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Local faceting in coarsening of nanolaminates with low angle boundaries in pure nickel

H. Xie [a], H.W. Zhang [a,b,*], J.G. Li [c], K. Lu [a]

[a] Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, China
[b] National Engineering Research Center for Equipment and Technology of Cold Strip Rolling, College of Mechanical Engineering, Yanshan University, Qinhuangdao 066004, China
[c] Superalloy Division, Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, China

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ABSTRACT

Nanolaminated structures with low angle boundaries were fabricated in a pure Ni single crystal with a Brass orientation by using dynamic plastic deformation supplemented by cold rolling. The coarsening mechanism of the nanolaminates at elevated temperatures was investigated by means of in situ TEM observations. Upon annealing, shortening and fragmentation of the nanolaminates are initiated by local faceting, forming flat facets that are inclined with each other by ~70° and aligned with one of the {111} planes. The underlying mechanism was analyzed in terms of the anisotropy of boundary energy.

Recent studies have demonstrated that nanolaminated structures with low angle boundaries exhibit extraordinary thermal stability as well as high hardness [1]. By using surface mechanical grinding treatment with high strain rates and high strain gradients, the nanolaminated structure with low angle boundaries can be refined down to 20 nm in pure Ni [2] and interstitial free steel [3], which is one order of magnitude smaller than the steady-state grain size induced by traditional severe plastic deformation. It exhibits an ultrahigh hardness of 6.4 GPa [1], and an onset temperature for structural coarsening of 506 °C that is 40 °C higher than the ultra-fine grained counterparts. The enhanced thermal stability originates from low boundary energies and the laminated structures. Naturally, understanding the coarsening mechanisms of the nanolaminated structures at elevated temperatures is of great significance for developing nanostructures with novel performance.

The mechanism responsible for thermally induced structural coarsening of laminated structure in highly deformed metals has been comprehensively investigated [4]. Apart from the removal of dislocations through mechanisms such as climb, glide and annihilation of dislocations [5,6], the evolution of lamellar boundaries to a more equiaxed configuration has been the key issue. One mechanism responsible for the initiation of boundary migration via locally pulling the lamellar boundaries toward the front interconnecting boundaries was proposed during the annealing of medium strain [7] and high strain lamellar structure [8]. Another initiation mechanism was suggested by the motion of Y-junctions that are composed of three high angle boundaries as observed for annealing of highly deformed pure Al [9]. For the nanolaminated structures with low angle boundaries, boundary migration mechanisms may differ from that with high angle boundaries. This is thus the purposes of the present investigation to address the structural coarsening process in nanolaminated structures with low angle boundaries, in particular the early stage of boundary migration.

Previous investigations have demonstrated that nanolaminated structure with a large fraction of low angle boundaries can be fabricated in a polycrystalline Ni by applying high strain rate compression, i.e. dynamic plastic deformation (DPD) [10]. The formation of nanolaminated structure is orientation dependent. The nanolaminate regions with low angle boundaries are usually alternatively distributed with those with both high and low angle boundaries [11]. Hence, by selecting a proper stable orientation in the initial sample, nanolaminates with only low angle boundaries are expected to be fabricated.

A single crystal Ni (99.945 wt.% purity, Table 1) with an orientation close to {110}⟨112⟩ (Brass orientation) was chosen for the DPD treatment. Brass orientation is a stable orientation that does not induce significant orientation spread during deformation [12]. The sample geometry was defined according to Fig. 1a (rolling direction (RD), transverse direction (TD) and normal direction (ND)). The Ni sample was made into prism of 10.0 × 8.0 × 6.8 mm (ND × RD × TD) and subjected to DPD in a channel die supplemented by cold rolling. The deviation from the ideal Brass orientation was determined by electron backscatter diffraction (EBSD) using an HKL technology channel 5

* Corresponding author at: Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, China.
E-mail address: hwzhang@ysu.edu.cn (H.W. Zhang).

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Table 1
Chemical compositions (wt.%) of the single crystal Ni used in the present study.

C	Si	Mn	P	S	Cr	Fe	Al	Co	Cu	Ti	Mg	Ni
<0.002	0.008	0.002	0.005	<0.001	<0.001	0.014	0.008	0.007	0.001	0.003	0.003	99.945

EBSD system, being ~3° (Fig. 1b). DPD was carried out at a strain rate of 10^2–10^3 s^{-1} to a von Mises effective strain (ε_{vm}) of 3.0, with loading direction parallel to ND. The strain was determined according to the thickness reduction, i.e., $\varepsilon_{vm} = (2/\sqrt{3}) \ln(t_o/t_f)$, where t_o and the t_f are the initial and final thickness. Since the single crystal Ni sample was too thin (750 μm) to be deformed further in the channel die DPD, cold rolling with roughly identical stress state as channel die DPD was supplemented to produce an extra effective strain of 1.0 (thickness reduction of 60%), leading to an accumulative effective strain of ~4.0.

The structural coarsening was investigated with thin foil and bulk samples, respectively. In-situ annealing of the thin foils was performed in a transmission electron microscope (TEM) by tilting the foils close to [110] which is parallel to the two active slip planes in Brass orientation. The typical deformation structure was firstly quantitatively characterized, and then the TEM foil was heated at a rate of ~1 °C/s up to 300 °C. Afterwards, temperature was increased at ~0.5 °C/s to the setting temperatures (400 °C, 450 °C and 500 °C), during which the microstructure evolution was recorded. For the bulk samples, thermal annealing was carried out in vacuum followed by furnace cooling. Structural characterization for the deformed and the annealed samples was performed on a JEM 2100 TEM by observing the RD-ND section. The boundary misorientation angle as well as the crystal orientation were determined by using the Kikuchi diffraction method, as described in [13].

