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Formation of nanolaminated structure in an interstitial-free steel

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Nanolaminated (NL) structure has been produced in an interstitial-free steel by means of surface mechanical grinding treatment. The NL structure is characterized by an average lamella thickness of ~20 nm and also exhibits a strong deformation texture. Various dislocation substructures and individual dislocations exist inside these lamellae from submicron size to a few nanometers. Due to this extraordinary grain refinement, the NL structure exhibits a record hardness of 5.3 ± 0.6 GPa.

Keywords: Nanolaminated structure; Interstitial-free steel; Surface mechanical grinding treatment; Grain refinement; Plastic deformation

Plastic deformation to high strains may cause significant microstructural refinement in metals, leading to substantial hardening. Over the past few decades ultrafine-grained (UFG) and nanograined (NG) materials have been fabricated via various severe plastic deformation (SPD) processes [1–5]. Although intense investigations aimed at achieving finer microstructures have been carried out in different materials by manipulating the deformation parameters (e.g. strain, strain rate and temperature) as well as deformation mode [6], it is still challenging to produce nanostructures (with length scales <100 nm) in pure or slightly alloyed metals. Our recent work has demonstrated that high-rate shear deformation with large strain gradients facilitates grain refinement [2]. We previously produced a novel nanolaminated (NL) structure with an average lamella thickness of 20 nm in a typical face-centered cubic (fcc) pure nickel by using surface mechanical grinding treatment (SMGT) [2]. The NL structure was found to be both ultrahard (with a Vickers hardness of 6.4 GPa) and stable (more stable than ultrafine-grained Ni). The objective of the present work is to explore possibility of generating the NL structures in an engineering material, namely interstitial-free (IF) steel, which is widely used in the automobile industry.

The average grain/cell size of interstitial-free steel processed by various SPD techniques, including equal-channel angular pressing (ECAP) [7], accumulative roll-bonding (ARB) [8], high-pressure torsion (HPT) [9], etc., is in the submicron regime (100–300 nm). Recently, Wu et al. [10] reported the formation of gradient nanostructures in IF steel by means of surface mechanical attrition treatment (SMAT), during which process the sample is deformed by the high-speed impact of vibrating steel balls on the surface. Typical nanostructures in the top surface of SMAT IF steel are roughly equiaxed grain/cells with an average size of ~100 nm [10]. Further refinement of the microstructures in IF steel below 100 nm has not yet been reported. In the present study, we utilized the SMGT process on commercially available IF steel to synthesize NL structures in a body-centered cubic (bcc) metal and to explore the structure–mechanical property relationship.

The commercial-purity IF steel (99.5 wt.% purity, see Table 1 for detailed chemical composition) used in this work was annealed at 1173 K for 10 h in vacuum followed by furnace cooling, leading to a fully recrystallized structure with an average grain size of ~27 μm and 92% high-angle grain boundaries. Detailed information about the SMGT setup can be found in Ref. [11]. During the SMGT processing, a cylindrical sample (with a diameter of 10 mm and length of 100 mm) rotates at a velocity of 300 rpm with respect to a hemispherical WC/Co tool tip with a radius of 4 mm. With a preset penetration depth of 30 μm into the sample, the tool tip slides at a velocity of 6 mm min^{-1} along the rod axis from one end to the other in one pass. In order to generate large plastic deformation underneath the tip, the process was repeated for 10 passes at room temperature. The microstructures of the samples were analyzed by both field emission gun scanning electron microscopy (FEG-SEM) and transmission electron microscopy (TEM). The as-treated samples were electrodeposited with a protective Ni layer and cut in cross-sections containing the normal and shear directions. A FEI Nova NanoSEM 430 microscope in electron channeling contrast (ECC) mode was used to obtain an overall morphology of the gradient microstructure. Detailed microstructure observations were carried out by high-resolution TEM in an FEI Tecnai G^2 F20 operated at 200 kV. Foils for TEM observation were prepared by using a Gatan 691 ion-milling system with a liquid nitrogen cooled stage.

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Table 1. Chemical composition (wt.%) of the IF steel used in this study.

C	O	N	P	S	Si	Ti	Mn	Cu	Ni	Al
0.037	0.0027	0.0062	0.04	0.003	0.04	<0.01	0.29	0.031	0.016	0.034

After SMGT processing, the gradient structural morphology observed from the cross-section (Fig. 1a) could be subdivided into three different regions: (i) a severely deformed layer in the top 200 μm that experiences severe shear deformation, producing typical lamellar structures parallel to the shear direction, as revealed by TEM observations; (ii) a transition layer beneath the top surface (200–500 μm in depth). The grains were collectively elongated toward the shear direction. Variations of contrast within the grains (see the white arrows) caused by the differences of crystallographic orientation between adjacent volumes were also seen in ECC mode [12]; (iii) a deep matrix retaining original structures that are not affected by the shear deformation at a depth of >500 μm. Grains in this region are of equiaxed morphology and uniform contrast in the grain interiors. Such a gradient structure is very typical for samples subjected to surface nanocrystallization techniques (SMAT [13] or SMGT [11]) [14,15], although the thicknesses of the deformation layer and nanostructured layer depend on the nature of the materials and the processing parameters.

