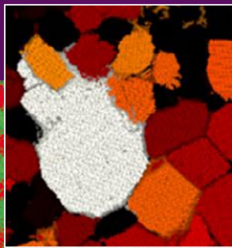

Fatigue and fracture of nanostructured metals and alloys

Lei Lu,* Qingsong Pan, Khalid Hattar, and Brad L. Boyce*

Metals and alloys with nanoscale structural features (such as grain size or twin thickness <100 nm) exhibit exceptional strength and unusual deformation mechanisms. But, the suppressed dislocation slip, grain-boundary instability, and limited strain hardening in these nanostructured metals can be detrimental to fatigue and fracture properties. In this article, recent advances in understanding the structural origins of fatigue and fracture resistance of nanocrystalline and nanotwinned metals and alloys are reviewed. Based on this understanding, microstructural engineering strategies, such as gradient grain size, controlled boundary mobility, or hierarchical nanotwins, alter the deformation modes and provide promising paths to develop nanostructured materials with improved fatigue and fracture properties.

Introduction

Most service failures of metallic structural components are caused by fatigue and fracture under complex loading conditions.[1] Accordingly, new strategies that mitigate these failure modes through material design will result in improved component reliability and lifetime cost savings. In coarse grained (CG) metals, the fundamental deformation processes governing fatigue and fracture, such as dislocation motion and twinning,[2] can be suppressed or altered in nanostructures with at least one characteristic length scale < 100 nm,[3,4] providing opportunities to improve performance of engineering metals and alloys in service. While several nanostructuring strategies, including nanoprecipitates,[5,6] nanolaminates,[7,8] and nanoporosity,[9,10] can influence fatigue and fracture behavior, this article focuses specifically on two broadly studied nanostructure cases: nanocrystalline and nanotwin structures.

Under fatigue loading, CG metals and alloys typically exhibit cyclic strain localization, accumulative damage with the formation of persistent dislocation patterns, and severe surface roughening, all leading to fatigue crack nucleation.[1,2] Simply refining the CG grains down to submicron or nanoscale (i.e., ultrafine grained [UFG] and nanocrystalline [NC, also described as "nanograined"]), can remarkably improve the strength and enhance the high-cycle fatigue limit, compared to their CG counterparts.[11–13] Moreover, while the high-cycle fatigue endurance limit (e.g., stress to cause failure in 10^7 cycles) of CG metals is largely independent of initial grain size,[1,2] for UFG metals, grain size reduction leads to substantial increases in fatigue endurance limit, a trend that may saturate or even reverse in the nanocrystalline regime.[14] However, UFG and NC metals typically suffer from the following limitations: (1) substantially suppressed dislocation slip and limited deformation capacity;[15] (2) microstructural instability resulting in grain growth;[13,16] as well as (3) modest strain hardening and correspondingly limited ductility, fracture toughness, and low-cycle fatigue resistance under strain control.[11,13,17–20]

To address these issues, researchers have focused on several hierarchical modifications to nanostructured alloys, including heterogeneous gradient/bimodal grain structures,[21–23] heterogeneous grain-boundary (GB) chemistry/structure,[24] and nanotwinned (NT) materials.[25–29] In parallel, the development of several unique *in situ* nano- and micromechanical testing techniques[16,30–32] have allowed for a more detailed understanding of fatigue and fracture behavior of nanocrystalline and nanotwinned metals and alloys.

In the following sections, recent advances in the understanding of fatigue and fracture behavior of nanocrystalline and nanotwinned metals and alloys are reviewed with a

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doi:10.1557/s43577-021-00054-y

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particular emphasis on the novel fatigue and fracture properties of nanotwinned metals. Finally, the challenges and prospects for developing fatigue and fracture resistance through hierarchically nanostructuring are addressed.

