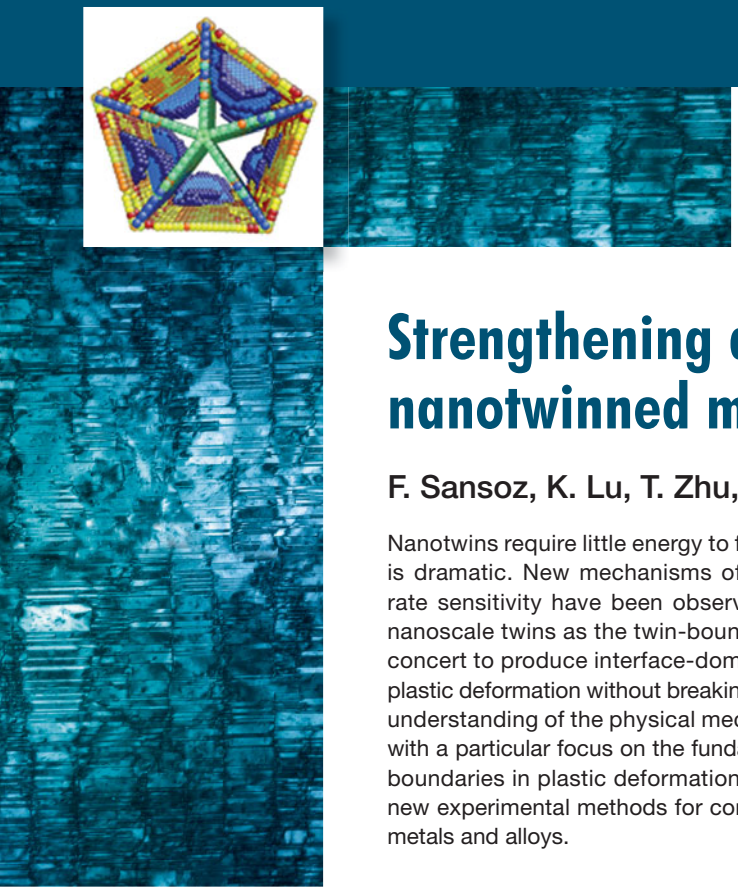

Strengthening and plasticity in nanotwinned metals

F. Sansoz, K. Lu, T. Zhu, and A. Misra

Nanotwins require little energy to form in metals, but their impact on strength and ductility is dramatic. New mechanisms of strengthening, strain hardening, ductility, and strain-rate sensitivity have been observed in nanowires, films, and bulk materials containing nanoscale twins as the twin-boundary spacing decreases. These mechanisms can act in concert to produce interface-dominated nanomaterials with extreme tensile strength and plastic deformation without breaking. This article reviews recent theoretical and experimental understanding of the physical mechanisms of plasticity in nanotwin-strengthened metals, with a particular focus on the fundamental roles of coherent, incoherent, and defective twin boundaries in plastic deformation of bulk and small-scale cubic systems, and discusses new experimental methods for controlling these deformation mechanisms in nanotwinned metals and alloys.

Introduction

The exceptional macroscopic tensile strength and high ductility of nanotwinned (NT) metals,[1] comprised of coherent twin boundaries (CTBs) with nanometer-scale spacing, are now well established. Growth and deformation nanotwins have been shown to form ubiquitously in many materials, including pure elements and engineering alloys of various shapes and sizes, from bulk to thin films, and nanowires (NWs).[2] Elucidating the microscopic origin of strength, plasticity, and twin-size effects in NT metals has become an important objective for fundamental research and engineering applications. New NT metals and alloys have been synthesized, and new experiments have flourished in recent years. In parallel, new high-resolution diffraction tools and more sophisticated atomistic simulations have been developed to probe the microstructure of deformation in NT metals at the atomic scale.

