Consecutive crystallographic reorientations and superplasticity in body-centered cubic niobium nanowires

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Plasticity of metallic nanowires is often controlled by the activities of single deformation mode. It remains largely unclear whether multiple deformation modes can be activated in an individual metallic nanowire and how much plasticity they can contribute. In situ nanomechanical testing reveals a superior plastic deformation ability of body-centered cubic (BCC) niobium nanowires, in which a remarkable elongation of more than 269% is achieved before fracture. This superplastic deformation originates from a synergy of consecutively nucleated multiple reorientation processes that occur for more than five times via three distinct mechanisms, that is, stress-activated phase transformation, deformation twinning, and slip-induced crystal rotation. These three coupled mechanisms work concurrently, resulting in sequential reorientations and therefore superplastic deformation of Nb nanowires. Our findings reveal a superior mechanical property of BCC Nb nanowires through the close coordination of multiple deformation modes, which may have some implications in other metallic nanowire systems.

INTRODUCTION

Metallurgical nanowires have unique mechanical properties, such as ultrahigh strength (1, 2) and excellent deformation abilities (3–5), making them potentially suitable for applications in various micro-/nanoelectromechanical devices. In the past decade, the mechanical behavior of nanosized body-centered cubic (BCC) metals has attracted more attention because of their ultrahigh strength and excellent high-temperature performance (6). In bulk BCC metals, screw dislocations with low mobility usually dominated the deformation at room temperature and moderated strain rates due to the nonplanar dislocation core and high lattice resistance (7–9), resulting in a relatively poor deformability as compared with their face-centered cubic (FCC) counterparts (10–13). In the nanoscale regime, although ultrahigh stress favored a marked increase in the mobility of screw dislocations (9, 14), BCC nanopillars still showed poor tensile ductility owing to the deformation localization induced by dense dislocation activities (12, 14–16). Recently, both experimental and theoretical studies revealed that deformation twinning or phase transformation that rarely occurred in bulk BCC metals can be activated in a variety of BCC metallic nanowires (including molybdenum, tungsten, iron, and tantalum) under ultrahigh stress (17–21), which could act as alternative deformation modes to accommodate the deformation-induced structural reorientation (22). For example, deformation twinning can cause a reversible change of crystallographic orientation in loading cycles (20) and contribute to the pseudoelasticity, shape memory, and energy storage effects in BCC metallic nanowires (17, 18, 20), while phase transformation can stimulate a structure transition from the (001)-oriented BCC phase to a ⟨110⟩-oriented FCC phase at the crack tip of molybdenum thin film (23) or on the fracture surface of molybdenum nanopillars (19). However, in previous studies, BCC metallic nanowires only showed a solely dominant deformation mode (17, 18, 20–22). Given that the change of nanowire orientation by twinning/phase transformation may alter the deformation geometry and thereby stimulate other deformation modes in the subsequent deformation, it is natural to ask whether multiple deformation modes can be activated to coordinate with each other and contribute to a superior mechanical property in BCC metallic nanowires. Besides, as far as we know, current understandings of the twinning-/phase transformation–induced reorientations in BCC metals were mainly obtained from simulations due to the technological challenges, and it is unclear whether there are other operating reorientation mechanisms in the deformation of BCC metallic nanowires, especially in experiments.

Here, we adopted niobium (Nb) nanowires as the model system to investigate the deformation-induced reorientation and related plasticity in BCC metallic nanowires. By conducting nanomechanical testing of in situ fabricated Nb nanowires inside a transmission electron microscope (TEM), we uncovered a unique superplastic deformation of Nb nanowires, governed by a synergy of consecutively occurring multiple reorientations, where more than five individual reorientations took place before fracture, giving a remarkable uniform elongation of 269%. These unique, consecutively occurring multiple reorientations and resultant superplastic deformation have yet to be reported in studies on metallic nanocrystals. Atomistic analysis demonstrated that the multiple reorientations proceeded through three distinct mechanisms, including the BCC–FCC–BCC phase transformation, deformation twinning, and slip-induced crystal rotation. In the following, we will first discuss the different deformation modes observed in Nb nanowires and then show the superplastic deformation of Nb nanowires through their synergy.

