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Simultaneous improvement of tensile strength and ductility in micro-duplex structure consisting of austenite and ferrite

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ABSTRACT

A micro-duplex structure consisting of austenite and ferrite was produced by equal channel angular pressing and subsequent intercritical annealing. As compared to coarse-grained (CG) counterpart, the strength and ductility of micro-duplex samples are enhanced simultaneously due to smaller grain sizes in both phases and more uniformly distributed austenite in ferrite matrix. The average yield stress and uniform elongation are increased to 540 MPa and 0.3 as compared to 403MPa and 0.26 of its CG counterpart respectively. The Hall–Petch coefficients of austenite and ferrite grain boundaries were quantitatively measured as 224.9 and 428.9 MPa $\mu m^{1/2}$ respectively. In addition, a Hall–Petch type coefficient was used to describe the ability of phase boundary to obstruct dislocation motion, which was measured as 309.7 MPa $\mu m^{1/2}$. Furthermore, the surface-to-volume ratio of phase boundary in micro-duplex structure was estimated to be $1.17\times10^6\ m^{-1}$, which is increased by an order of magnitude as compared to $1.2\times10^5\ m^{-1}$ of its CG counterpart. Based on the strain gradient theory, a model was proposed to describe the effect of surface-to-volume ratio of phase boundary on strain hardening rate, which shows a good agreement with the experimental results.

1. Introduction

Extensive investigations over the past few decades have demonstrated that the nanostructured (NS) materials have poor tensile ductility although the strength is significantly increased as compared to their CG counterparts [1–4]. The low tensile ductility of NS materials is mainly attributed to their low strain hardening ability because the conventional dislocation mechanism is suppressed by the extremely small grains [1,5]. However, some researches during past decade also exhibit that the well-designed microstructures could achieve high strain hardening ability, including the introduction of a gradient or bimodal grain size distribution [6,7], the preexisting nano-scale growth twins [2], dispersion of nano-precipitates [8,9], transformation and twinning induced plasticity [10,11], and a mixture of two or multiple phases with varying size scales and properties [12].

Many natural and man-made materials consist of dual or multiple phases, which make them exhibit much better strength–ductility synergy than those single phase materials [13]. To elucidate the relationship between microscopic mechanical behaviors and bulk mechanical properties of dual and even multiple phase materials, more and more advanced *in-situ* experiments and computer simulations were conducted over the past two decades [14–18]. In terms of micro-mechanics, three conclusions could be addressed from those investigations. Firstly, the soft phase always tends to yield earlier than the hard one, leading to an inhomogeneous distribution of plastic strain across phase boundary even under uniaxial tensile test. Thus, secondly, the plastic strain gradient occurs between two dissimilar phases when plastic deformation happens. Thirdly, with different plastic strain accommodated by the hard and soft phases, the applied load born by hard phase is greater than that by the soft, resulting in inhomogeneous stress partitioning between two phases.

The above plastic deformation features are believed to be responsible for the optimized mechanical properties of dual and multiple phase materials. During plastic deformation, the hard phase is relatively elastically deformed and bears most of the applied load, while the soft one provides strain hardening ability and accommodates most of the plastic deformation. Thus high strength and good tensile ductility could be achieved simultaneously. Moreover, the plastic strain gradient across phase boundary requires the generation of geometrically necessary dislocations (GNDs), which would lead to an extra strength over the rule-of-mixture (ROM) prediction. In addition, with grain size decreasing to nanometer range, the lattice dislocations could glide on the phase boundaries and penetrate them into adjacent phases [19–22]. The phase boundary therefore play a similar role for NS dual phase materials as the twin boundary does for

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nano-twinned metals, thus reducing the strain localization and enhancing the interaction between dislocations during plastic deformation [2].

Since the interaction between two phases results from the strength difference in essence, it is reasonable to deduce that those features of inhomogeneous stress and strain distribution also occur in NS dual phase materials if strength difference still exists. This interaction has a potential to improve the dislocation storage ability due to the generation of GNDs, especially considering the increased surface-to-volume ratio of phase boundary. In the case of Cu/M (M=Nb, Ta, Fe, *etc.*) NS composites, the bulk strengths tested by experiments exceed the ROM predictions [23]. To describe this strengthening behavior qualitatively, an additional term of yield stress was introduced by considering the interplay between the phases [24,25].

Although promising in mechanical properties [26–30], the contributions of individual phase to the overall strength and strain hardening are difficult to analyze quantitatively, which are important in establishing the microscopic mechanical models and tailoring the macroscopic mechanical properties. These naturally raise two fundamental questions: How strong does the phase boundary impede the dislocation motion as compared to the grain boundaries of individual phases? And how much contribution does the phase boundary have to the overall strain hardening ability of duplex microstructure?

In the current study, the influences of phase boundary on strength and strain hardening rate are investigated. The micro-duplex samples were fabricated by equal channel angular pressing (ECAP) and subsequent intercritical annealing. Then the strengthening abilities of grain and phase boundaries were quantitatively analyzed. Moreover, the strain gradient theory was used to describe the effect of phase boundary on strain hardening behavior of micro-duplex structure.