This article highlights recent important advances enabled by these experimental techniques and atomistic models. First, we discuss the phenomenon of strain hardening in low-dimensional metals such as twinned face-centered-cubic (fcc) NWs where, in the absence of grain boundaries (GBs), strengthening and enhanced plasticity emerge from the complex interplay between twin boundaries (TBs) and free surfaces. Next, we focus on the microscopic mechanisms of plasticity in columnar-grained NT Cu films with a particular emphasis on the role of TB defects in strengthening and softening processes. We further extend this discussion to the mechanisms responsible for the excellent plastic flow stability of this material under severe rolling deformation. Last, we present the results of an innovative experimental approach using bundles of nanotwins induced by dynamic plastic deformation (DPD) (i.e., deformation with high strain rates) to dramatically strengthen coarse-grained Fe-based and Cu-based alloys.

Strain hardening in one-dimensional nanotwinned metals

Metallic NWs usually exhibit ultrahigh strength but low tensile ductility owing to their limited strain-hardening capability.[3–5] Single-crystalline NWs of fcc metals (**Figure 1**a) deform via dislocation-mediated plasticity[6–9] or via deformation twinning and lattice reorientation,[10–12] without exhibiting pronounced hardening. To promote strain hardening, one can engineer TBs into NWs to act as barriers to dislocation motion. Figure 1b–d shows schematics of different-ordered arrangements of TBs in NWs, including horizontal, inclined, and vertical (fivefold twinned) TBs.

Several groups have studied NWs with horizontal TBs (Figure 1b).[13–16] In periodically twinned NWs, strain-hardening behavior was only evidenced in low stacking-fault energy (SFE) metals such as NT Au and Ag.[17] In contrast, no strain hardening was observed in high SFE metals like NT Cu pillars.[13] This fundamental difference is attributable to the

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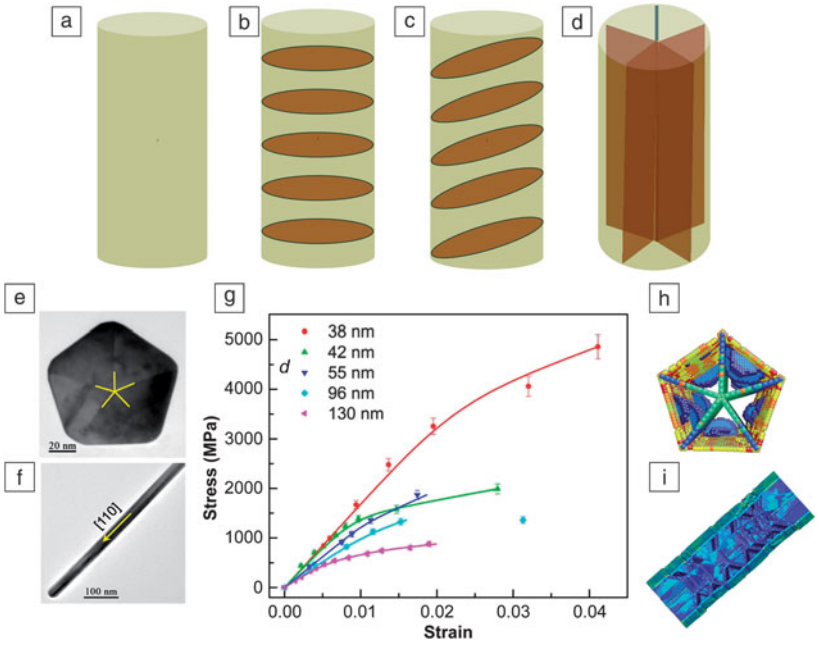

Figure 1. Strain hardening in nanotwinned nanowires (NWs).[22] (a–d) Schematics of NWs with different-ordered arrangements of twin boundaries (TBs) (colored in brown): single crystal without TBs, with horizontal, inclined, or vertical TBs. (e–f) Scanning electron microscope images of a fivefold twinned Ag NW. (g) Measured stress–strain data (symbols) for fivefold twinned Ag NWs of various diameters, *d*, with the power-law fitting curves (solid lines). (h–i) Molecular dynamics snapshots showing (h) surface nucleation of dislocations and (i) obstruction of surface-nucleated dislocations by vertical TBs in a fivefold twinned Ag NW.