Fatigue and fracture of nanocrystalline metals and alloys

Key reviews on the subject of fatigue response in UFG[33] and NC metals[34] indicate that high-cycle fatigue response of numerous NC metals is closely associated with fatigue-induced grain growth. Post-mortem inspection using the scanning electron microscope (SEM) and transmission electron microscope (TEM) shows that fatigue-induced grain growth primarily occurs at the site of crack initiation, and to a lesser extent, along the entire flank of propagating fatigue cracks of fatigued NC metals.[35] To eliminate the possibility that such behavior was induced by post-mortem specimen preparation using focused ion beam sectioning,[36] fatigue-induced grain growth has been directly observed using a new *in situ* TEM high-cycle fatigue capability. As shown in the sequence of bright-field images at different stages of the fatigue life (**Figure 1**a–c) and the corresponding crystal orientation map (Figure 1d), grain growth from the 10-nm regime to 100-nm regime preceded the crack initiation at 840,000 cycles. Details of the methodology utilized and other examples of *in situ* TEM fatigue results can be found elsewhere.[30] In a parallel effort, a nondestructive synchrotron x-ray diffraction technique was also developed to observe the progression of localized anomalous grain growth associated with *in situ* fatigue loading.[16,31] Both *in situ* observations demonstrate that the cyclic strain localization induced grain growth precedes fatigue crack initiation and that the subsequent fatigue crack initiation occurs at the regions of grain growth. It suggests the possibility of impeding fatigue crack initiation via the suppression of grain coarsening.

Currently, there are no phenomenological models to describe the relationship between fatigue loading and grain growth. However, atomistic modeling has recently confirmed fatigue-induced grain growth, albeit under low-cycle fatigue conditions. In one such study, NC Al with 5- and 10-nm grain sizes were cycled for 10 cycles, resulting in substantial grain rotation, and evolution of the GB character, including an increase in special GBs (esp. Σ3 and Σ11) for the 5-nm case.[37,38] Molecular dynamics (MD) simulations of NC Ni repeatedly cycled to either 0.5% or 1% strain for 200 cycles[39] revealed that fatigue-induced grain growth was more pronounced than that under either constant strain (stress relaxation) or unstrained thermal exposure (**Figure 2**).[39]

With regard to fracture behavior in ultrafine grained and nanocrystalline metals, reports are limited since the accurate quantification of fracture toughness is often limited by the finite form-factors available for NC metals.[40,41] However, tensile ductility measurements do suggest a general decrease of toughness with decreasing grain size.[42,43] Reduced ductility has been verified by several models, due to both the suppression of dislocation-mediated plasticity in NC systems[44,45] and quasi brittle intergranular fracture with fine/shallow dimples and cracks nucleated and propagated along high density high-angle GBs.[42,46,47]

In addition to GB stability influencing fatigue behavior, as previously discussed, there is a growing recognition of the importance of GB stability in controlling fracture behavior at the nanoscale. Atomistic modeling has shown that GBs that migrate by strongly coupling to shear stress can substantially increase the local resistance to crack

