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Gradient twinned 304 stainless steels for high strength and high ductility

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ABSTRACT

Gradient materials often have attractive mechanical properties that outperform uniform microstructure counterparts. It remains a difficult task to investigate and compare the performance of various gradient microstructures due to the difficulty of fabrication, the wide range of length scales involved, and their respective volume percentage variations. We have investigated four types of gradient microstructures in 304 stainless steels that utilize submicrotwins, nanotwins, nanocrystalline-, ultrafine- and coarse-grains as building blocks. Tensile tests reveal that the gradient microstructure consisting of submicrotwins and nanotwins has a persistent and stable work hardening rate and yields an impressive combination of high strength and high ductility, leading to a toughness that is nearly 50% higher than that of the coarse-grained counterpart. Ex- and in-situ transmission electron microscopy indicates that nanoscale and submicroscale twins help to suppress and limit martensitic phase transformation *via* the confinement of martensite within the twin lamellar. Twinning and detwinning remain active during tensile deformation and contribute to the work hardening behavior. We discuss the advantageous properties of using submicrotwins as the main load carrier and nanotwins as the strengthening layers over those coarse and nanocrystalline grains. Our work uncovers a new gradient design strategy to help metals and alloys achieve high strength and high ductility.

1. Introduction

Materials with gradient or hierarchical microstructure often have far super mechanical properties than those of monolithic nanostructured materials, and have attracted rising interest in materials research community [1–3]. Such gradient microstructure can be formed thorough different grain sizes [4,5], diverse twin spacing [6], or a combination of micro- and nano-sized grains and twins [6]. It has been conjectured and experimentally demonstrated that gradient hierarchical microstructure has the potential to evade the strength-ductility tradeoff dilemma in materials science [3,5,6]. Unlikely conventional materials, however, it is nontrivial to fabricate highly controlled gradient and more sophisticated hierarchical microstructures [3]. A systematic study of various gradient structures is obviously desirable but remains to be a daunting task due to a vast combination of different length scales and their volume fractions. As such, there exists little experimental effort to tackle this challenging problem.

Nanotwinned (NT) metals (defined as materials with average twin spacing $\lambda < 100$ nm) are strengthened primarily by coherent twin boundaries (CTBs) and also known for their combination of good strength and ductility, high electrical conductivity, and high thermal stability [7]. NT-metals can be considered inherently as materials with multiple/hierarchical length scales [8], as the grain space parallel and vertical to CTBs is vastly different. This trait of NT-metals offers ample room for strain hardening and thus high ductility. One major limitation of NT-metals is that high-density twins are predominantly formed in medium- to low-stacking-fault energy materials [9]. For this reason, bulk NT structures have only been realized in a handful of metals, limiting their broad applications. For practical utility purpose, a bulk part of studies on NT-metals have been focused on model pure metals such as copper and silver [7,10], which may bear less engineering applications than alloys such as steels. Questions remain as to how to take advantage of CTBs to achieve extraordinary mechanical properties in many real-life structural materials. In light of the emerging research on gradient microstructure and given the fact that CTBs exist in many metals and alloys (not necessarily with high density and in bulk form), an investigation of gradient hierarchical microstructures that contain CTBs is expected to generate novel

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mechanical properties and yield scientific insights for technological applications.

In this work, we systematically investigate the gradient hierarchical structures in 304 stainless steel (304 SS) (also known as A2 stainless steel), a material that is widely used by industry and home such as machinery parts and screws. 304 SS is a super corrosion-resistant material but relatively soft in coarse-grained (CG) form with a yield strength as low as ~270 MPa [11]. Enhancing its strength has been the focus of many past studies (see refs. [12–20]). Unfortunately, strength and toughness are generally mutually exclusive [21]. The key is to increase the strength of these materials without sacrificing their useful tensile ductility (i.e., high toughness). Novel microstructure design strategies are much needed in order to retain both high strength and high toughness of 304 SS or other steels at large. Although gradient microstructure has been shown to have good strength, good ductility, and better fatigue life [5,6,22], it is unclear what type of gradient structures offer better solutions to above problems. For the first time, we report that a gradient microstructure comprises of NTs ($\lambda < 100$ nm) and submicrotwins (STs, $100\text{ nm} < \lambda < 1000$ nm) exhibit impressively higher strength and toughness than other gradient microstructures that contain nanocrystalline (NC), ultrafine-grained (UFG), and CG structures. We investigate the mechanistic insights of these observations using *ex-* and *in-situ* transmission electron microscopy (TEM) and discuss their implications to other materials in general.