relative change as a function of the unstable SFE between the stress required to nucleate new dislocations from free surfaces and that to overcome the TB resistance on the glide of partial dislocations.[17] Furthermore, past studies[18,19] have shown a strong inverse square root dependence of the elastic strain limit and tensile yield stress on twin thickness in ultrathin twinned NWs, resulting in surface dislocations always being emitted from the NW region with the largest twin size. Effective hardening due to these horizontal TBs requires uniformly small twin spacing across the entire length of the NWs, thus imposing a challenge to NW synthesis.

Inclined TBs (Figure 1c) in NWs are subjected to nonzero-resolved shear stresses during axial tension/compression of NWs and are prone to migrate, causing twin coarsening.[3] In contrast, vertical TBs (i.e., the fivefold twinned structure in Figure 1d) in axially loaded NWs are subjected to zero-resolved shear stresses and are more stable than the inclined TBs. Interestingly, NWs containing vertical TBs have shown promising results for strain hardening. Zhu et al.[20] reported pronounced strain-hardening behavior in Ag NWs with a well-defined fivefold vertical twin structure (Figure 1e–f). Filleter et al.[21] further studied this type of Ag NWs of various diameters and found that the Ag NWs with smaller diameters exhibited stronger strain hardening than those with larger diameters.

Recently, Narayanan et al.[22] reported an in-depth study of the work-hardening response and size effects in fivefold twinned Ag NWs by combining *in situ* tensile testing inside a scanning electron microscope and molecular dynamics (MD) simulations. Figure 1g shows that Ag NWs of smaller diameters harden more than those with larger diameters. MD simulations further showed that the hardening response and size effect were dominantly controlled by surface nucleation of dislocations as well as effective obstruction of dislocations by TBs (Figure 1h–i). They highlighted the important role of statistical variation of source strengths of surface-nucleated dislocations in macroscopic hardening of the fivefold twinned Ag NWs.[23]

Microscopic plasticity mechanisms in bulk nanotwinned metals

Compression of NT Cu pillars without GBs[13] revealed that TBs alone are not sufficient to mediate the plasticity of NT metals, and that a reasonable mix of TBs and GBs acting as a continuous source for dislocations is helpful to achieve high ductility. Significant experimental progress was made by controlling GB growth in {111}-textured NT Cu films fabricated using either physical vapor deposition[24] or direct electrodeposition.[25] These films exhibited long columnar grains grown in the same upward direction with TBs perfectly parallel to the film, see **Figure 2**a. In contrast to NT metals with randomly oriented grains,[1,26] columnar-grained microstructures present strong anisotropy in plastic behavior when the loading direction changes with respect to the TB orientation.[27–29] Ye at al.[27] have experimentally observed a twin-orientation-dependent hardness and strain-rate sensitivity in NT Cu, and argued that hard-deformation modes, such as slip-transfer mechanisms, were apparently more strain-rate sensitive than soft deformation modes (e.g., threading dislocations gliding between TBs).

You et al.[29] used experiments and atomistic simulations to study isolated dislocation processes under either pure tension, compression, or shear loadings and identified three distinct processes of plastic deformation in columnar-grained Cu with TBs (Figure 2). First, uniaxial loading perpendicular to TBs produces partial dislocations emitted from high-energy GBs that are blocked by TBs if their slip plane crosses the interface (hard slip mode, Figure 2b), contributing to strengthening.[30,31] Second, shearing parallel to the TB planes (or tension of inclined TBs) forms twinning partial dislocations propagating along TBs (soft slip mode, Figure 2c), leading to softening by detwinning.[32] However, MD simulations also predicted detwinning in NT Cu, but not in NT Pd, proving that SFE plays a critical role in this behavior.[33] Third, tension parallel to TBs (e.g., films deformed in pure tension) gives rise to threading dislocations confined inside the twin lamellae and propagating in a direction parallel to TBs (Figure 2d). If the twin spacing is

