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Grain boundary segregation induced strengthening of an ultrafine-grained austenitic stainless steel

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ABSTRACT

The influence of grain boundary segregation on the strength of a nanostructured austenitic stainless steel was investigated. The steel was nanostructured by severe plastic deformation at two different temperatures to form ultrafine-grained states different by microstructure parameters. It is shown that despite the difference in grain size both nanostructured steels demonstrated the same level of strength. For the first time it is directly observed that severe plastic deformation at elevated temperature leads to formation of Mo–Cr–Si rich grain boundary segregations in the steel. Considering different contributions to the material strengthening, we demonstrate that grain boundary segregations can lead to significant enhancement of the yield stress.

1. Introduction

It is well known that reducing grain size leads to significant strengthening of metallic materials as well as particles, twins, a composition of solid solution, defects and so on. It was recently demonstrated an ultrafine-grained (UFG) Al–Mg alloy produced by severe plastic deformation [1] can exhibit an additional strengthening due to unusual grain boundary segregations induced by deformation [2,3] which suppress emission of dislocations from the grain boundaries. This paper demonstrates that similar effect can be observed for the 316 austenitic stainless steel. It is demonstrated that UFG 316 steel can exhibit extraordinary high strength fairly exceeding the Hall–Petch expectations. The understanding of the physical origin of such high strength in commercial alloys is very important from the viewpoint of potential advanced applications.

2. Experimental

Conventional stainless steel 316 (Fe–0.03C–17Cr–0.41Si–1.72Mn–0.01P–0.03S–12.9Ni–2.36Mo, wt%) was annealed at 1050 °C for 1 h and air quenched. Afterward it was nanostructured by high pressure torsion (HPT) which is known as one of the most effective deformation techniques for grain refinement [1]. HPT of specimens (disks 20 mm in diameter and 0.7 mm in thickness) was performed under a pressure of 6 GPa with 10 die-set rotations at room temperature (RT) and at 400 °C.

Microstructures were characterized by Scanning Transmission Electron Microscopy (STEM) using an ARM200F JEOL microscope operated at 200 kV. The grain size, d, was calculated from an area located at 10 mm from the disc center. Atom probe tomography (APT) analyses were performed with a Cameca FLEXTAP. Laser pulses with a wavelength of 313 nm and an energy per pulse adjusted to get an equivalent pulse fraction of 20% of the standing voltage were used. X-ray diffraction (XRD) measurements were performed with Rigaku Ultima IV diffractometer using CuK$_\alpha$ radiation. The obtained XRD patterns were treated by Rietveld refinement to calculate values of lattice parameter a, coherent domain size d_{XRD} and elastic microdistortion level $\langle\varepsilon^2\rangle^{1/2}$ using the MAUD software [4]. Dislocation density was estimated via (1) [5]:

$$\rho_{XRD} = \frac{2\sqrt{3}\langle\varepsilon^2\rangle^{1/2}}{bd_{XRD}}, \quad (1)$$

where b is the Burgers vector.

Tensile tests were performed at room temperature with the strain rate of $5\times10^{-4}\,s^{-1}$ with the help of a tensile machine for

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specimens with a gauge of 5.0 × 1.0 × 0.8 mm. Microhardness was measured in terms of H_V using Micromet-5101 device.

3. Results and discussion

The initial annealed and air quenched 316 steel exhibited typical coarse-grained structure with a mean grain size of 22 μm. STEM observations showed that HPT RT steel had heavily distorted microstructure with the mean grain size of about 40 nm (Fig. 1a). Selected area electron diffraction patterns (Fig. 1a) showed spots forming uniformly distributed Debye–Scherrer rings typical for ultrafine structures dominant with high angle grain boundaries [1]. The analysis of the rings proved that the given state is characterized mainly by γ-Fe phase (FCC). Few spots corresponding to martensite were also detected, suggesting the presence of a small fraction of this phase. XRD measurements revealed no resolvable martensite peaks for the given UFG state, indicating that the martensite fraction is below XRD sensitivity (less than 5%).

HPT 400 °C steel had less distorted equiaxed UFG microstructure with a larger grain size (about 90 nm— Fig. 1b).

Few grains with twins were observed (see Fig. 1b) with a typical spacing of 5–15 nm. The area fraction of twinned grains was estimated as (f=0.07). The analysis of selected area electron diffraction patterns and phase XRD analysis testified the microstructure being formed by plain austenite.

XRD-measured coherent domain size values for both UFG steels were in a good agreement with a tendency captured by TEM (Table 1). However, absolute values of the TEM grain size fairly exceed XRD coherent domain ones. This is a typical feature of UFG materials, especially produced by SPD [1] . This feature is explained by the fact that the coherent domain size determines a size of non-distorted areas in crystallites matching the Bragg's criterion for X-ray scattering. In UFG materials these areas could be considerably less than the grain size due to high internal stresses and SPD-induced defects. For the further analysis we used the TEM grain size and relied to corresponding references for comparison.