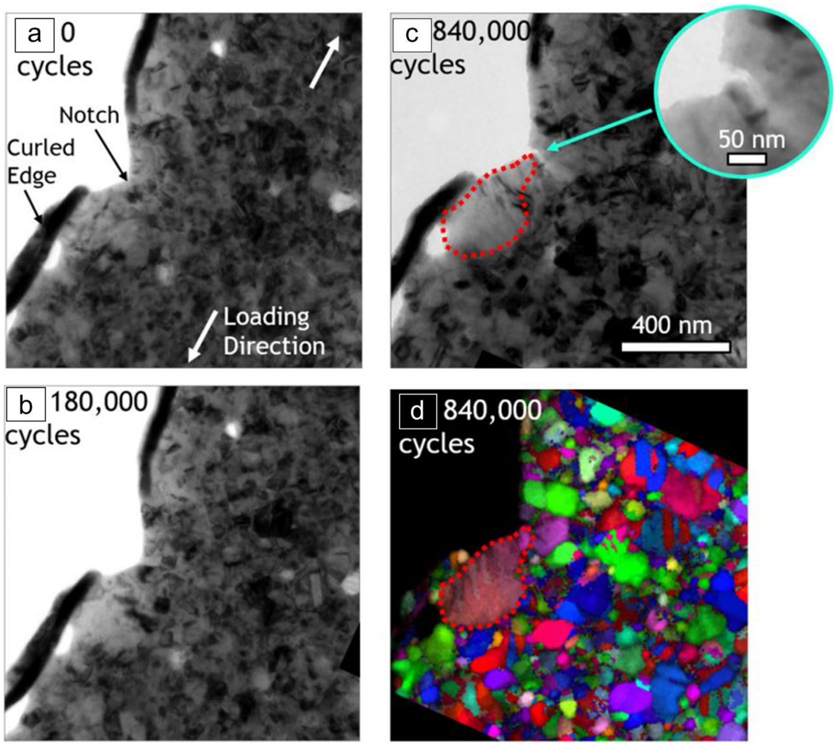

Figure 1. (a–c) Snap shots from a bright-field *in situ* transmission electron microscope high-cycle fatigue experiment of a notched nanocrystalline Cu tensile thin film, which resulted in localized grain growth (red outline) and nanocrack nucleation (inset). (d) Automated crystal orientation mapping showing grain growth (red outline) associated with fatigue crack initiation. Fatigue testing was performed at 200 Hz using a dogbone-shaped electron-transparent tensile film loaded on a microelectromechanical systems push-to-pull device with the Hysitron PI-95 picoindenter.[30]

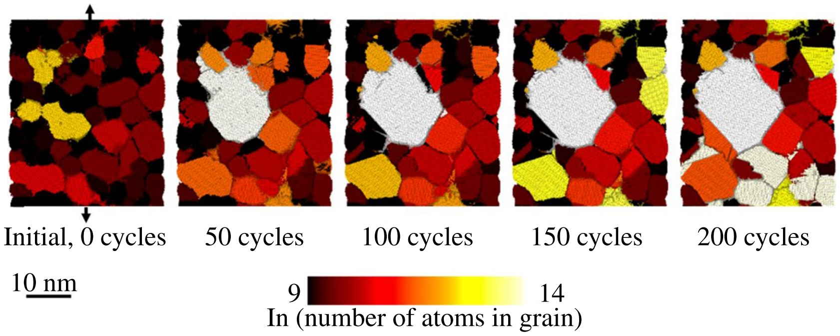

Figure 2. Cycle-induced grain growth observed in nanocrystalline Ni using molecular dynamics simulations. Grains are colored according to their size. In these simulations, a 35-nm diameter, 41-nm long Ni nanowire consisting of ~ 500 equiaxed grains was loaded to 1% strain and unloaded for up to 200 cycles.[32,39]

propagation.[48] Moreover, under some conditions, GB migration can serve to locally close and potentially heal cracks.[49]

To address microstructural stability for improved thermal and mechanical performance, there has been extensive interest in controlling GB mobility through microstructural modifications. In addition to traditional kinetic mitigation strategies such as Zener pinning or solute drag, there has also been an interest in thermodynamically stabilized NC metals.[50] In these binary alloys, a solute is selected for its propensity to segregate at the GBs and lower the energetic driving force for grain growth. While such strategies have resulted in grain structures that resist thermally driven evolution, some solutes can simultaneously embrittle the GB.[51,52] Such GB embrittlement has been observed experimentally; however, modest solute content may lead to inhomogeneous embrittlement and subsequent distributed stable nanocracks rather than a single catastrophic crack.[53] Such distributed cracking is a known toughening mechanism.

