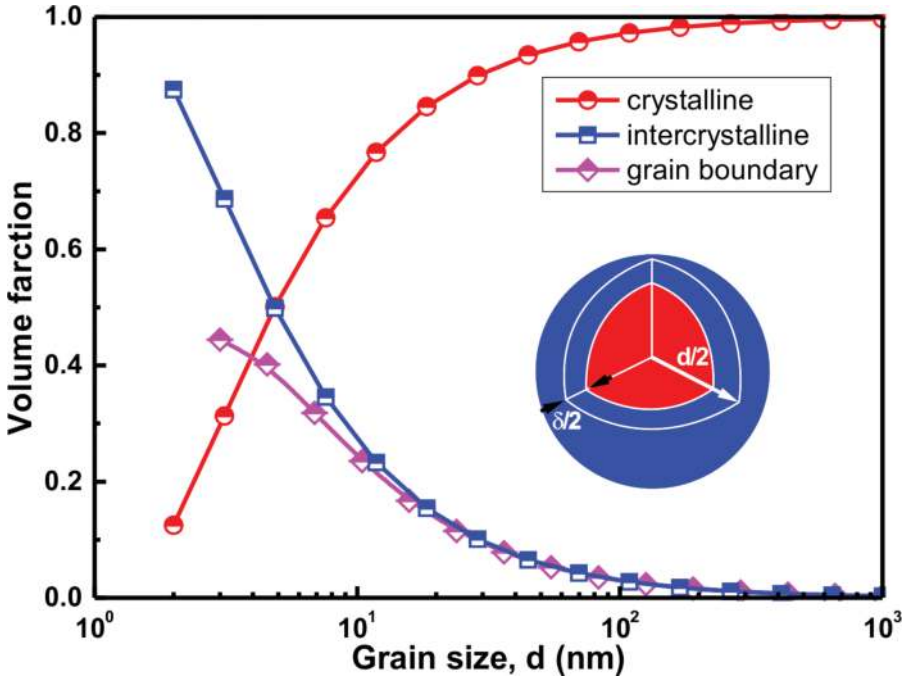

Figure 1. The contribution of different microstructural elements to the volume fraction as a function of grain size (*d*), assuming a grain-boundary thickness (δ) of 1 nm.

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stability phenomena in NC materials, the GB effect on the monotonic and cyclic deformation processes of NC materials. Finally, the application method to enhance the microstructural stability of NC and potential investigations in the future are discussed.

2. Grain boundary structure

For understanding the properties of NC materials, it is a prerequisite to have a detailed knowledge of the GB structure from the atomic (local) scale to the microstructural scale. A considerable effort has been made to understand the GB structure of polycrystalline materials, since the 1950s. To describe the GB crystallographically, a number of parameters must be defined. From a macroscopic perspective, a planar GB between two adjacent grains has five degrees of freedom. Four degrees of freedom are accounted for the crystallographic orientation of the rotation axis and the normal of the GB plane. The fifth is defined as the misorientation angle (θ) [12]. There are several criteria to classify the GB. According to θ value, GBs are typically classified to low-angle boundaries with $\theta \leq 15°$ and high-angle boundaries with $\theta > 15°$ [13]. The coherency of homophase low-angle GBs can be described through the dislocation model [14]. The degree of coherency is related to the spacing of misfit dislocations within the GB. Based on the relative orientation of the rotation axis and the GB plane normal, the GB can be classified as a tilt and twist boundary. If the rotation axis is perpendicular to the GB plane normal, this GB is called a tilt boundary, whereas if the rotation axis is parallel to the GB normal, the GB is defined as a twist boundary. Although tilt and twist GBs occupy only a small fraction of the GB phase space, they are frequently observed experimentally, which suggests that they are energetically favored over other types of GBs [15].

Numerous theoretical efforts have been made to characterize the GB structures. The widely used models to analyze and predict the atomistic structures of GB include the coincidence site lattice (CSL) model and the structural unit (SU) model. In the CSL model, a coincidence index (Σ) is the ratio of the volume of the CSL cell to that of the lattice unit cell. The reciprocal value of the Σ represents the fraction of the lattice points belonging to the abutting crystal. GB with lower value of Σ contains a higher density of coincident sites and is expected to have low energy. While in the SU model, those GBs with specific misorientation angles called favored GB is constituted only from one type of structural units. Any intermediary GB between two favored GBs can be described by a linear combination of SUs comprising one or several neighboring favored boundaries [16, 17]. Twenty-one <1 1 0> symmetric tilt GBs are investigated by Rittner and Seidman with atomistic simulations, using an embedded-atom method potential for low stacking-fault energy fcc metal [18]. They found that the favored boundaries are the $\Sigma = 1$ (0 0 1), $\Sigma = 27$ (1 1 5), $\Sigma = 11$ (1 1 3), $\Sigma = 3$ (1 1 1), $\Sigma = 9$ (2 2 1) and the $\Sigma = 1$ (1 1 0) interfaces. The structural units associated with each of these boundaries are denoted by A–E, as shown in **Figure 2**. To reduce the number of distinct SU, distortions exceeding 15% are occasionally permitted in the SU of the GB region.