The steel in both UFG states demonstrated high hardness as well as high yield stress values (Table 1). The results of strength measurements were interpreted in terms of the Hall–Petch relation. Fig. 2 demonstrates the generalized Hall–Petch plot drawn for the reference data on 316 austenite stainless steel produced by rolling [6], liquid dynamic compaction [7], HPT at RT [8,9]. The data obtained in the present work were plotted against this

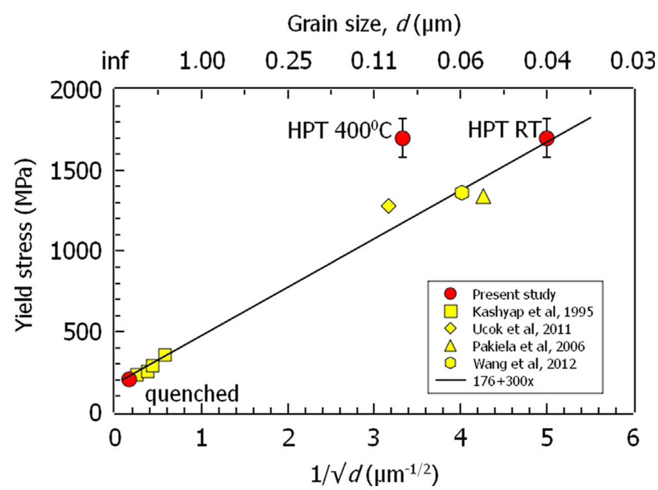

Fig. 2. A Hall–Petch plot comparing the data of the present work with the literature data [6-9].

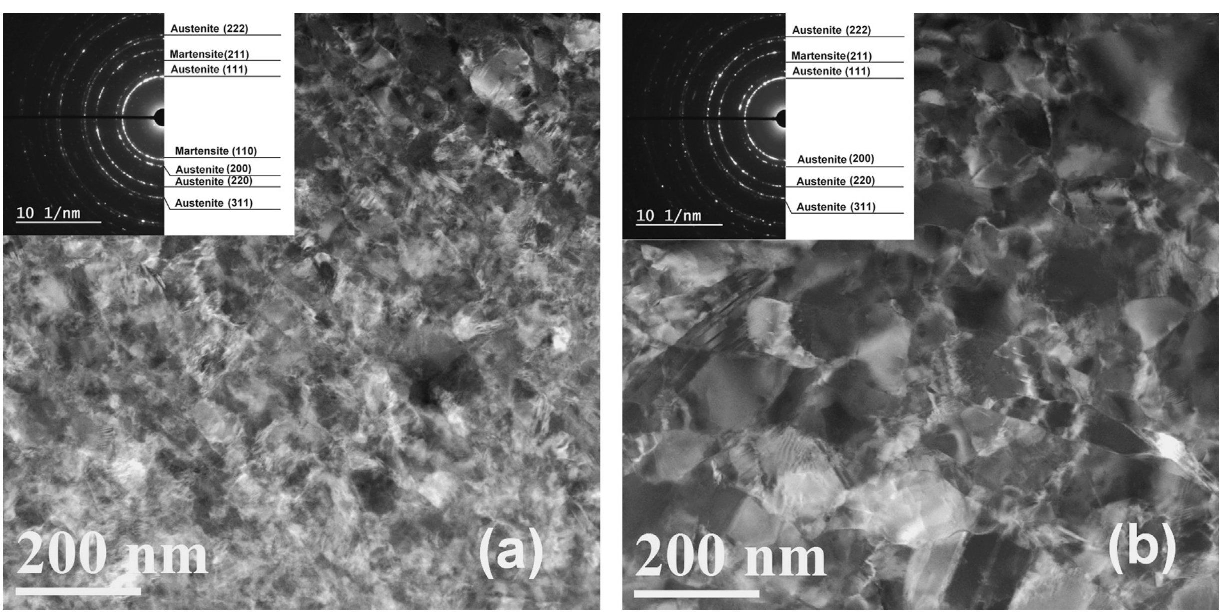

Fig. 1. STEM dark field images and selected area electron diffraction patterns for HPT RT 316 steel (a) and HPT 400 °C 316 steel (b).

Table 1
Microstructure parameters and hardening contributions for HPT RT and 400 °C 316 steel. Strength data are given in MPa.

State	d (nm)	d_{XRD} (nm)	ρ_{XRD} (m^{-2})	σ_0	σ_ρ	σ_{tw}	σ_{gb}	$\sigma_{0.2}^{calc}$	$\sigma_{0.2}^{exp}$	$\sigma_{0.2}^{HP}$	Hv
HPT RT	40	19	1.0×10^{15}	176	660	–	864	1700	1700	1670	5850
HPT 400 °C	90	55	0.6×10^{15}	176	510	121	536	1345	1720	1170	5750