RESULTS

Deformation-induced phase transformation

Deformation-induced phase transformation was an important mode to accommodate the structural reorientation in BCC metals under ultrahigh stress (19, 23, 24). Figure 1 and movie S1 show that, in Nb nanowires, the deformation-induced phase transformation and resultant crystallographic reorientation were realized by a two-step process. Initially, the Nb nanowire had a uniform diameter of ~15 nm and a perfect [100]-BCC lattice (Fig. 1A, inset). Upon tension, the accumulation of elastic shear strain led to a severe distortion of the [100]-BCC lattice,
characterized interplanar angle in FCC phase) first and then to 60° in the [111]-Nb, confirming the BCC-FCC-BCC phase transformation. At- omistic analysis indicated that the FCC phase existed in front of the phase boundary and acted as the precursor for the following phase transformation (Fig. 1C). With the gradual extension of the phase boundaries (the cyan dashed lines in Fig. 1, C and D), plastic deformation of the Nb nanowire proceeded continuously through the two-step BCC-FCC-BCC phase transformation, as demonstrated by the axial elongation and radial shrinkage of the nanowire in Fig. 1D. After fracture, the FCC-Nb domain can stably exist on the fracture surface, bounded by two BCC phases (fig. S1). This observation further proved the deformation-induced BCC-FCC-BCC phase transformation, which was a common deformation mechanism in the strained Nb nanowires (Fig. 1 and fig. S2).

The uncovered phase transformation from BCC to metastable FCC followed the Bain phase transformation that was used to model the FCC-to-BCC martensitic transformation (26). In experiments, the ultra-high shear stress should be the driving force to stimulate the phase transformation from BCC-Nb to metastable FCC-Nb with higher energy (27). Figure 1H schematically illustrates the atomistic model of the BCC-to-FCC transformation in the Nb nanowire, where the yellow and red spheres represent the BCC and FCC lattices, respectively. In this model, a condensed unit cell of the FCC structure can be drawn within four BCC cells such that a half of the BCC structure was a quarter of the FCC structure. With the accumulation of shear strain, severe lattice distortion occurred and converted the BCC unit cell to an FCC unit cell by elongating 20% in the z axis and contracting 12% along the x and y directions. The orientation relations between the original BCC phase and the metastable FCC phase were [100]//[011] and (011)\((\overline{1}11)\), compatible with the Nishiyama-Wassermann relationship (28). Subsequently, the transition from metastable FCC to [111]-BCC followed a reverse process by rotating the nanowire by 60° and elongating it by 29% (Fig. 1J), the red and blue unit cells represent the FCC and the new BCC lattice, respectively, and the orientation relations between these two phases were [011]//\(\overline{1}11\) and \((\overline{1}11)\)//(011), following the Kurdjumov-Sachs relation (29).
Deformation twinning

Theoretical studies showed that deformation twinning was a competing deformation mode in BCC metallic nanowires, such as W, Mo, Ta, and Fe (17, 18, 20, 21, 30), contributing to the superelasticity and reversible deformation behavior (18, 20). Here, direct experimental observations presented a twinning-mediated crystallographic reorientation in Nb nanowires. When an Nb nanowire was loaded along the [121] direction (Fig. 2, A and B), a deformation band was emitted from the surface and then penetrated the entire nanowire rapidly, resulting in a sudden yielding of the nanowire and tuning the nanowire axis to the [121] direction. Given the threefold symmetry of the BCC lattice in the [111] zone, the symmetrical relation between the deformation band and the matrix could not be identified; however, the structural analysis showed that the boundaries of this deformation band were closely located on the (112) twin planes (fig. S3, with a mismatch of less than 3°), suggesting that the observed deformation band should be a deformation twin. With further straining, this deformation band thickened laterally via the smooth migration of its boundaries (Fig. 2C), agreeing well with the layer-by-layer thickening behavior of deformation twinning, which confirmed that the observed deformation band was a mechanical twin (18, 20). This twinning-dominated deformation occurred frequently in Nb nanowires once their axes were reoriented to the [121] direction (see Fig. 2, D to G, and movie S2 for additional examples). In Fig. 2 (D to G), the nanowire diameter was 14.7 nm. Upon deformation, a twin was emitted from the grain boundary–surface intersection and then extended into the crystal on the (211) twin planes (Fig. 2E); subsequently, lateral growth of this twin occurred through the smooth migration of twin boundaries (Fig. 2, F and G), contributing to the plastic deformation.