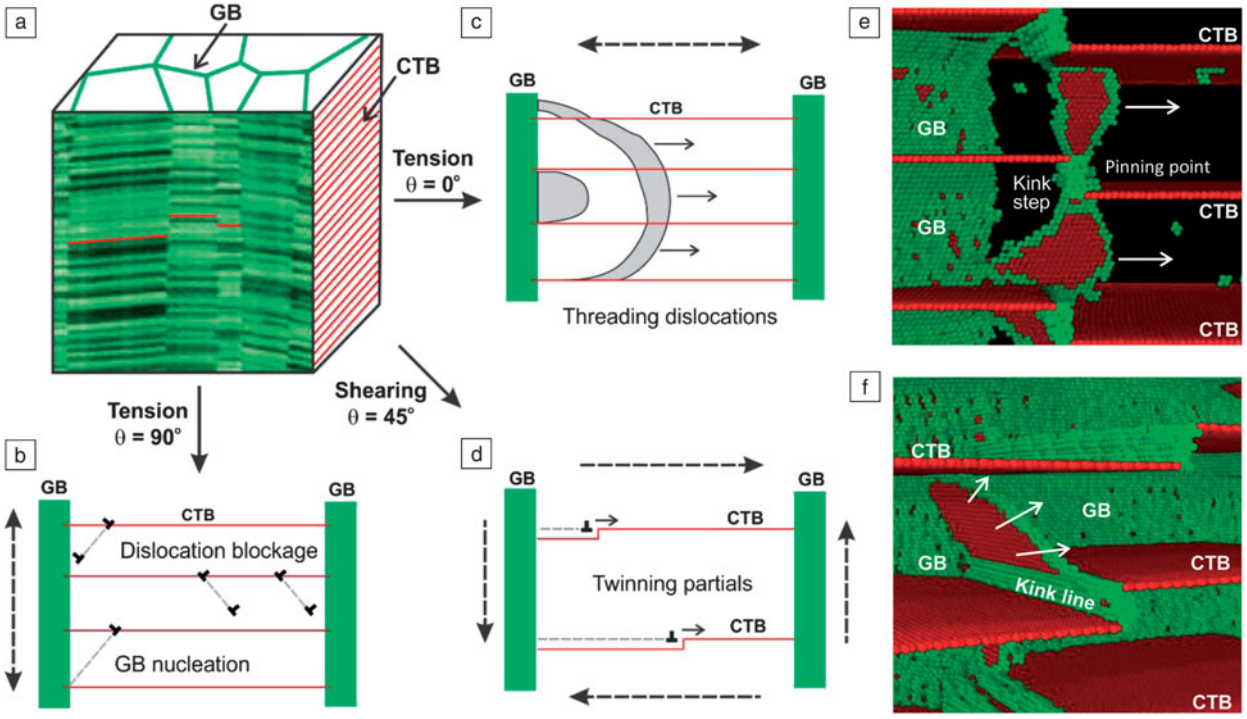

Figure 2. Plasticity of bulk columnar-grained nanotwinned metals with perfect or defective twin boundaries (TBs). (a) Schematic of grain boundary (GB) and TB morphology. Adapted with permission from Reference 27. © 2012 AIP Publishing, LLC. (b) Uniaxial loading perpendicular to the TB planes resulting in strengthening by blockage of GB-nucleated dislocations by coherent twin boundaries (CTBs), or (c) parallel to the TB planes leading to the propagation of threading dislocations cutting the CTBs with little resistance. (d) Pure shear loading ($\theta = 45°$) causing softening by detwinning. Atomistic simulations of (e) pinning of a threading partial dislocation by a kink-step defect leading to strengthening, and (f) dislocation emission at a kink-line–GB intersection leading to softening.[34] Note: White arrows in (e–f) indicate the direction of propagation of the partial dislocation in each image.