The TEM observations shown in Figure 1b–d indicate that deformation-induced lamellar boundaries are formed, with a gradual decrease in lamella thickness from submicrometers to a few nanometers approaching the top surface. Lamellar boundaries are relatively straight and sharp, almost parallel to the shear direction. In the lamellae interior, there are many dislocation walls/tangles that further subdivide those lamellae into finer scales. Some of those dislocation walls/tangles are marked by white arrows. It should be noted that the NL structure with lamellar thickness ranging from a few nanometers to ∼70 nm was formed in the top 5 μm thick surface layer (see Figs. 1d and 2f). Due to the presence of a high density of crystallographic defects, lamellar boundaries are blurred. A sketch map in Figure 1e outlining those lamellar boundaries as well as some interconnecting boundaries enables the lamellar morphologies to be captured. The structural details within those nanolamellae were investigated by high-resolution TEM (see Fig. 2). It can be seen that many isolated full dislocations were identified within the nanolamellae as small as 10 nm in size (Fig. 2a). One-dimensional $\{110\}$ fringes (slip planes) of the boxed regions are shown in Figure 2b–d, revealing the presence of dislocations. We surveyed a few typical nanolamellae having $\{110\}$ fringes, from which we counted the number of dislocations. Discounting those dislocations immediately inside the lamellar boundaries and those invisible at a certain crystallographic orientation, the dislocation density within nanolamellae was estimated to be $\sim 2 \times 10^{15}\ \mathrm{m}^{-2}$. Such a high dislocation density may cause internal stress and crystal distortion inside lamellae, which could partly explain the frequently observed wrinkle-like contrast (see the white arrows) within the nanolamellae in Figure 1d. The presence of glide dislocations has also recently been reported by Hughes and Hansen [16] in a nanostructured copper sample with an extreme grain size of ∼5 nm, which is indicative of profuse dislocation activities at the finest scale (<10 nm) in high stacking fault energy metals.

In the topmost surface layer, the formation of nanolaminated structure, rather than equiaxed nanograins as reported in SMAT IF steel [10] or Fe [17], should be related to the main stress state (simple shear deformation). This assumption is underpinned by the presence of strong deformation textures. The selected-area electron diffraction (SAED) pattern from the NL structure (Fig. 2e) shows strong maximum intensity but discontinuity on the ring of $\{110\}$ reflections, indicating obvious deformation textures [18]. The 6-fold symmetric discrete pattern observed in Figure 2e is analogous to a pattern obtained within shear bands in nanostructured Fe [19], which is related to the activation of $\langle 111\rangle/\{110\}$ slip systems of bcc Fe. As we know, severe shear deformation may result in crystallite rotations, thus generating lattice-preferred orienta-

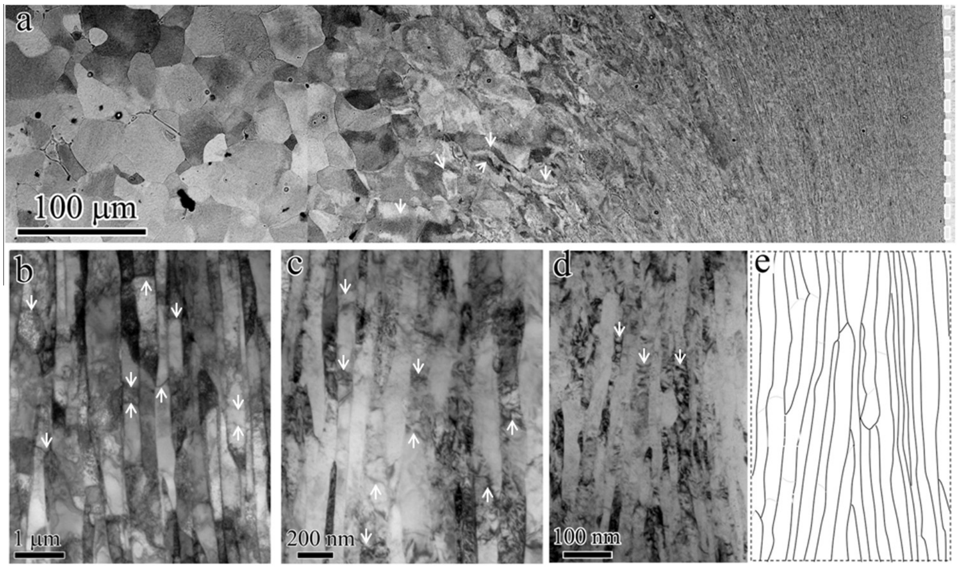

Figure 1. (a) A SEM cross-sectional observation of the SMGT IF steel sample; (b–d) TEM observations of the SMGT IF steel sample at different depths: (b) 120 μm, (c) 20 μm and (d) 5 μm. (e) A sketch map of the NL structure in (d). The yellow dashed line in (a) indicates the interface between the sample surface and the electrodeposited Ni. White arrows in (b–d) indicate dislocation substructures within the lamellae. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)