Slip-induced crystal rotation

Dislocation activities in nanosized BCC metals often caused necking instability (10). Our current study, however, showed that consecutive crystallographic reorientations and thus good ductility can be achieved through dense dislocation activities in Nb nanowires. Figure 3 presents the dislocation-dominated deformation in an Nb nanowire under [110] tension. Since the [110] tension is an antitwinning direction in BCC metals, it would favor the dislocation slip on {110} planes because of the lower energy barrier (30), rather than the twinning on {112} planes (even if it has a higher Schmid factor; see table S1). Therefore, we observed dense dislocation activities in the [110]-oriented Nb nanowire under tension (Fig. 3). Upon straining, dislocations were emitted from free surfaces of Nb nanowires (evidenced by the inverse FFT image in the inset of Fig. 3B and fig. S4), which was consistent with previous studies where free surface usually acted as the preferential dislocation nucleation site in nanosized metals (19, 20, 31, 32). Associated with the surface nucleation of these dislocations was the formation of new surface steps (marked by the red arrows in Fig. 3, B and C). Subsequently, these dislocations propagated into the nanowire on (011) planes and then annihilated at the opposite surface rapidly, contributing to the plastic deformation of the Nb nanowire. As the slip events took place progressively on the adjacent (011) planes, a homogeneous elongation of

**Fig. 2.** Deformation twinning–mediated reorientation in Nb nanowires. (A to C) Deformation twinning in an Nb nanowire with a diameter of ~13.7 nm. Under [121] tension, a twin band nucleated from the free surface, penetrated the whole nanowire, and then thickened gradually via the migration of its twin boundaries, resulting in a crystallographic rotation of the nanowire matrix by 21°. (D to G) Deformation twinning induced reorientation in another Nb nanowire with the same loading geometry. The Nb nanowire had a diameter of ~14.7 nm before deformation.
the Nb nanowire occurred with uniform diameter shrinkage (Fig. 3, C to E). Accompanying the dislocation slips was the gradual rotation of the nanowire, which caused a partial loss of the original orientation, as shown by the region within the red dashed line in Fig. 3C. This region expanded along the nanowire axis with the dislocation slips and induced the reorientation of the whole nanowire (Fig. 3, C to E). We noticed that during the slip-induced crystal rotation, the zone axis and lattice structure of the Nb nanowire remained nearly unchanged (Fig. 3, D to F). Theoretically, if a single crystal was deformed freely under tensile loading, the uniform glides of dislocations on the well-defined parallel slip planes were likely to cause a shift of the loading axis (33). However, the grip heads could provide additional constraints to coordinate the rotation of these slip planes toward the tensile axis, thereby changing the tensile orientation and resulting in the elongation of the nanowire, as schematically shown in Fig. 3K (the yellow arrow and the blue arrow point out the slip direction of dislocations and the rotation direction of slip planes, respectively). This slip-assisted crystal reorientation further proceeded and dominated the subsequent deformation (Fig. 3, F to I), contributing to a superplastic elongation of the nanowire. Before fracture, the Nb nanowire thinned gradually at the necking region, and a four-atom-length atomic chain was formed through the structural reconstruction (Fig. 3J and fig. S2), similar to the ones observed in other ductile metals (34, 35).