Recent studies also have shown ductility enhancement in nanostructured metals through the introduction of heterogeneous grain size.[21–23] These heterogeneous nanostructured metals typically have the smallest nanocrystalline grains at the sample surface with a gradient to coarser grains in the interior,[21–23] as can be produced by techniques such as surface mechanical attrition treatment.[54,55] The superior ductility primarily originates from strain gradients, which induce the accumulation and interaction of new dislocation structures, unique interfacial behavior, and resulting strain hardening.[22,23] Under cyclic loading, the cyclic elastic and plastic strains are spatially inhomogeneous but smoothly graded, thereby delocalizing deformation with controlled homogeneous or abnormal grain coarsening.[56] This results in a combination of both low-cycle and high-cycle fatigue resistance that is not achieved in their homogeneous NC counterparts.[56–58] The improved crack propagation resistance arises from crack tip blunting in the CG and more homogeneous stress field with the lower stress concentration ahead of the crack tip in the gradient structure.[59,60] In summary, both engineering the spatial distribution of nanostructure and altering heterogeneous GB chemistry and structure by alloying are effective in enhancing the resistance to fatigue and fracture. A specific structural modification that has shown substantial promise in improving fatigue and fracture resistance is the incorporation of a high density of nanotwins, explored in the next section.

Fatigue and fracture of nanotwinned metals and alloys

Unlike random high-angle GBs in NC metals, the coherent twin boundary (CTB) is a special type of low energy interface, exhibiting a mirror refection of atomic arrangement on both sides.[15] Without changing the grain size, introducing a high density of nanoscale CTBs (i.e., NT) in grain interiors has been recognized as another novel route of achieving a combination of high strength, ductility,[25,29,61] and fatigue and fracture resistance.[62,63] This exceptional combination of properties has been attributed to the unique interaction between dislocations and twin boundaries (TBs) at the nanometer scale. **Figure 3**a–c show the typical overall three-dimensional microstructure of highly oriented NT metals synthesized by direct-current electrodeposition[62,64,65] and magnetron sputtering.[27,66,67] In these materials, the polycrystalline grains are mostly columnar in shape, with sizes ranging from a few hundred nanometers to several microns, and are subdivided by highly oriented nanoscale twin lamellae (Figure 3b–c). Coherent TBs in all grains are roughly oriented perpendicular to the growth direction (or parallel to the deposition plane). NT metals with highly oriented nanoscale twins exhibit strong plastic deformation anisotropy as the activation of slip system and dislocation-TB interactions are controlled by the loading direction with respect to TBs.[68,69] For instance, when the loading axis is approximately parallel to TBs, dislocations with their slip plane inclined to the twin plane, but slip Burgers vector parallel with the twin plane (e.g., BCD in twin, BCD^T in the matrix and CB, as described the Thompson tetrahedra model and shown in Figure 3f) are activated and bowed out from TB–GB junctions[68] (i.e., namely threading dislocations with a hairpin-like loop configuration and shown in Figure 3d), like those observed in nanoscale thin films and multilayer materials.[70]

Figure 4 shows that both high fatigue limit under stress control and enhanced fatigue life under strain control are simultaneously achieved in the NT structure,[64,65] which is in distinct contrast to the tradeoff trend between high-cycle and low-cycle performance of CG, UFG, and NC counterparts.[11,13]

