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## Strengthening high-stacking-fault-energy metals via parallelogram nanotwins

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### ABSTRACT

Twins readily form during growth or deformation in metals of low stacking-fault energy. Fabrication of high-density growth nanotwins in high stacking-fault energy materials is technically challenging. We synthesize nanotwinned Ni foils with controlled twin patterns – pulsed electrodeposition produces conventional parallel nanotwins while direct current electrodeposition generates a new pattern of nanotwins in the parallelogram form. The parallelogram nanotwins confine dislocation motion in the cage of twin boundaries and lead to a superior strengthening effect in Ni.

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Nanotwinned (NT) metals exhibit unique properties of ultra-high mechanical strength, enhanced ductility, superior thermal stability, and excellent electrical conductivity [1]. The attractive physical properties stem from the nature of  $\Sigma 3\{111\}$  coherent twin boundaries (CTBs) and the characteristic size at nanoscale [2,3]. CTBs with reduced spacing lead to the strengthening effect that is analogy to the classical Hall–Petch behavior. Meanwhile, nanoscale twins enhance the deformation ductility by retaining ample room to accommodate the dislocation accumulation. In addition, a small fraction of  $\Sigma 3\{112\}$  incoherent twin boundaries (ITBs) often coexist with CTBs in nanotwinned materials [4,5]. Propagation of ITBs contributes additional ductility of NT metals under a field of stress [5]. Despite the attractive mechanical properties, prior studies on NT metals focused on materials of low stacking-fault energy (SFE) or a low ratio of the unstable twin-fault energy ( $\gamma_{\text{utf}}$ ) to the unstable stacking-fault energy ( $\gamma_{\text{usf}}$ ) [6]. For high-SFE metals, dislocations are the predominant carriers to mediate the plastic deformation, although twins are occasionally activated at extreme conditions such as at crack tips, during high-pressure torsion or ball milling, and in deformed nanocrystalline grains [7–10]. Conventional processes to fabricate high-density growth nanotwins in low-SFE metals include electrodeposition, magnetron sputtering, electron-beam evaporation, plastic deformation, and phase transformation [11–14]. Synthesis

of high-density growth twins in metals of high SFE has been extremely challenging. A few efforts were made to fabricate growth nanotwins in Ni/Cu multilayers or by adding alloying elements in the plating bath to facilitate the nucleation of twin seeds [15,16]. Successful synthesis of high-density nanotwins in pure metals of high-SFE, however, is rare, which limits the utilization of nanotwins to strengthen a broad variety of engineering materials [11,17].

The primary scope of this study is to report a feasible route to synthesize bulk Ni (SFE 125 mJ/m<sup>2</sup>) with a high density of nanoscale twins by electrodeposition. Twin orientations can be controlled by varying the input current – pulsed electrodeposition fabricates Ni samples with nanotwins parallel to each other in grains, while direct current (DC) electrodeposition leads to a regular two-dimensional pattern that two sets of parallel twins constitute parallelograms. The average grain size and twin thickness can be well controlled by modifying a series of electrodeposition parameters. The parallelogram nanotwins, distinct from the conventional parallel twins in low-SFE materials, lead to ultrahigh mechanical strength and tensile ductility. The enhanced mechanical properties of parallelogram NT samples are attributed to the confinement effect for dislocation motion thus a larger capacity of dislocation storage and accumulation.

Table S1 summarizes the experimental conditions. Each parameter is varied separately in a given range to test the individual effect on the microstructure of as-deposited samples. The quantitative effects of the bath temperature, current density, Ni<sup>2+</sup> concentration,

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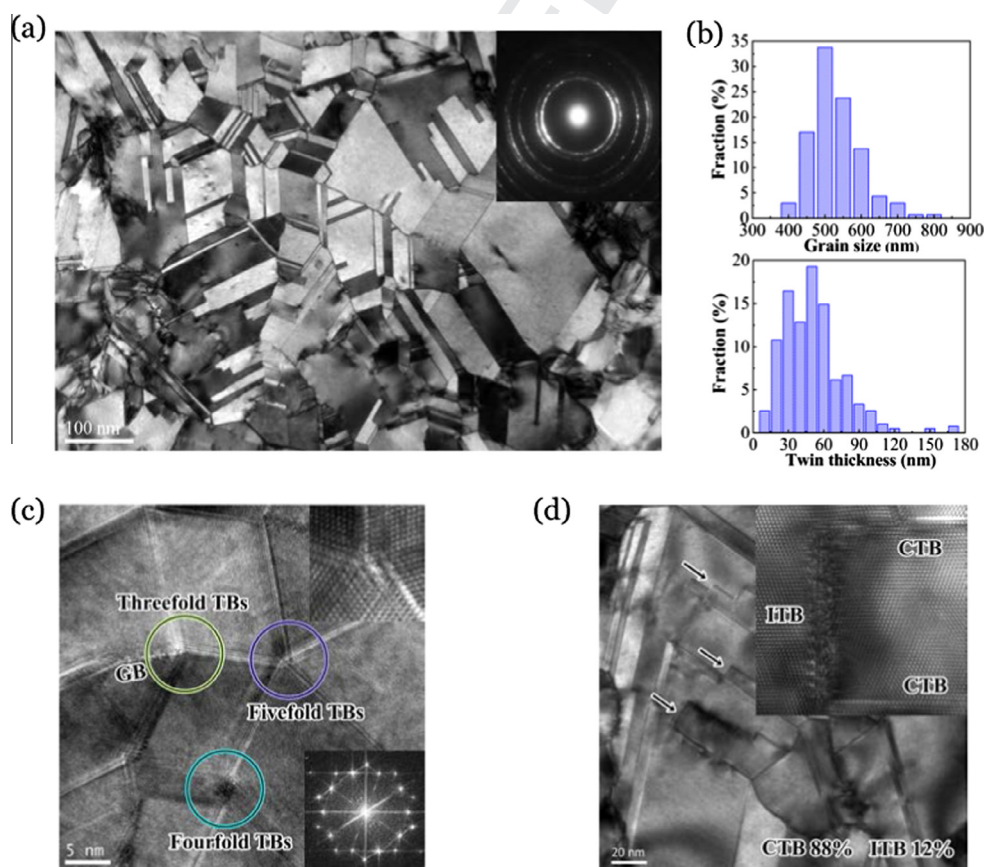
and ratio of current on-time and off-time ( $T_{on}/T_{off}$ ) on the average grain size, twin thickness, and twin length are illustrated in the Tables S2–S5. Overall, the increase of the above experimental controls results in a decrease of the average grain size and an increase of the twin density (Table S6). The explicit mechanism of nanotwinned Ni fabricated by the simple conditions of electrodeposition remains unclear at this writing due to the complex nature of electrochemical processes. Nevertheless, we aspire to disclose the experimental details that repeatedly produce the intriguing nanotwin patterns as summarized in the Tables S1–S6.