2. Experimental procedures

The UNS S32304 duplex stainless steel (DSS) was used in this investigation, with chemical compositions (wt%) of 0.02C, 0.5Si, 1.2Mn, 23.5Cr, 4.0Ni, 0.4Mo, 0.13N, 0.024P, 0.002S, and balanced Fe.

The as-received billets of 10 mm in diameter were annealed at 1373 K for 2 h, followed by oil quenching in vacuum of about 10^{-4} Pa, in order to obtain the CG samples with nearly 50:50 phase balance between austenite and ferrite.

To fabricate the micro-duplex samples, the as-received billets of 10 mm in diameter were firstly solutionized at 1623 K for 2 h to form single ferrite microstructure (in vacuum of about 10^{-4} Pa and followed by oil quenching). ECAP technique was then used to refine the grain size of ferrite *via* a split die with two channels intersecting at inner angle of 90° and outer angle of 30° [31,32]. The ECAP was conducted at ambient temperature for 1 pass since further pressing is hugely difficult. At last, the ECAPed samples were intercritically annealed at 1173 K for different time to generate micro-duplex structure (in vacuum of about 10^{-4} Pa and followed by water quenching).

The dog-bone shaped tensile specimens were designed with rectangular cross-section of 2×1 mm^2 and gauge length of 8 mm and machined by electrical discharging along extrusion direction on the *Y* plane [32,33]. Tensile tests were conducted using an Instron 8871 test machine at room temperature with strain rate of 5×10^{-4} s^{-1}. At least three times of tensile testing were conducted for each microstructure.

An Olympus PMG3 optical microscope (OM) was used to examine the microstructures and measure the phase fractions. The chemical etchant used for OM observation consists of 30 g $K_3Fe(CN)_6$, 10 g KOH and 100 ml H_2O. Remaining the solution temperature at 353 K, the OM sample was immersed into it for 3 min.

X-ray diffraction (XRD) was taken to investigate the effect of tensile deformation on volume fraction of individual phases by using Rigaku D/max 2400 X-ray diffractometer with Cu K_α radiation, and a step size of 0.02°.

The micro-duplex structure before and after tensile tests was investigated by electron back-scattered diffraction (EBSD) using a field emission gun scanning electron microscope. Specimens for EBSD investigation were prepared on the *Y* plane by standard mechanical grinding and polishing procedures. In the final step, samples were electro-polished using a solution of 95% ethyl alcohol and 5% perchloric acid ($HClO_4$) at 253 K with voltage of 38 V. The EBSD scans were carried out at 15 kV in the center of the gauge section at a step size of 100 nm. The raw data were post-analyzed using TSL OIM software, and the average misorientation of a given point relative to its neighbors was calculated using an orientation gradient kernel method. In this study, the kernel average misorientation (KAM) was calculated up to the second neighbor shell with a maximum misorientation angle of 2°.

3. Results

3.1. Mechanical property

Uniaxial tensile tests were conducted to investigate the mechanical properties of different dual phase microstructures. The engineering stress–strain curves are shown in Fig. 1(a). The yield stress of micro-duplex samples annealed for 10 min at 1173 K is increased to 540 MPa, as compared to 403 MPa of CG DSS. More importantly, the uniform elongation is increased simultaneously, which is 0.3 as compared to 0.26 of CG DSS.

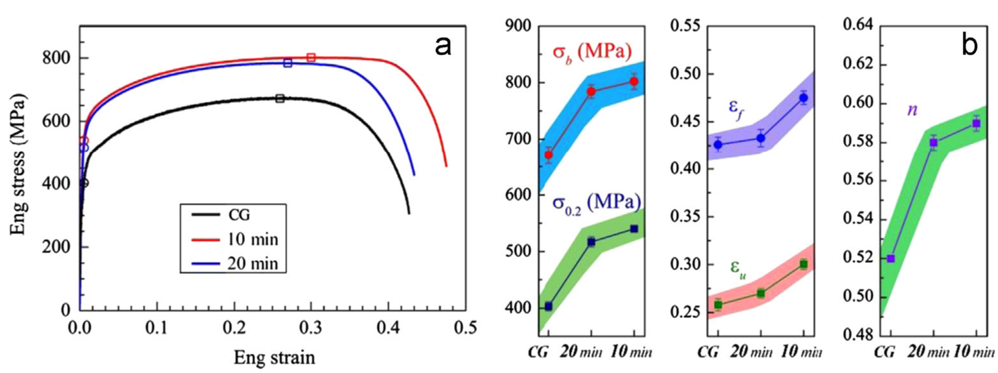

Fig. 1. (a) Tensile engineering stress *vs.* strain curves; (b) simultaneous increase of mechanical properties such as yield stress ($\sigma_{0.2}$), ultimate tensile strength (σ_b), uniform elongation (ε_u), elongation to failure (ε_f) and strain hardening exponent (n).