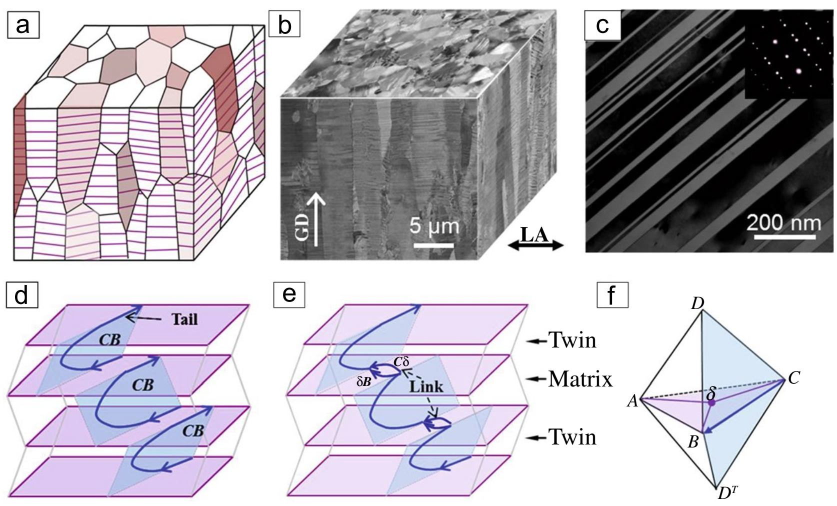

Figure 3. (a) Three-dimensional illustration of the microstructure of metals containing highly oriented nanotwins (NTs). (b, c) Three-dimensional stereographic scanning electron microscope and cross-sectional transmission electron microscope images of as-deposited NT Cu. Schematics of threading dislocations in tensioned NT (d) and correlated necklace dislocations (e) in cyclically deformed NT metals with the twin planes parallel to the loading axis. (f) Double Thompson tetrahedron representation of the slip systems in matrix (upper) and twin (lower).[62,71] GD denotes the growth direction, which is normal to the deposition plane. The double arrow denotes the cyclic loading axis (LA).

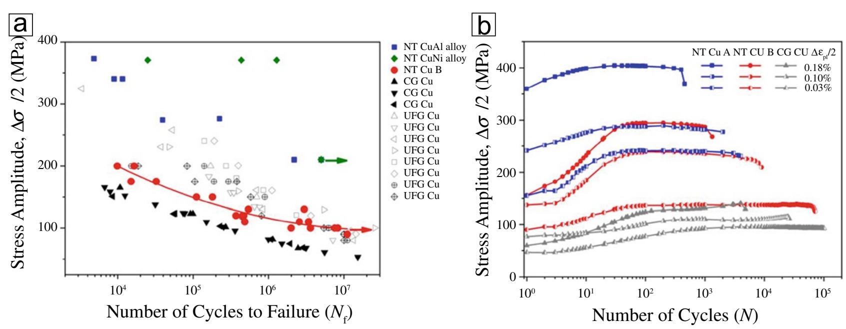

Figure 4. High-cycle and low-cycle fatigue properties of nanotwinned (NT) metals.[65,67] (a) Dependence of high-cycle fatigue life (N_f) on the stress amplitude ($\Delta\sigma/2$) of NT Cu and NT Cu alloys under stress control; (b) cyclic stress response ($\Delta\sigma/2$) of NT Cu at constant plastic strain amplitude $\Delta\varepsilon_{pl}/2$. For comparison, $\Delta\sigma/2$ versus N_f data of their course grained and ultrafine grained counterparts are also included.

As shown in Figure 4a, the fatigue limit of electrodeposited NT Cu is almost twice that of CG and nearly comparable to that of UFG counterparts. In addition, alloying has been proven to result in a higher fatigue limit as demonstrated in the NT CuAl and NT CuNi alloys, which also have smaller twin thickness and corresponding higher strength.[6]

Under constant plastic strain amplitude ($\Delta\varepsilon_{pl}/2$), the cyclic stability with constant stress amplitude (i.e., $\Delta\sigma/2$) is maintained in NT Cu after an initial short cyclic hardening stage (Figure 4b),[65] which is fundamentally distinct from cyclic hardening of CG[1] and continuous cyclic softening of UFG Cu[18,72] under strain control. Additionally, the stable $\Delta\sigma/2$ level increases with increasing $\Delta\varepsilon_{pl}/2$ and/or decreasing twin thickness. The enhanced fatigue life is achieved in NT Cu with a larger grain size, which is approximately comparable with that of CG counterpart at the same $\Delta\varepsilon_{pl}/2$.[13,58]