The advent of transmission electron microscopy (TEM), especially in the development of high-resolution transmission electron microscopy (HRTEM) has provided us with a very powerful tool to explore the atomic structure of internal interfaces. **Figure 3** shows the HRTEM

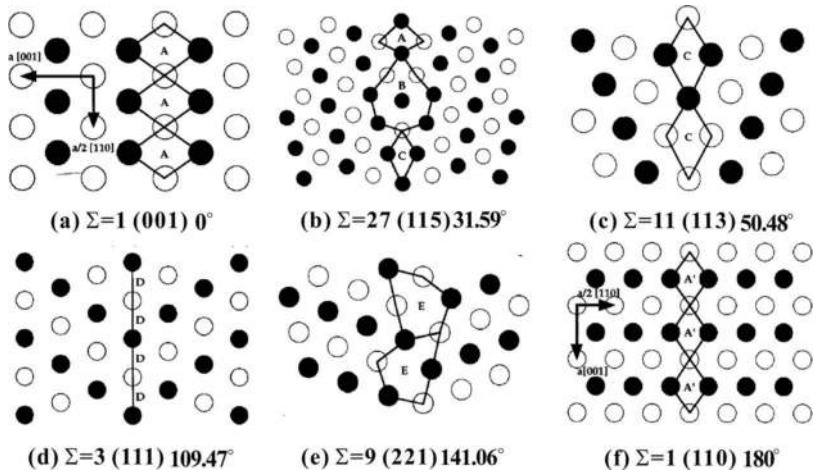

Figure 2. Equilibrium bicrystal interface structures of <1 1 0> symmetric tilt boundaries and two perfect crystal orientation [18]. The structures are viewed along the tilt axis [1 1 0], with the open and filled circles indicating atomic positions in alternate (2 2 0) planes [18].

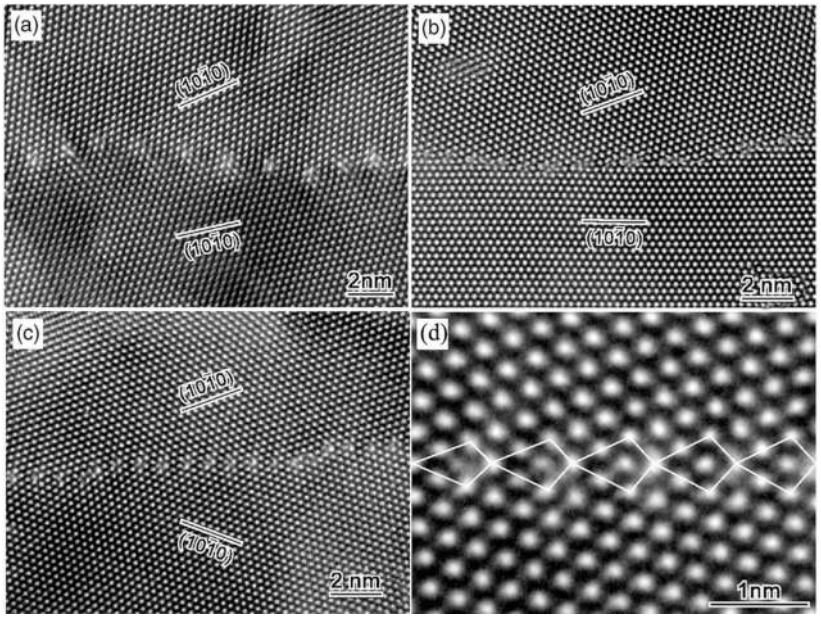

Figure 3. HRTEM images of [0 0 0 1]-tilt grain boundaries in ZnO bicrystals. (a) 10.6 ± 0.1° boundary composed of a dislocation array. (b) 20.1 ± 0.2° near Σ = 7 boundary having a facet structure. (c) 20.0 ± 0.2° near Σ = 7 boundary with a symmetric structure. (d) Higher magnification image of (c) with the boundary core structural units represented by quadrilaterals [19].

micrographs of the atomic structure of ZnO thin films [19]. It is evident that the low-angle GB is composed of edge dislocation along the GB plane as shown in **Figure 3(a)**. **Figure 3(d)** is the higher magnification of **Figure 3(c)**, having a periodic and nearly mirror symmetric character as clarified by drawings of SUs. The HRTEM greatly contributes to the experimental verification of the basic concepts of atomic structure of the GB [12]. TEM characterization results indicate that the GB of NC material is essentially the same as that of the coarse-grained materials. It should be noted that GB structures of NC materials are highly dependent on the alloy composition and processing steps in the manufacturing of NC materials.