The as-deposited Ni foils have in-plane dimensions of 30 mm by 30 mm and a thickness of 30  $\mu\text{m}$  with a uniform columnar-grained microstructure and high purity (Figs. S1 and S2). Here we will focus on the characterization of the parallelogram nanotwins. Information on parallel twins is included in the Supplementary materials. Fig. 1a shows a transmission electron microscopy (TEM) image on the morphology of parallelogram twins embedded in sub-micron grains. The grain sizes are between 400 nm and 800 nm. Each grain is divided into parallelograms by a high density of growth twins. Measurements of the twin thickness along the  $[110]$  orientation show a wide distribution ranging from several nanometers to about 120 nm. High-resolution TEM (HRTEM) observations illustrate rich microstructural features. Multiple-fold twin boundaries exist at the junctions of parallelogram twins to fulfill the geometric compatibility (Fig. 2c) [18–20]. A mismatch angle presents at the junction of multiple-fold twin boundaries. In fivefold twins, for instance, the angle between two neighboring  $\{111\}$  planes is  $1.47^\circ$  smaller than that required to perfectly cover one fifth of a circle. Such a mismatch gap is accommodated by stretching and

broadening individual twin boundaries, which may act as a source of stress concentration and dislocation nucleation. Multiple-fold twin boundaries also connect with grain boundaries (GBs) and form a complex twin network structure. CTBs and ITBs also coexist (Fig. 2d). The densities of CTBs and ITBs in parallelogram nanotwins are  $4.5 \times 10^{14} \text{ m}^{-2}$  and  $5.8 \times 10^{13} \text{ m}^{-2}$ , respectively. The microstructural features of parallel nanotwins are shown in Fig. S3. It is noteworthy that the densities of CTBs and ITBs in parallelogram NT samples are markedly larger than their counterparts in parallel nanotwins  $-2.9 \times 10^{14} \text{ m}^{-2}$  for CTBs and  $1.9 \times 10^{13} \text{ m}^{-2}$  for ITBs.

Thermal stability of the growth nanotwins in as-deposited Ni foils is characterized by isothermal annealing. Detailed experimental procedure is shown in the Supplementary materials. *In situ* TEM observations of the microstructural evolution of parallelogram twins annealed at 100  $^\circ\text{C}$ , 200  $^\circ\text{C}$ , 300  $^\circ\text{C}$ , 400  $^\circ\text{C}$ , 500  $^\circ\text{C}$ , and 600  $^\circ\text{C}$  are shown in Fig. 2. Detwinning is observed in the samples annealed above 400  $^\circ\text{C}$ .

Tensile tests of NT foils are performed at a nominal strain rate of  $10^{-4} \text{ s}^{-1}$  at ambient temperature. Fig. 3 shows the high strength of parallelogram NT samples in comparison with that of parallel NT samples, a coarse-grained polycrystalline Ni sample, and a nanocrystalline Ni sample. The microstructures of nanocrystalline and coarse-grained Ni and grain size distributions are shown in Fig. S4. The parallelogram NT Ni samples also show considerable tensile ductility, with an elongation-to-failure strain of 6.5%, which is much higher than the nanocrystalline specimen. A slight strain hardening appears in the plot of engineering stress versus engineering strain during the major stage of plastic deformation.



**Fig. 1.** (a) The bright-field TEM image shows high-density parallelogram nanotwins in a Ni sample synthesized by DC electrodeposition. Electron diffraction pattern (inset) shows roughly equiaxed submicron grains with random orientations. (b) Statistical distributions of the grain size and the twin thickness. (c) Multiple-fold twin boundaries exist at the junctions of parallelogram twins to fulfill the geometric compatibility. The fivefold TBs are broadened to accommodate the mismatch angle as shown in the HRTEM image (inset). (d) CTBs and ITBs coexist. The percentage of CTBs and ITBs are 88% and 12% respectively.

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