NT samples with highly oriented nanoscale twins exhibit history-independent, stable cyclic response with a one-to-one correspondence between $\Delta\sigma/2$ and $\Delta\varepsilon_{pl}/2$, independent of $\Delta\varepsilon_{pl}/2$ and cyclic number under a series of step-by-step increasing and decreasing $\Delta\varepsilon_{pl}/2$ (**Figure 5**a).[62] It is in contrast to conventional history-dependent, unstable cyclic response of materials associated with cumulative microstructural damage and strain localization.[2,13]

In the case with TB parallel to the cyclic loading axis, NT structures are stable, without detectable changes in TB morphologies, TB spacing, grain size, and texture before and after cyclic deformation. This is attributed to a new type of single slip and stable correlated necklace dislocations (CNDs) detected in NT,[62] as shown in Figures 3e and 5b. Under cyclic loading, most CNDs are formed by linking single slip threading dislocations in neighboring twin channels through dissociation and merging of their misfit tails on TBs, provided that the nanotwins are tilted within about 15 degrees of the cyclic loading axis.[62,73] CNDs with full burgers vector can move collectively back and forth along CTBs in micron-scale-length twin channels, without accumulating any damage to the CTBs. The CND motion is markedly distinguished from the detwinning controlled cyclic deformation of magnetron sputtered NT Cu and Cu alloys samples with submicron-sized grains, a distinction that would benefit from further in-depth study.[66,67]

The reversibility of CNDs accommodates the cyclic plastic strain in adjacent twins, thus resulting in less surface roughening with smaller surface height fluctuation (~ 300 nm) of zigzag slip bands observed on the NT-Cu samples cycled to failure.[66,67] Such unusual delocalized cyclic behavior governed by mobile, single slip CNDs without destroying the stability of NT structure contributes to the fatigue properties of NT metals, with the stable and history-independent cyclic response, longer fatigue life and higher fatigue limit.[62,64,65]

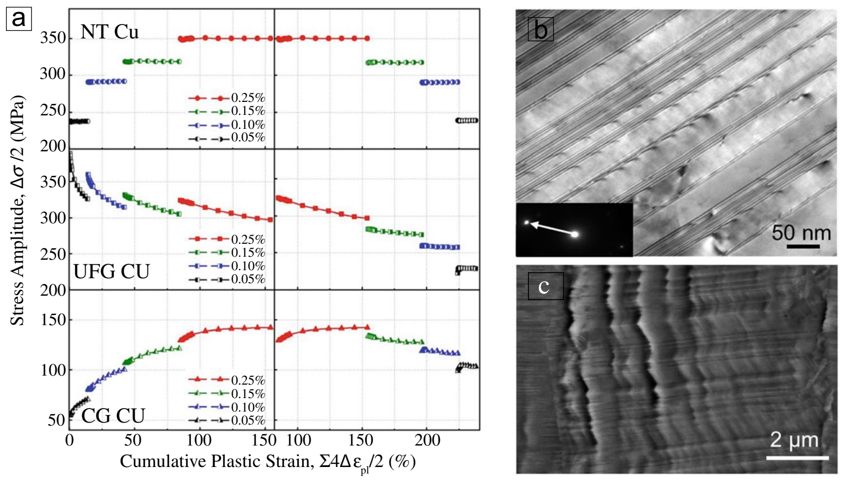

Figure 5. History-independent cyclic deformation behavior of nanotwinned (NT) Cu.[62] Cyclic stress response as a function of the cumulative plastic strain $\Sigma\Delta\varepsilon_{pl}/2$ of NT Cu, ultrafine grained Cu and course grained Cu cyclically deformed in stepwise increasing $\Delta\varepsilon_{pl}/2$ from 0.05% to 0.25% and decreasing back to 0.05%, with cyclic numbers of 70 at each $\Delta\varepsilon_{pl}/2$. (b, c) Cross-sectional transmission electron microscope and scanning electron microscope observations of NT Cu after cyclic loading, showing typical segments of correlated necklace dislocations overlapping with moiré fringes of twin boundaries (TBs) uniformly distributed under two-beam diffraction condition with $g = [\bar{2}\ 0\ 0]_M$ (inset in b) and surface zigzag slip bands across TBs.