3. Microstructural stability

3.1. Grain growth under monotonic deformation

Although NC materials show superior mechanical properties as compared to the coarse-grained counterpart, their application has been severely limited by their microstructural instability under the monotonic mechanical deformation [20–33]. For pure NC metallic materials, the grain coarsening process often occurs even at ambient temperatures [34–36]. Ames et al. reported on the observation of the room temperature grain growth in high-purity NC Pd with an initial grain size of about 10 nm [35]. They found a transition from an initially self-similar slow growth to abnormal grain growth. However, they argued that abnormal grain growth is a transient state since a monomodal grain size distribution was observed in the late stage of coarsening. Discontinuous grain growth in NC materials during the deformation processes seems to be a common phenomenon. Zhang et al. examined the effect of the temperature and sample purity on the grain coarsening behavior and found that many of the grains under the indenter have grown to several hundred nanometers while the unindented microstructure remains unchanged [20, 28], as shown in **Figure 4**. Due to the complex and large stress/strain field of the Vickers indenter is not yet known, the detailed mechanism of the grain growth is not known. However, the increased rate of growth at cryogenic than at the room temperature indicates that the growth is primarily mechanical, not diffusion-driven. In order to elucidate the effect of stress and strain on mechanically induced grain growth, Rupert et al. fabricated specimens with specially designed stress and strain concentrators to reveal the relative importance of these parameters on grain growth [24]. Statistical results of grain size in horizontal-hole specimens showed that grain growth occurred at both high strain region and high stress region. However, grain size was greater in the high stress region, indicating that the grain growth is driven by stress. Statistics results of grain size of angled-hole specimens demonstrated that grain growth was scaled with shear stress. Gianola et al. have investigated the tensile mechanical properties of 180-nm-thick NC Al films with grain size about 40 nm [22]. The specimens that exhibit high strength maintain their NC microstructure, the limited elongation, and the dramatic strain softening. By contrast, specimens that undergo discontinuous grain growth show intermediate strengths and the unexpected development of a region of extended plasticity. Statistical results of grain size outside of the deformed region of specimens that exhibited the grain growth are similar to the initial state indicating that the grain growth is directly tied to the applied stress or deformation in the sample [37].