2. Materials and methods

2.1. Four types of hierarchical microstructures

As illustrated schematically in Fig. 1, we have investigated four types of 304 SS with multiple length scales, from ST (Type-I) microstructure to the one (Type-IV) that involves four different length scales, including NC (grain size $d < 100$ nm), UFG ($100\text{ nm} < d < 1000$ nm), ST and CG ($d > 1$ μm) structures. Note that akin to NT structure, ST itself also contains multiple length scales, including a broad distribution of λ. For description simplicity, however, here we refer to Type-I as the material with one featured microstructure (i.e., ST); Type-II has two featured length scales (i.e., ST and NT), and Type-III and Type-IV have three and four featured length scales, respectively. Our baseline material is the ST 304 SS (Type-I). While metals strengthened by NTs have been broadly investigated, the studies of ST-strengthened microstructure are less often but could bear broader implications to engineering applications. Our Type-II material mixes ST with NT and forms one of the simplest gradient microstructure. In Type-III 304 SS (i.e., ST+NT+NC), we investigate whether NC layers can further enhance the strength and toughness. Finally, a rather complex microstructure is studied; i.e., Type-IV (ST+NC+UFG+CG) with length scale spanning approximately three orders of magnitude from tens of microns to nanometers. In this design, ST and CG are expected to serve as plastic layers, whereas NC grains act as the main strengthening agent and UFG as a glue layer.

2.2. Materials fabrication

The chemical composition of 304 SS is 0.04 C, 0.49 Si, 1.65 Mn, 7.8 Ni, 16.8 Cr, 0.37 Mo and balanced Fe (all in wt%). The 304 SS sheets with dimensions of 70 mm × 50 mm × 1 mm and 100 mm × 90 mm × 1 mm were bombarded *via* surface mechanical attrition treatment (SMAT) to prepare type-III and IV steels, respectively. Different ball type, diameter, and vibrating frequency are used. The detailed processing parameters of SMAT has been reported in ref. [11]. Both sides of the 304 SS sheets were treated. The Type-I and II steels were obtained by removing 270 μm and 150 μm thickness from both surfaces of Type-III SMAT steels, respectively (see Fig. 1).

2.3. Tensile property measurements

Tensile samples of Type-III and IV steels were cut into dogbone shape with a gauge length of 30.0 mm and a width of 5.0 mm. For Type-I and Type-II 304 SS, the dogbone specimens were machined from Type-III steels with a gauge length of 6 mm, and the surfaces were polished to remove NC and/or NT layers. The dimensions of tensile samples for Type-I and II 304 SS are 6.0 mm × 1.0 mm × 0.4 mm and 6.0 mm × 1.0 mm × 0.7 mm, respectively. All tensile tests were performed using an MTS Alliance RT/50 testing system at a strain rate of $6.7 \times 10^{-4}\ \text{s}^{-1}$ and at room temperature. At least three tests were completed for each specimen type.

2.4. Microstructure characterization

In order to review the overall ST and NT structures, the cross-section of Type-II specimen was etched in solution (2 mL HF+3 mL HNO_3+95 mL H_2O) and examined *via* scanning electron microscopy (SEM, Hitachi S-4200). Conventional TEM was carried out using a JOEL-2010 microscope with an operating voltage of 200 kV. The λ was measured from TEM images when $\lambda < 1$ μm, and by SEM when λ in the micrometer range. As not all grains contain CTBs, the twin density (the percentage of grains

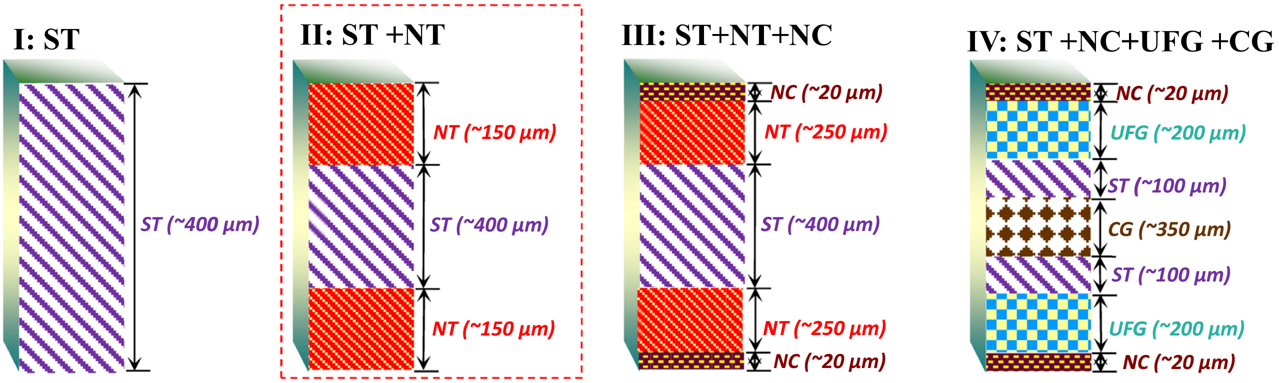

Fig. 1. (Color online) Schematic illustrations of four types of gradient 304 stainless steels (304 SS). Type-I 304 SS is composed of submicrometer twins (ST) with an average twin spacing ($100\text{ nm} < \lambda < 1000$ nm). Type-II 304 SS contains ST-layer core (~57 vol%), sandwiched by two outside nanotwinned (NT, $\lambda < 100$ nm) layers (~43 vol%). Type-III 304 SS (ST+NT+NC) is formed by adding two more outside nanocrystalline (NC) layers (grain size $d < 100$ nm) on Type-II material. Type-IV 304 SS includes four length scales, including NC, ultrafine-grains (UFG) ($100\text{ nm} < d < 1000$ nm), ST and coarse-grains (CG, $d > 1$ μm).