Likewise, NT structures are more resistant to direct void nucleation than NCs with general GBs in the fracture tests, owing to the low energy of CTBs itself, the transmission of many types of dislocations across CTBs, and the effective release of stress concentration along CTBs by diverse dislocation-CTB interactions.[63] Furthermore, once a void/crack nucleates, its extension can also be effectively retarded by several unique crack-TB interactions induced toughening mechanisms observed in the NT film. These include crack tip blunting/arresting through dislocation emission along CTBs, periodic zigzag deflection of the crack path by CTBs and nanoscale twins serving as ductile crack-bridging ligaments.[63] As a result, the critical J-integral has been observed to increase with decreasing twin thickness of pulsed electrodeposited NT films with constant grain sizes.[74] This size dependence is in marked contrast to the grain size effect in NC metals.[15] NT structures with a large grain size were found to exhibit a higher fracture resistance, which was attributed to the enhanced crack tip plasticity, reduced number of microvoid nucleation sites and larger crack path tortuosity.[75]

Concluding remarks and perspective

This article presents a brief overview of recent studies on the fatigue and fracture of NC and NT metals and alloys. The emerging consensus is that the fatigue and fracture resistance of homogeneous nanocrystalline pure metals are limited by their suppressed dislocation plasticity, unstable grain structure, and modest strain hardening capacity. To address these issues, hierarchical and chemical modifications to NC metals (e.g., tailoring gradient nanostructure, introducing chemically modified stabilized grain boundaries, and incorporating a high density of CTBs at the nanoscale) are demonstrated to enhance the resistance to fatigue and fracture. In particular, by tailoring hierarchical features of NT structure, such as grain size, spacing and orientation of CTB, superior fatigue and fracture resistance are achieved in NT metals (e.g., the history-independent, stable cyclic response, longer fatigue life, higher fatigue limit and fracture toughness).

Despite the considerable progress made in understanding and controlling the fatigue and fracture behavior of nanocrystalline and nanotwinned metals and alloys, there are still numerous unresolved topics ripe for future investigations. For instance, the available processing pathways to produce hierarchical nanostructured alloys remain quite limited, constraining their practical applications. Moreover, as a result of constrained processing paths, systematic studies of the isolated effects of individual microstructural parameters such as grain size, twin thickness, twin orientation, GB characteristic, and chemical impurity on their intrinsic fatigue and fracture properties are limited. With a deeper understanding of the physical mechanisms contributing to fatigue and fracture of nanostructured metals, and with an expanding palette of methods to synthesize hierarchical microstructures that are resistant to fatigue and damage progression, this class of alloys are expected to grow in utility and importance.

Acknowledgments

L.L. acknowledges financial support by the National Natural Science Foundation of China (NSFC, Grant Nos. U1608257 and 51931010), the Key Research Program of Frontier Science and International partnership program (Grant No. GJHZ2029), CAS, and LiaoNing Revitalization Talents Program (Grant No. XLYC1802026). Q.P. acknowledges support by NSFC (Grant Nos. 51601196, 52071321) and the Youth Innovation Promotion Association CAS (Grant No. 2019196). B.L.B. and K.H. were supported by the US Department of Energy (DOE) Office of Basic Energy Science, Materials Science and Engineering Division. B.L.B. and K.H. would like to acknowledge D. Bufford for his work in obtaining Figure 1, C. Kunka for compiling data for Figure 2, and acknowledge use of the microscopy facilities at the Center for Integrated Nanotechnologies for this work. Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International, Inc., for the US DOE's National Nuclear Security Administration under Contract No. DE-NA-0003525. The views expressed in the article do not necessarily represent the views of the US DOE or the United States Government.

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