The deformation microstructure of the Brass-oriented single crystal Ni is characterized by a typical laminated structure (Fig. 1c). The dominant features are the long and parallel lamellar boundaries that are roughly perpendicular to the ND and parallel to RD. Some lamellar boundaries are straight and sharp, others are curved and loose in which high-density dislocations exist. In the domains between lamellar boundaries, isolated dislocations with a fairly low density and interconnecting boundaries are observed.

Compared to the microstructure in the DPD polycrystalline Ni with medium-high strains [10,14], the present sample shows distinct features. Firstly, the spacing between lamellar boundaries is much smaller, spanning from 25–330 nm with an average of 79 nm (Fig. 1e). On the contrary, for a polycrystalline Ni (99.96 wt.% purity) deformed by DPD to a strain of 2.7, the average boundary spacing is 115 nm [10], whereas cold rolled Ni (99.99 wt.% purity) with a strain of 4.5 shows an average boundary spacing of 133 nm [14]. Secondly, low angle boundaries dominate the microstructure. As in the boundary sketch in Fig. 1d, most lamellar boundaries together with all the interconnecting boundaries are of low angle with misorientation angles below 15°. Misorientation measurements of 552 boundaries (Fig. 1f) show a unimodal distribution, with 96% low angle boundaries. This is distinct from that in the laminated structure formed in polycrystalline Ni where >40% [10,14] or 80% [8,15] boundaries are of high angle boundaries. Finally, the lamellar boundaries are deviated significantly from the trace of primary slip planes. According to the inserted traces of {111} plane, the deviation angle reaches ±30° and 90°, respectively. In contrast, the extended lamellar boundaries formed during monotonic plastic deformation at low to medium strain are generally aligned with the one of the traces of primary slip planes [16].

Structural coarsening of the nanolaminates with low angle boundaries during in-situ TEM annealing below 350 °C is very slightly, only loose dislocations are annihilated during annealing to some extent.

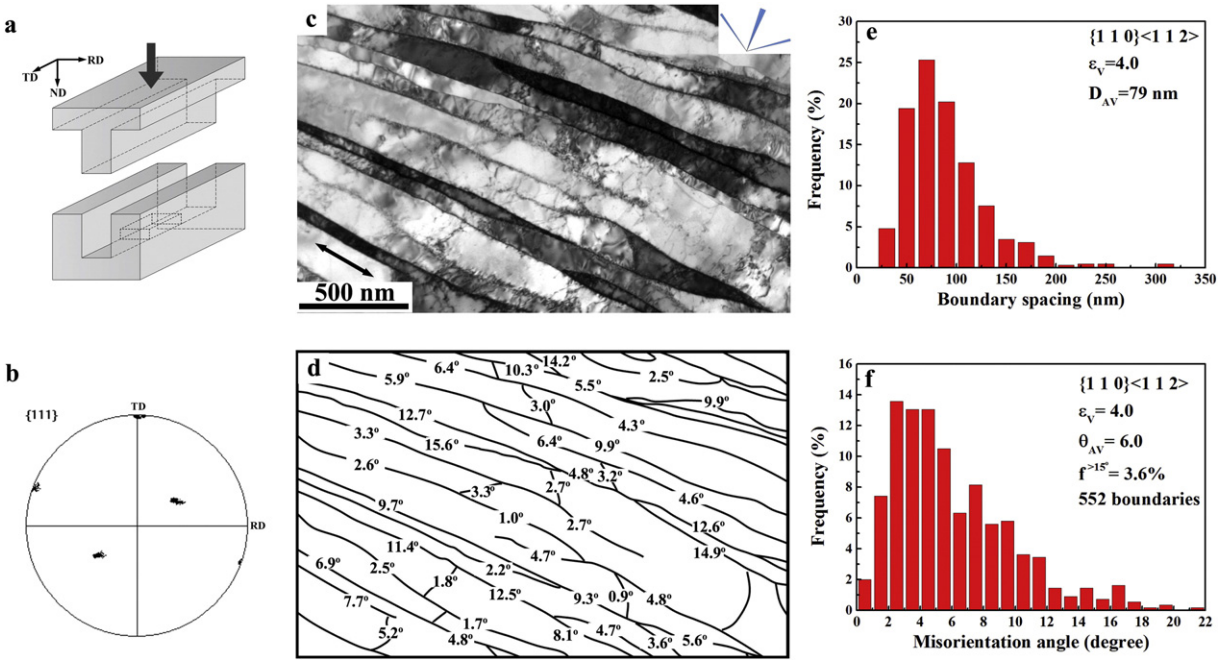

Fig. 1. (a) Schematic illustration of the sample coordination and the DPD process with a channel die, (b) the {111} pole figure of initial single crystal. A TEM image (c), the corresponding boundary sketch (d), histograms of boundary spacing (e) and boundary misorientation (f) of the nanolaminates with low angle boundaries in the deformed sample. The black arrow in (c) indicates the RD, while the traces of {111} planes are inserted as blue lines.