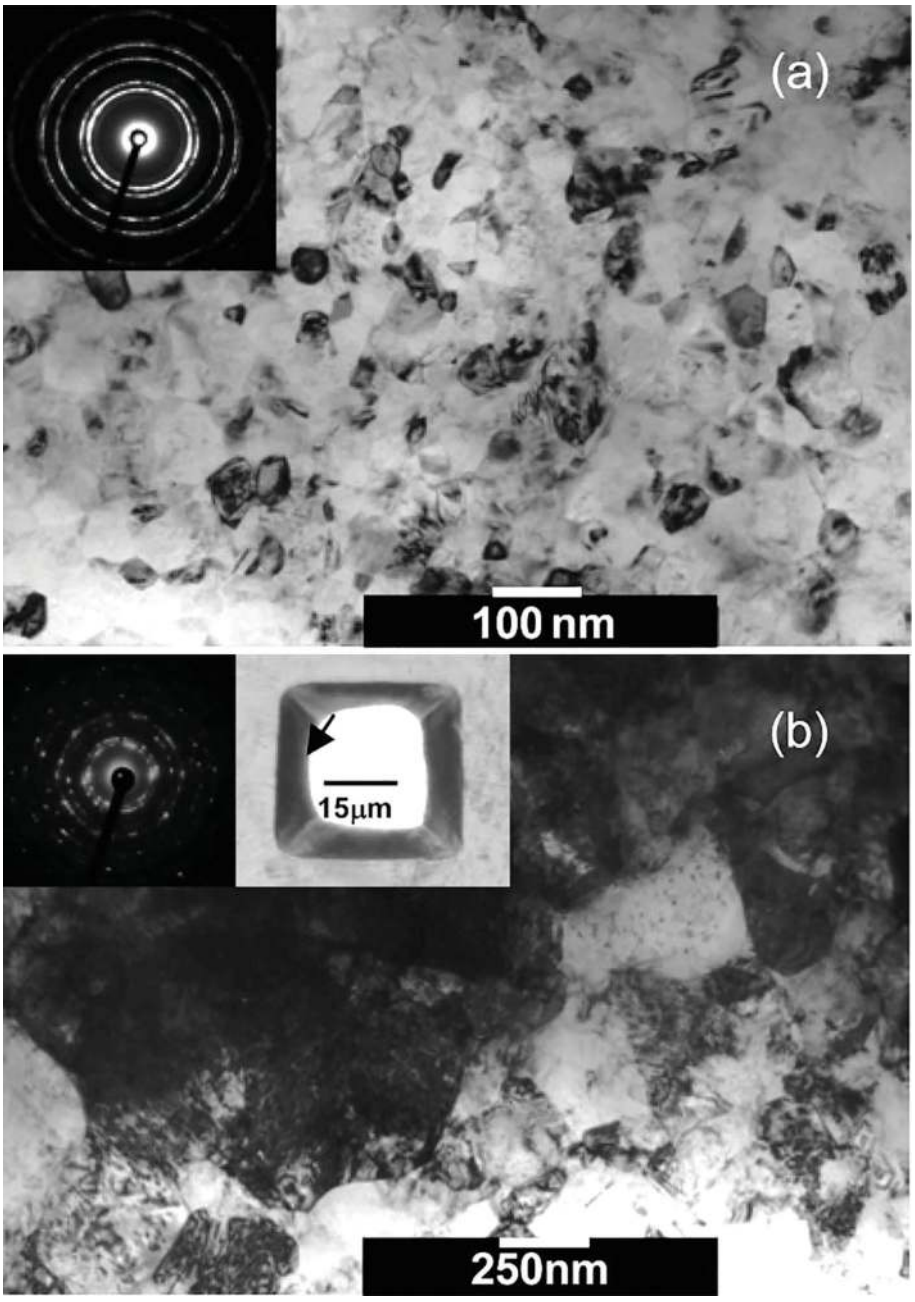

Figure 4. TEM images of Cu sample after indentation tests (a) in a region away from the indents, and (b) inside an indent made at room temperature with a dwell-time of 30 min [20].

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3.2. Grain growth under fatigue test

Grain growth is also observed during the cyclic deformation of NC materials [38–50]. Early fatigue experiments by Witney et al. on the NC Cu reported modest grain growth due to pull-pull cyclic loading [38]. They found that the protrusions stick out on the order of a micrometer which is far greater than the initial grain size, and extend several microns parallel to the surface, similar to extrusions formed during the fatigue test of a coarse-grained Cu. Detailed TEM studies of NC Ni-Fe alloy under cyclic deformation demonstrates that the grain coarsening is accompanied by the fatigue crack growth. Stress concentration at the crack tip causes the lattice reorientation under the cyclic deformation [41]. GB dislocations play a critical role in grain rotation and in the formation of subgrain in larger grains. Boyce and Padilla have reported the fatigue crack initiation and growth behavior in NC Ni, Ni-Mn, and Ni-Fe alloy [42]. They found localized regions of grain growth during fatigue loading. Coarsened Ni grains did not favor any particular orientation, while Ni-0.5Mn coarse grains showed a <1 1 0> preferred orientation. Their observations also suggest that grain stability is an important factor affecting the crack initiation and propagation process in these NC alloys. Meirom et al. reported fatigue-induced grain coarsening during crack propagation in NC Pt films with a strong <1 1 1> texture [43, 44]. They found a clear evidence of increased grain size in the crack wake and ahead of the crack tip. Coarsened grains underwent a nearly one order of magnitude increase in size compared with the as-received one. They also found that many of the grain boundaries in the Pt films are "low-angle" in character and the grain coarsening by the annihilation of low-angle GB through the dislocation slip near the crack tip [43, 44]. Recently, Zhang et al. investigated the fatigue behavior of 100-nm-thick NC Cu film on a polymer substrate [49]. They found that