**Consecutive reorientations and superplasticity in Nb nanowires**

Multiple reorientation processes can be activated in Nb nanowires through the coordination of the above-discussed deformation modes, giving rise to a superplastic deformation, in contrast to the plasticity dominated by the sole deformation mode in other metallic nanowires (17, 18, 20–22). For instance, the Nb nanowire in Fig. 4 and movie S3 exhibited remarkable deformability through the sequentially occurring multiple reorientations, in which more than five orientation transitions were recognized on the basis of the structural changes, contributing to a superelongation of more than 269% before fracture (Fig. 4G). A detailed analysis showed that the sequentially occurring reorientations in this Nb nanowire with a diameter of ~13.7 nm were controlled by the synergy of the above-discussed mechanisms that either changed the nanowire zone axis (Fig. 4, A and B) or rotated the nanowire lattice (Fig. 4, C to F). Figure 4 (A and B) and fig. S2 show that the first crystallographic reorientation arose immediately after limited elastic deformation through the BCC-FCC-BCC phase transformation, which changed the original nanowire with [100] lattice and [011] axis (Fig. 4A) into a nanowire with [111] lattice and [121] axis (Fig. 4B). A noticeable diameter shrinkage and uniform elongation of this nanowire accompanied the first reorientation. With further straining, the second reorientation occurred but proceeded in a different regime. In this process, a twin band nucleated from the free surface and then thickened gradually via the smooth migration of twin boundaries, resulting in a change of the nanowire orientation from [121] to [011], as well as a uniform elongation (Fig. 4C). Subsequently, dense dislocation activities governed the superplastic deformation of this Nb nanowire (Fig. 4, D to F), which caused the occurrence of several correlated reorientation events (more than three individual reorientations) without shear localization. The nanowire zone axis remained nearly unchanged (remained as [111]), but the crystal lattice rotated gradually around the zone axis during these reorientations [Fig. 4, D to F, the cyan dashed lines represent the position of the (011) plane after the third reorientation for reference].
with these reorientations was a uniform axial elongation of the nanowire (Fig. 4, D to F). Figure 4G shows the deformed morphology of the Nb nanowire before fracture. Obviously, the nanowire experienced a remarkable length increase (from 13.9 to 51.3 nm before necking) through the close synergy of different deformation modes, corresponding to a uniform elongation of ~269%, much higher than that of metallic nanowires reported in the literature (3, 19). Notably, statistical measurements showed that the consecutive multiple reorientations and superplastic deformation took place frequently in Nb nanowires with different diameters, as illustrated in Figs. S6 to S8. Previous studies showed that crystallographic reorientation can occur in metallic nanowires through phase transformation or deformation twinning (3, 4, 17, 18, 20, 32); however, to the best of our knowledge, these consecutively occurring multiple reorientation events and the resultant superelongation have never been reported before.

**DISCUSSION**

Experimental studies often showed the dislocation plasticity in BCC nanopillars (10). However, further reduction of crystal size enhanced the nucleation stress of dislocation in BCC nanocrystals markedly, such that other deformation modes (for example, phase transformation and deformation twinning) can be activated (17–22), as demonstrated by the current study. For the phase transformation, accumulation of high lattice strain favored by the small size should be considered as a necessary driving force to thermodynamically stimulate the lattice instabilities and, therefore, structural transformation from a low energy state to a high energy state in metallic nanowires. Indeed, phase transformation that rarely occurred in bulk metals was common in their nanosized counterparts under ultrahigh stress (19, 32). Ab initio calculation showed that BCC-Nb had two metastable phases of FCC and body-centered tetragonal structures under ultrahigh shear stress (36). Therefore, the BCC-FCC-BCC phase transformation should be an energy-favored process in Nb nanowires. Reduction of crystal size can also facilitate the deformation twinning in BCC metallic nanowires (20), and the twinning-dominated plasticity has been revealed in the simulations of different BCC metals (17); however, the experimental observation of the twinning in Nb remains largely lacking, probably because the initial nucleation of deformation twinning in BCC nanowires usually needs to overcome a high-energy barrier (37). For the slip-induced crystal rotation, the
small crystal size may facilitate the uniform nucleation of dislocations from multiple surface sites (evidenced by the slip-induced surface steps at different surface sites in Fig. 3, B, C, and G), and these uniformly nucleated dislocations were able to annihilate at the opposite surface rapidly without shear localization, delaying the premature necking. Although the dislocation slip tended to change the loading geometry, constraints from the grip heads required the nanowire axis to remain aligned to its original direction (that is, the loading direction). As a result, lattice rotation of Nb nanowires occurred synchronously with the dense dislocation activities. It needs to be pointed out that surface diffusion can contribute to large plasticity in FCC metallic nanocrystals (5, 38); however, no obvious surface diffusion was detected during the uniform deformation of BCC Nb nanowires [although the formation of an atomic chain may involve some diffusional surface reconstruction (fig. S5), its contribution to the superplasticity should be negligible]. A previous study showed that the surface diffusion was more significant in nanosized metals with a lower energy barrier for atomic surface diffusion (5); here, Nb had a strong bonding energy between atoms (39) and a very high activation energy of 1.12 eV for surface self-diffusion (40) (see the summarization in table S2), which would result in a negligible surface diffusion during the deformation of Nb nanowires.