small, dislocation loops can extend over several twins to form necklace-like jogged dislocations that can move freely from one GB to the opposite GB.[34,35]

However, *in situ* synchrotron x-ray diffraction scattering tensile tests[34] have shown significant nonreversible peak broadening after failure of columnar-grained NT Cu, corresponding to a buildup of dislocations, unlike the behavior of nanocrystalline metals.[36] This contradicts the assumption that threading dislocations confined between two TBs glide freely without creating new dislocations. Furthermore, significant detwinning occurred at low temperatures in grains not favorably oriented to activate twinning partial dislocations due to low resolved shear stress. High-resolution transmission electron microscope (TEM) inverse-pole-figure-orientation mapping[34] revealed that TBs originally thought to be perfect contain many kink-like steps along the interfaces and are imperfect. These steps were found to be incoherent segments, 1–5 nm in height, substantially larger than the height of a single Shockley twinning partial previously reported on CTBs. MD simulations[34] and experiments[37] have found several new kink-dependent deformation mechanisms in NT Cu with defective TBs that could elucidate the observed abnormal phenomena: (1) Pinning of threading dislocations intersecting kink lines (Figure 2e); (2) dislocation nucleation from kink-line–GB intersection (Figure 2f); (3) detwinning from kink motion; and (4) twinning partial dislocations emitted from kink steps.

Microstructure evolution of nanotwins during severe rolling deformation

Another important aspect in the deformability of high-strength NT materials[38] is the plastic flow stability (i.e., changes in the morphology and texture during plastic deformation). In the case of columnar-grained NT Cu composed of 5-nm-thick twins, the plastic flow stability was evaluated by room-temperature rolling of NT foils stacked between stainless-steel sheets.[39] Despite the very fine structure and high strength, NT Cu films exhibited stable plastic flow in room-temperature rolling without localized shear bands that are known to limit deformability in high-strength nanocrystalline metals. Examination of the rolled microstructures revealed the following key characteristics of the deformation behavior of NT Cu after rolling to 50% reduction: (1) the average twin thickness did not change significantly; (2) the {111} fiber texture was retained; and (3) significant work hardening occurred.[39]

Figure 3 shows cross-sectional TEM images and corresponding measured twin thickness statistics for NT Cu foils.

