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Viewpoint Paper

Architectured surface layer with a gradient nanotwinned structure in a Fe–Mn austenitic steel

H.T. Wang, N.R. Tao and K. Lu*

Shenyang National Laboratory of Materials Science, Institute of Metal Research, Chinese Academy of Sciences, 72 Wenhua Road, Shenyang 110016, People's Republic of China

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Abstract—A surface layer with a gradient decrease in twin density has been produced in a Fe–Mn austenitic steel, which corresponds to a gradient drop in hardness from 5.3 GPa in the top surface to 2.2 GPa in the coarse grained core. The dependence of hardness on the twin thickness was determined, showing a weaker strengthening effect of twin boundaries than that of conventional grain boundaries in this alloy. Superior strength–ductility synergy was observed in tensile tests of the gradient nanotwinned layer.

Keywords: Architectured materials; Nanoscale twins; Surface mechanical grinding treatment; Gradient structure

1. Introduction

Materials with spatial gradients in composition and structure are of considerable interest as they exhibit enhanced mechanical, tribiological, biomechanical, and opto-electronic performance compared with their homogeneous counterparts [1–4]. Mechanistic analysis and experimental measurements have indicated that the damage and failure resistance of material surfaces can be significantly altered through such gradients [2]. Materials with gradient structures often exhibit better mechanical performance loaded under complex stresses. The global performance of bulk materials may be substantially altered when their surface layers are transformed into a gradient structure.

Recent studies indicated that a gradient in grain size distribution with depth, ranging from nanometers to micrometers, can be synthesized in the surface layer of bulk materials via surface plastic deformation approaches such as surface mechanical attrition treatment (SMAT) [5–8] and surface mechanical grinding treatment (SMGT) [9,10]. Gradient distributions of plastic strain and strain rate are generated in a treated surface layer of a certain depth, inducing different degrees of grain refinement at different depths and resulting in a gradient distribution of grain sizes. Such a gradient nanograined (GNG) surface layer has been realized in pure metals and alloys [7–12].

The global tensile properties are significantly enhanced by a thin GNG layer on a coarse grained (CG) pure Cu bar sample: the yield strength is doubled while the tensile ductility is unchanged compared with those of the CG Cu sample [10]. The fatigue properties of a 316L stainless steel sample was obviously elevated after a GNG structure was formed in its surface layer [13]. These enhanced mechanical responses originate from the unique gradient structure, which may exhibit different mechanical behaviors with higher resistance against stress concentration and cracking. The onset of permanent damage or cracking may be suppressed by the GNG structure, in which stress is redistributed and crack propagation across the gradient structure is retarded [14,15].

While GNG structures are drawing increasing attention due to their enhanced mechanical behavior, another type of gradient structure might be equally interesting, i.e. gradient nanotwinned (GNT) structure. Investigations over the past few years have revealed that nanoscale twin boundaries (TBs) can efficiently strengthen materials without compromising other useful properties, such as ductility, work hardening, electrical conductivity, and resistance to electromigration [16–19]. Submicrograined pure Cu containing nanoscale twins exhibit a tensile strength as high as 1 GPa and considerable ductility and work hardening capabilities [16,17]. Micro-sized austenitic steels strengthened using the nanotwinned

* Corresponding author. Tel.: +86 24 2390 6826; fax: +86 24 2399 8660; e-mail: lu@imr.ac.cn

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austenite grains show an enhanced strength–ductility synergy [20–22]. The origin of these enhancements is that TBs are effective in blocking dislocation motion, and at the same time they can act as slip planes to accommodate dislocations [23,24]. TBs have also been proven to be more resistant to cracking under cyclic loading [25,26].

The objective of the present work was to explore the processing of a novel surface layer with a GNT structure in a Fe–Mn austenitic steel with a low stacking fault energy and to reveal the mechanical properties of the GNT surface layer.

2. Sample preparation and surface mechanical grinding treatment (SMGT)

A Fe–Mn alloy with the chemical composition 28.3 wt.% Mn, 2.61 wt.% Si, 2.76 wt.% Al, 0.02 wt.% C and 0.007 wt.% S was chosen for the present study. The alloy was prepared by induction melting and homogenized at 1050 °C for 24 h, forged into a cylindrical sample of 20 mm in diameter at 1200 °C, followed by annealing at 1000 °C for 3 h. The as-received material was fully austenite with grain sizes of ~50 μm. The tensile yield strength was about 255 MPa and the tensile strength was about 600 MPa with a uniform elongation of 57%.