Since phase transformation and twinning greatly released the lattice stress/strain and markedly changed the crystallographic orientation (20, 23), multiple twinning/phase transformation should be hardly activated in the same Nb nanowire owing to the released deformation stress. As a result, other deformation modes with lower activation energy (for example, dislocation slips) can be stimulated in the subsequent deformation. The close interplay of these deformation modes finally contributed to an improved deformability of the Nb nanowire. Statistical measurements in fig. S8A show that Nb nanowires with multiple deformation modes typically had higher uniform elongations. We also noted that few Nb bicrystal nanowires presented the sole deformation mode with poor ductility because of the existence of a weak grain boundary that resulted in crack and early fracture (fig. S8, B to D). It should be further pointed out that the operating deformation modes in Nb nanowires did not necessarily follow the Schmid law or obey the twinning-antitwining asymmetry (table S1). For example, phase transformation was activated under the [031] loading of Nb nanowires although the Schmid factor for dislocation slip was higher, while twinning occurred in the [121]-oriented Nb nanowire even though it had a smaller Schmid factor and was disfavored because of the antitwinning orientation. In BCC metals, the interplay between the nonplanar dislocation core and the applied stress field often causes the failure of the Schmid law (7), which may influence the twinning-antitwinning asymmetry in the nanoscale regime. Other factors, such as the size, cross section, and surface structure of metallic nanowire, also strongly affect their defect dynamics (1, 21, 31), which may have some contribution to the observed abnormal phenomenon in Nb nanowires. However, how these factors influence the competition of different deformation modes deserves systematical studies in the future.

In conclusion, in situ tensile testing uncovered a superplastic deformation behavior in BCC Nb nanowires. This superplastic deformation in Nb nanowires originated from a synergy of sequentially occurring multiple reorientations via three different mechanisms, including stress-activated phase transformation, deformation twinning, and slip-induced crystal rotation. Given that dislocation slip, deformation twinning, and phase transformation were common in BCC metallic nanowires (17–22), the revealed synergy of different deformation modes and the resultant large plasticity may have some implications in other metallic nanowire systems.

**MATERIALS AND METHODS**

Bulk Nb rods with a diameter of 0.25 mm and a purity of 99.9% were ordered from Alfa Aesar. In situ nanofabrication and nanomechanical testing of Nb nanowires were conducted inside an FEI Titan G80-300 TEM equipped with a Cs corrector. In a representative experiment, a nanofactory TEM–scanning tunneling microscope (TEM-STM) holder was used as the experimental stage, and two Nb rods with numerous nanoscale tips on the fracture surface were loaded onto the two ends of the TEM-STM holder, with one immovable at the sample side and the other at the mobile piezo-manipulator side. Then, in situ nanoscale welding was conducted to fabricate Nb nanowires inside the TEM. In a typical process, two nanotips with specific orientations on the fracture surface of the two Nb rods were connected together using a piezo-manipulator controller, and then a single-crystal or bicrystal Nb nanowire was fabricated inside the TEM by applying a square electric pulse (normally 0.7 to 1.4 V in amplitude and 20 ns in duration). Bicrystal nanowires were obtained as a result of the orientation differences between the two nanotips. In situ tensile loading was further applied on the as-fabricated nanowires at room temperature by moving the piezo-manipulator side backward with an estimated strain rate of $10^{-3}$ s$^{-1}$. Here, the diameters of as-fabricated Nb nanowires ranged from 10 to 29 nm.

**SUPPLEMENTARY MATERIALS**

Supplementary material for this article is available at http://advances.sciencemag.org/cgi/content/full/4/7/eaas8850/DC1

Fig. S1. Postfracture characterization of an Nb nanowire.

Fig. S2. Deformation-induced BCC-FCC-BCC phase transformation in an Nb nanowire.

Fig. S3. Identification of the twin plane in the Nb nanowire presented in Fig. 2.

Fig. S4. Surface nucleation of dislocation in an Nb nanowire.

Fig. S5. Formation of an atomic chain before the fracture of the Nb nanowire shown in Fig. 3.

Fig. S6. Multiple reorientations and superplastic deformation in an Nb nanowire.

Fig. S7. An additional example showing the multiple reorientations and superplastic deformation in an Nb nanowire.

Fig. S8. Statistical data showing the deformation modes and ductility of Nb nanowires with different diameters.

Table S1. Schmid factors for dislocation slip and deformation twinning under different loading directions of Nb nanowires.

Table S2. Activation energies of surface self-diffusion $E_D$ on the close-packed surface of FCC and BCC metals and their corresponding N-body potentials.

Movie S1. Deformation-induced phase transformation in an Nb nanowire with a diameter of ~15 nm.

Movie S2. Twinning-dominated deformation in an Nb nanowire with a diameter of ~14.7 nm.

Movie S3. Superplastic deformation of an Nb nanowire with a diameter of ~13.7 nm.

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