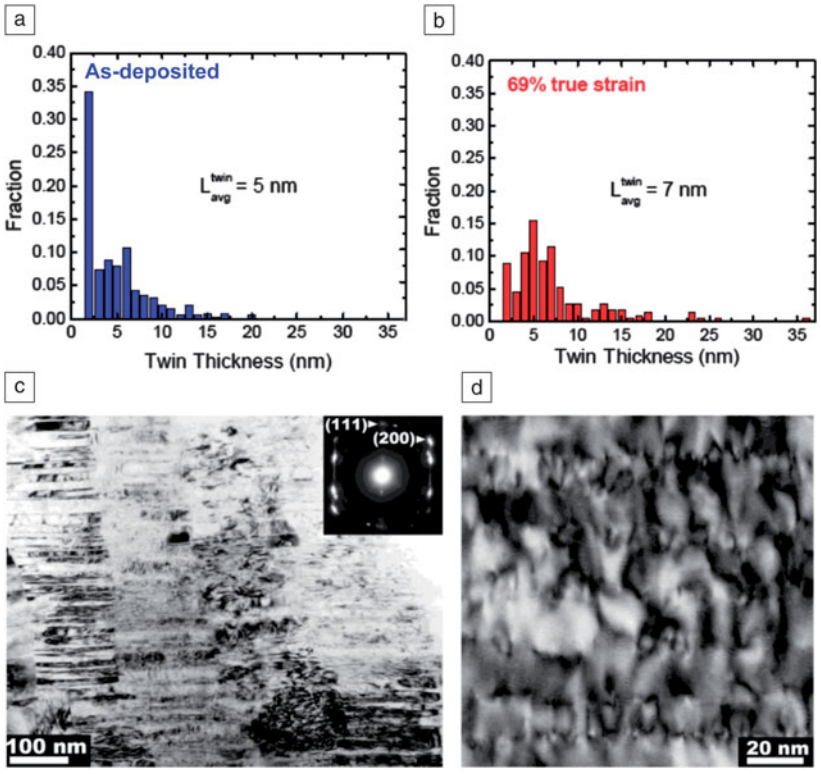

Figure 3. Plastic flow stability of rolled nanotwinned (NT) Cu foils. (a–b) Histograms showing transmission electron microscope (TEM)-measured statistics of twin-lamellae thickness before and after rolling, respectively. Note: the disappearance of the twins with thickness <2 nm after rolling. (c) TEM image and corresponding selected-area electron diffraction pattern showing the rolled NT microstructure and (d) higher magnification TEM image showing strain contrast along twin boundaries consistent with dislocation storage.[39]

The retention of average lamellae thickness after 50% reduction in sample thickness implies elimination of some twin interfaces during rolling. This is confirmed by the measured statistics of twin-lamellae thickness before and after rolling. Figure 3b shows that the fraction of twins with a thickness less than approximately 2 nm are missing in the histogram from the rolled sample. Indentation of NT Cu *in situ* in a TEM[40,41] revealed detwinning of very thin twins that was interpreted via atomistic modeling[40,42] and finite element analysis[43] as the migration of short incoherent TB segments connecting two parallel TBs.

For the twins that are retained after rolling, the measured {111} pole figures indicated preservation of the twin-plane orientation parallel to the rolling direction. The limited out-of-plane rotation of the twins can be interpreted in terms of symmetric slip, consistent with nanolayered composites where the interfaces act as effective barriers to slip transmission, thereby confining slip to the nanolayered channels. For compression normal to the TB, the resolved shear stress is constant for all active {111}⟨110⟩ slip systems, and plastic deformation can take place in a symmetrical mode on either side of TB, thereby suppressing net rotation of the (111) twin plane.[39,44]

Strengthening from deformation of nanotwins in bulk metals

High strain rates or low temperatures may effectively suppress dislocation slip and facilitate deformation twinning in fcc metals. Copious deformation twins with nanoscale thickness were induced by DPD (with rates typically $10^{1–3}$ s^{-1}) at low temperatures in a number of bulk metals and alloys with low or medium SFEs, including pure Cu,[45,46] Cu-Zn,[47] and Cu-Al alloys,[48] as well as austenitic steels.[49–51] The deformation twin thickness in Cu-Al alloys decreases (i.e., density of TBs increases) with a reduction in SFE induced by higher Al concentrations. For the same alloy, twin thickness becomes smaller when the sample is deformed at higher strain rates and lower temperatures. The twin thickness can be as small as 12 nm for Cu-Al alloys[48] and are on average 5 nm in Fe-25Mn steels[50] under the current DPD conditions.

Strength and ductility of the deformation-induced NT austenitic grains in a 316L stainless steel have been characterized by means of tensile tests and nanoindentation, respectively.[49,50,52] Ductility of deformation-induced NT austenitic grains is considerable, especially for those after recovery annealing. A uniform elongation of about 5% with considerable work hardening has been observed in a DPD 316L sample after recovery annealing in which 55% in volume are NT grains.[53] TEM observations[54] showed that at small tensile strains (<5%), the NT grains deform homogeneously in conjunction with the surrounding statically recrystallized (SRX) grains without generating notable strain localization near their interfaces, distinct from the conventional dual-phase structures.