The cylindrical Fe–Mn steel specimens were processed by means of SMGT at room temperature, the detailed principles of which were described in Li et al. [9]. In short, a hemispherical WC/Co tip was pressed (to a preset indentation depth) onto the cylindrical sample which was rotating at high speed. The surface layer of the sample underneath the tip was deformed at high strain rates. The degree of plastic deformation and the thickness of the deformation layer were controlled by several processing parameters including the WC/Co tip diameter (d_t), moving velocity of the tip (v_t), rotation velocity of the sample (v_s), preset indentation depth (δ), number of processing passes, the nature of the lubricant, and the processing temperature. During the SMGT process no material removal occurred and the sample size was unchanged after the treatment. In the present study these parameters were $d_t = 8$ mm, $v_t = 10$ mm min^{-1}, $v_s =$ 100r.p.m., and $\delta = 80$ μm in the first pass and 40 μm in the second pass, respectively. SMGT was repeated for two passes for each sample at room temperature. The specimens were dog bone-shaped, suitable for tensile tests, with a gage diameter of 6 mm and gage length of 30 mm.

3. Structure characterization

The cross-sectional microstructures of the SMGT sample were characterized using a FEI Nova NanoSEM 430 scanning electron microscope in the electron channel contrast (ECC) mode, as shown in Figure 1a. During preparation of the cross-sectional specimens for scanning electron microscopy (SEM) characterization, the as-SMGT sample surface was protected by electrodepositing a pure nickel layer of about 1 mm thick. The cross-sectional specimens were polished and etched in an electrolyte of 92% alcohol and 8% perchloric acid. The features of plastic deformation can be traced to a depth of ~500 μm. No structural features could be resolved by SEM in the top layer which was about 10 μm thick (Fig. 1b). In the deeper layer, blocks filled with straight and parallel strips (deformation twins) could be seen and their density increased with increasing depth. At depths greater than 80 μm the original grain boundaries could be identified in the deformed structure. In the layer about 80–200 μm deep twinned blocks constitute almost all the observed area (Fig. 1c). The twin density decreased gradually with increasing depth beyond 200 μm (Fig. 1d). No deformation twins were observed deeper than 500 μm.

Transmission electron microscopy (TEM) was used to characterize detailed microstructures in a JEOL 2010 electron microscope operated at 200 kV. In the top layer, ~10 μm thick, nanosized grains with random orientations are found (Fig. 2a and e), with an average grain size of about 40 nm. Underneath this layer is a mixed microstructure of nanosized grains (with similar sizes to the topmost layer) and nanoscale twin bundles. With increasing depth the twin density and volume fraction of the nanotwin bundles increass gradually while the volume fraction of nanograins (without an obvious change in size) falls. As the depth exceeded about 200 μm dislocation structures in the form of tangles and cells mixed with nanotwin bundles were identified. The volume fraction of dislocation structures increased with increasing depth.

Quantities of different structures (nanograined, nanotwinned, and dislocation structure) were statistically measured using SEM images in ECC mode at different depths from the top surface, as shown in Figure 3a. The volume fraction of nanotwinned structure increased sharply in the top 50 μm thick layer, reaching about 90% at a depth of 80–200 μm, and then decreased gradually. Correspondingly, the proportion of nanograined structure fell steeply in the top 50 μm thick layer and tended to zero as the depth exceeded 80 μm. The volume fraction of regions containing dislocation structures increased at greater depth within a span of 150–500 μm. However, this does not necessarily mean that the dislocation density increased with greater depth. The total dislocation density decreased in deeper layers as the plastic strain generated by SMGT dropped at greater depths.

Statistical measurements from numerous TEM images showed that the deformation twins are extremely thin. At a depth of about 45 μm the average twin thickness (λ) was about 6.3 nm and the average matrix thickness (Δ) was about 16.1 nm. As the depth increased no obvious change was noticed in twin thickness but Δ increased gradually (see Fig. 3b). λ was about 5.4 nm and Δ was about 126 nm at a depth of about 440 μm. Obviously, a gradient variation in twin density had been generated in the surface layer of the SMGT Fe–Mn steel samples. For simplicity the entire ~500 μm thick deformation layer is hereafter referred to as a gradient nanotwinned (GNT) layer.

Formation of this GNT structure can be understood in terms of the strain and strain rate distribution during the SMGT process. The topmost surface layer had undergone plastic deformation with the largest strains and strain rates (which can be as high as 10^2 s^{-1}). Strain in the top layer after SMGT processing for two passes can be >2. Under these deformation conditions pronounced twinning dominated the plastic deformation.