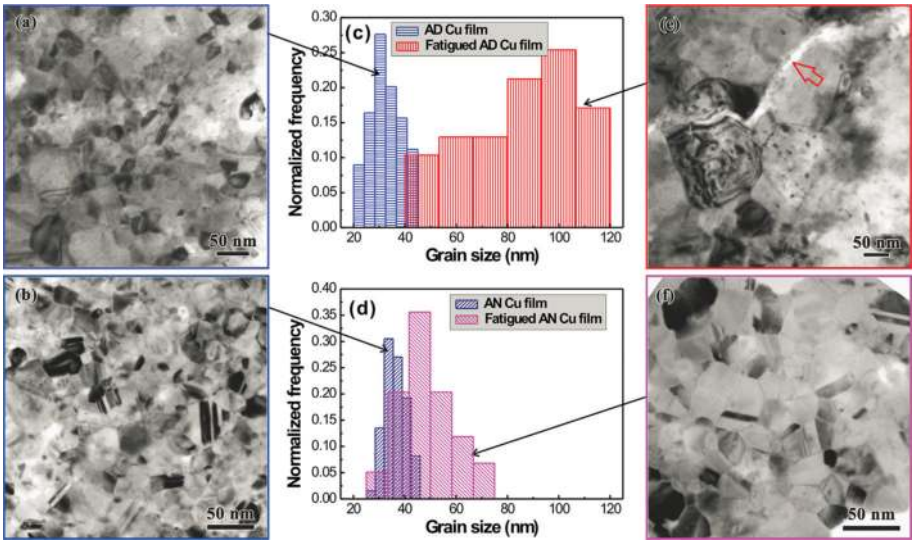

Figure 5. Microstructure of Cu films (a) as-deposited (AD) and (b) annealed (AN). (c and d) Statistical results of the grain size of the AD and the AN Cu films before and after fatigue test. Microstructure of Cu films after fatigue test (e) AD and (f) AN [49].

the mean grain size of both the as-deposited and annealed Cu samples increase after the fatigue test, as shown in **Figure 5**. However, the grain growth is greatly suppressed in the annealed NC Cu film, which leads to the enhanced resistance to the fatigue cracking as compared with that of the as-deposited one. The enhanced fatigue strength of Cu film after annealing may be related to the GB structure readjustment during the annealing process.

3.3. Grain boundary migration-theoretical model

Although the mechanical grain growth has been experimentally observed under various mechanical conditions, the underlying microstructural and atomic scale mechanisms are still open for debate. As mentioned in the introduction section, in an NC material, the fraction of atom residing at the GBs increases as the grain size decreases. Thus, GBs in NC materials promote the total free energy of the system. The reduction of this excess free energy through the removal of grain boundary area represents a large driving force for the grain growth. Grain growth in NC materials can be due to the rotation and coalescence of adjacent grains, as well as normal grain boundary movements. There are several theoretical models that have been proposed to describe the mechanically driven grain growth behavior, for example, stress-coupled GB migration [51–56] and grain rotation-induced grain coalescence [57].

The stress-coupled GB migration model is based on the argument that shear stress causes tangential movement of grains along GBs (GB sliding), and this produces a coupling with the normal motion of GBs (GB migration) [51]. Gutkin and Ovid'ko proposed a continuum disclination model for describing the stress-induced cooperative migration of an arbitrary tile GB [53–55]. The migrating GB was approximated by partial wedge disclination that can move under the applied shear stress, as shown in **Figure 6**. In the initial state, these GBs form two triple junctions. Under an applied shear stress, migration of GB3 from their initial position AB to a new position A′B′ occurs. Stress-induced migration of low-angle tilt GB3 results in the formation of two new triple junctions, A′ and B′. Straight-line defects (junctions) A, B, B′, and A′ are characterized by the disclination strength $\pm\omega$. The motion of the disclination produces rotational plastic deformation. The same is true for migration of two high-angle tile GBs with large angle gaps. It was shown that there is two critical stress, τ_{c1} and τ_{c2}, that controls the GB migration behavior. When the applied stress τ reaches τ_{c1}, the GB can migrate in the stable mode and their equilibrium position is determined by the level of τ. When $\tau > \tau_{c2}$, the GB migration becomes unstable when the GB propagation does not depend on the level of τ. In all cases, GB migration leads to the unstable growth of a grain at the expense of its neighbors. Energy methods calculation indicates that critical shear stresses strongly depend on the elastic modulus of the material, as well as on the strength of disclination-like defects appearing at the GB junctions in the process of GB migration.

For the case where the translational mode is mainly represented by GB sliding, Wang et al. suggested a theoretical model which describes the cooperative action of GB sliding and grain rotational deformation in mechanically loaded NC materials, as shown in **Figure 7** [25]. The grain rotation-induced grain coalescence model can be understood as follows [57]: with the applied force, GB dislocations glide results in the relative translational motion of GBs. However, the triple junctions impede the motion of GB dislocation. The blocked GB