NT austenitic steels with a single-phase duplex NT/SRX structure (NT grain outlined by yellow dotted line, **Figure 4**a), exhibit enhanced strength-ductility synergy and higher work-hardening rates than conventional austenitic and dual-phase steels (Figure 4b). Tensile ductility increases almost linearly with the volume fraction of SRX grains, thereby offering a promising approach to strengthening austenitic steels and other alloys.[55,57] A combination of 1.0 GPa tensile strength with elongation-to-failure of 27% is achieved in the NT 316L stainless steel.

Outlook for future research

Several questions remain for the underlying physics of strengthening and softening mechanisms in NT metals. TB imperfections play an important role in nanoscale plasticity of NT metals, yet their role has been largely ignored in past studies. Future computational and theoretical studies should focus on modeling the intrinsic contribution of TB defects in dislocation–CTB interactions.

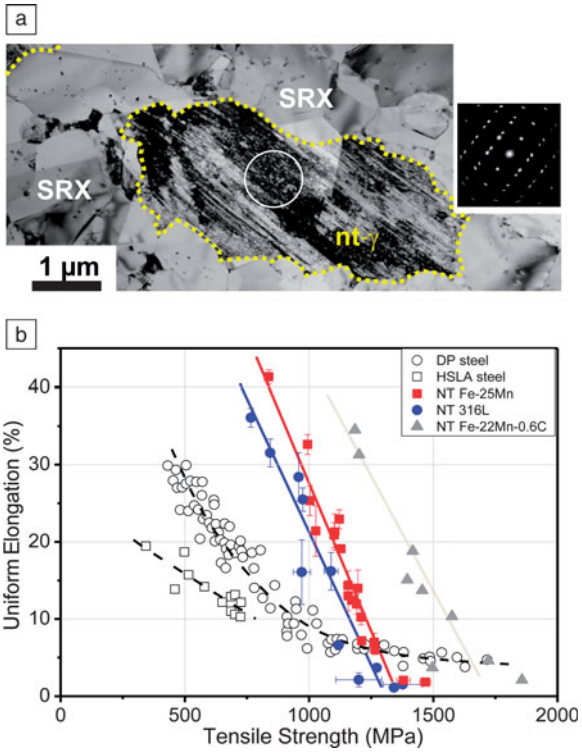

Figure 4. Strengthening by deformation nanotwins. (a) Bright-field transmission electron microscope (TEM) image of a nanotwinned (NT) austenitic grain (NT-γ; outlined by the yellow dotted line) embedded in the statically recrystallized grains in a dynamic plastic deformation 316L stainless-steel sample annealed at 750°C for 20 min.[49] Inset shows a selected-area diffraction pattern of the nanotwin grain (circled). (b) Variations of uniform elongation with tensile strength for three austenite steels strengthened by NT grains (Fe-25Mn,[50] 316L,[49] Fe-22Mn-0.6C),[55] in comparison with that of dual-phase (DP) steels and high-strength low-alloy (HSLA) steels. Reprinted with permission from Reference 56. © 1978 Springer.

Also, we note that the strain hardening in NT NWs other than fcc type has not been studied extensively. Recent *in situ* experiments inside a TEM have shown that twinning is the dominant deformation mechanism in body-centered-cubic tungsten NWs.[58] In the future, it would be interesting to explore the strain-hardening behavior and mechanism in non-fcc nanostructures that contain internal TBs, introduced by either growth or predeformation.

GBs are one primary source of dislocations in NT polycrystals. Dramatic changes in mechanical behavior of bulk NT metals are expected as a function of composition, structure, and morphology of the GB network. Past evidence has proved that microalloying can significantly enhance grain refinement in nanocrystalline Cu alloys,[59] where atom segregation to GBs not only prevents grain growth, but also plays an important role as a strengthening precursor. This strategy could hold great promise for the fabrication of nanocrystalline NT alloys with enhanced strength and ductility.

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