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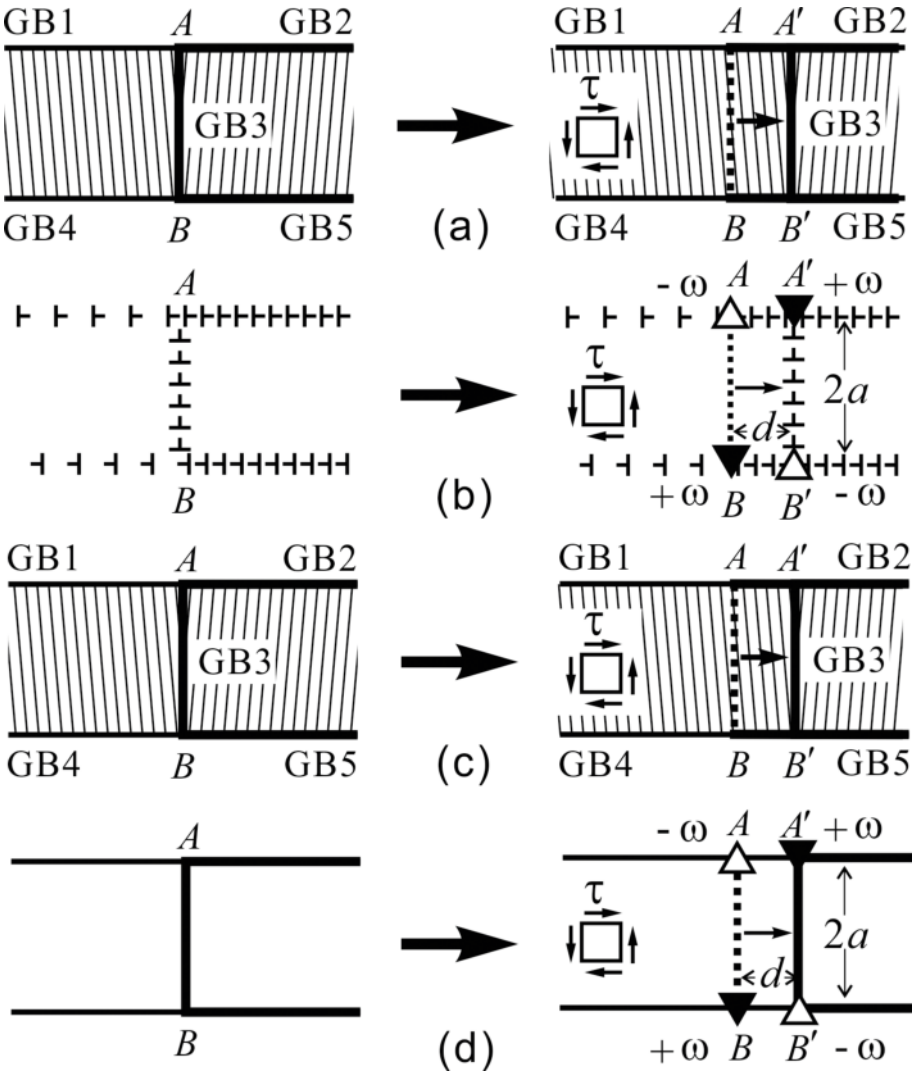

Figure 6. Schematic of stress-induced grain boundary (GB) migration (a and b) low-angle and (c and d) high-angle GB (GB3) by grain rotation through the glide of lattice dislocations (b) or motion of a dipole of wedge disclinations (d), respectively [54].

dislocation dissociates from the two climbing GB dislocation at the triple junction. With further plastic deformation, the dislocation split process happens repeatedly and the climbing GB dislocations form two dislocation walls along the GBs, which results in the rotation of the central NC grain. Multiple rotations bring the orientation of abutting grains closer together and reduce the GB misorientation angles, and even eliminate the GBs, leading to coalescence of smaller grains into larger ones.

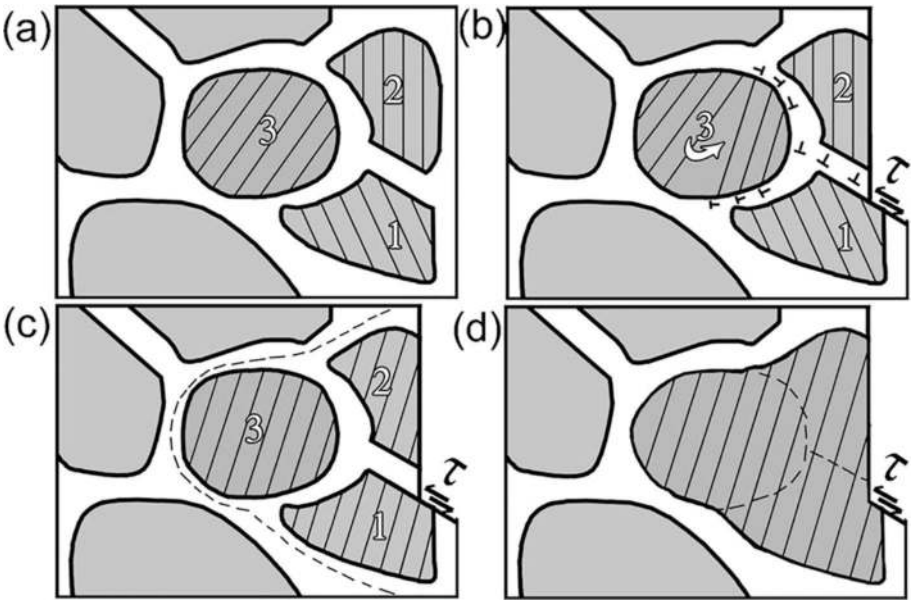

Figure 7. Combined grain boundary (GB) sliding and rotational deformation mode. (a) The GB of the nanocrystalline (NC) Ni dominated by high-angle GBs. (b) dislocations motion along GB impeded by the triple junction resulted in climbing GB dislocations, which caused the rotation of grain 3. (c) Repeated grain rotations leading to grain coalescence. (d) The formation of a larger grain with subGBs (highlighted by dotted line) [25].

3.4. Grain boundary migration-atomic scale mechanisms

Although theoretical models give some clues about the grain growth processes during mechanical deformation of NC materials, the exact microstructural scale and atomic scale mechanisms of the GB migration is still unclear. Quantitative information aimed to identify atomic scale mechanisms that reveal the influences of GB structure on the GB migration can be obtained by using in situ HRTEM and molecular dynamics (MD) simulation methods. Haslam et al. explored how grain rotation can induce grain coarsening [58]. They found that grain rotation decomposes a GB into multiple but distinct dislocations, which can then move by dislocation slip; these dislocations can annihilate at GBs or remain embedded within grains if the applied stress is relieved. The grain rotation mechanism purports that only some grains are able to rotate and thus coarsened grains can maintain their outer boundaries. This is consistent with the experimental result that GB of coarsened grain is characterized by low-angle boundaries [43, 44]. MD simulation of nanoindentation of NC Al films with a mean grain size of 7 nm showed that the grain rotation may be competing with the GB migration and the GB migration is likely dominant, as shown in **Figure 8** [59]. During cyclic deformation of 20-nm-thick Au thin film, Luo et al. found that grain growth of NC Au is closely correlated with twin formation [60]. Based on the atomic scale observations, as shown in **Figure 9**, they revealed that the formation of nanotwins is an effective way to assist grain coarsening. The grain coarsening process can be described as follows: the mutual nucleation of nanotwins near the GB changes the local

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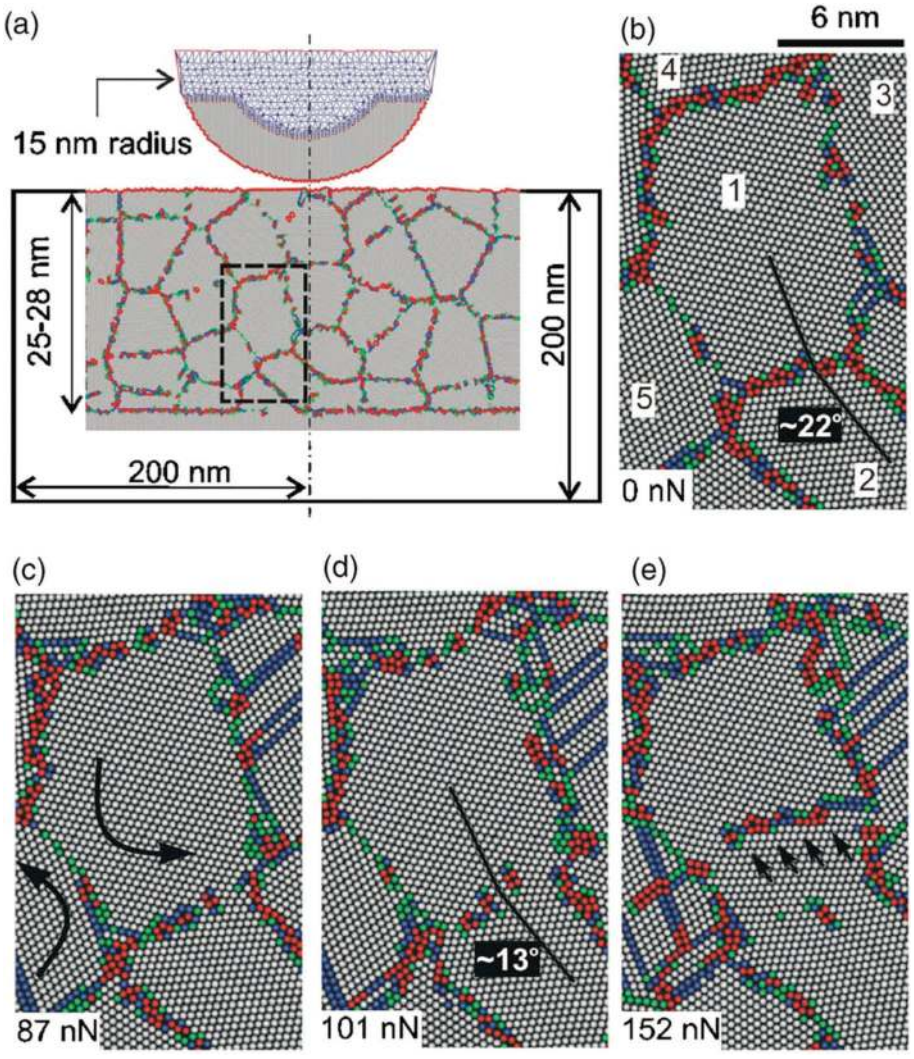

Figure 8. Atomic-level detail of the coalescence of grains. Deformation of Al film with grain size of 7 nm. (a) Contact zone before indentation simulation. (b–d) Rotation of grain 1 due to grain boundary (GB) sliding and transformation of the structure of the GBs. (e) Dissociation and migration of the GB between grains 1 and 2 [59].

grain misorientation and results in the GB dissociated into smaller segments, which is more mobile than their parent one.

Recently, by using in situ HRTEM, Luo et al. have reported the adjustment of GB structures of Cu film during a self-driven GB migration, which involves GB dissociation, partial dislocation emission from GB, and faceting/defaceting [61]. Furthermore, they revealed that GB migration ability is closely related to the local GB segment consisting of "hybrid" structural units.

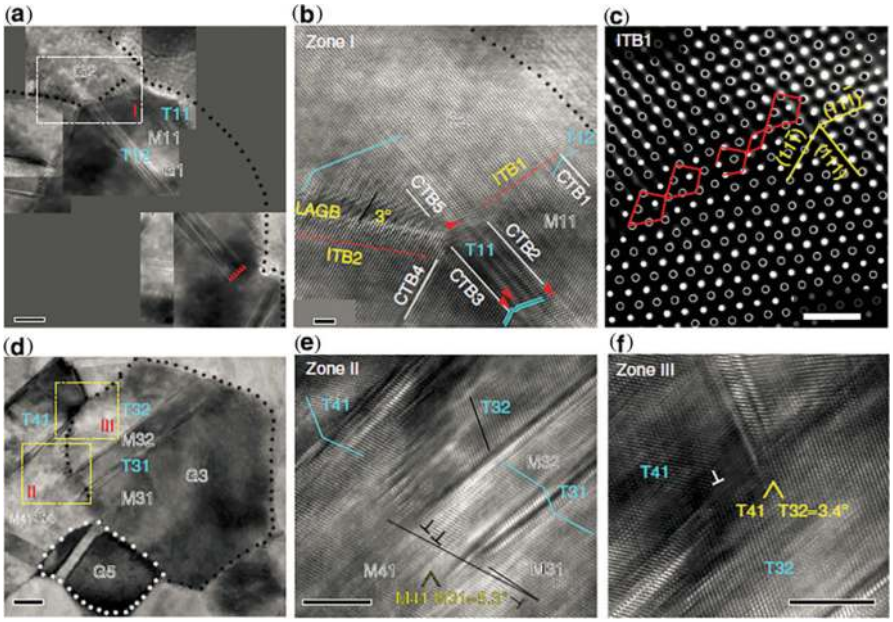

Figure 9. GB dissociation induced by twin formation. (a and d) TEM images of two typical examples of large grains with parallel multi-twins in the fatigued samples. (b and c) and (e and f) The corresponding details of the microstructures, respectively. Scale bars: (a and d–f) 5 nm, (b and c) 1 nm [60].

4. Summary and future work

As mentioned in the introduction section, the high volume fraction of atoms resided at GB makes the microstructural stability problem intrinsic to NC materials. In general, two approaches are used to stabilize the grain structure: by kinetically hindering the GB mobility or by thermodynamically lowering the GB energy through solute segregation [62]. Studies have shown that providing a short annealing treatment to allow for grain boundary relaxation can increase the fatigue life of Cu films [49]. HRTEM investigation on the detailed GB characters pre- and post-annealing processes will give more clues about the underlying mechanism. Recently, methods for stabilizing NC materials by the control of interface structure was reviewed by Lu [63]. It has been argued that nanostructures with a high-density of coherent twin boundaries (CTB), which are low energy, low mobility boundaries with a high degree of crystallographic ordering, would provide the required resistance to the thermal coarsening while enhancing the strength. The addition of solute atoms (alloying) can pin the GB and/or lower the GB energy, improving the microstructural stability of NC [64, 65]. However, due to the complexity induced by alloying, more experimental and theoretical investigations on the structural characteristics need to be done in the future.

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