

## Mechanical Properties and Microstructure of a Duplex Nanostructure

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**Abstract.** The nanostructure was obtained in a duplex stainless steel (DSS) by means of equal channel angular pressing. The mechanical properties were characterized by uniaxial tensile tests, while the microstructure was investigated by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). It was shown that the yield strength in a deformed nanostructure increased significantly, from 402 MPa to 1461 MPa as compared to its coarse-grained counterpart. In contrast, the uniform elongation decreased significant to only 2% together with elongation to failure of 9.8%, much lower than those of 25.4% and 42.6%. After annealing at 700°C for 10 minute, however, uniform elongation increases to 5.3% with the yield strength of 1200 MPa. TEM observation exhibited that deformation twins prevail in the austenite phase whereas the dislocations of high density present in ferrite. The plastic behavior in both phases was analyzed based on the deformation twinning and the presence of dislocation. Finally, the effect of the microstructure on mechanical properties was discussed.

### Introduction

Nanostructured materials usually possess high strength due to their small grain size, but the limited ductility attributed to low strain hardening ability is the heel of Achilles for their structured application. Therefore, intensive researches with different micro-structural designs have been sparked to improve the ductility of nanostructured materials during past decades <sup>1-4</sup>. One effective approach to optimize the strength and ductility is mixed phase structure<sup>5</sup>. It's expected that the harder phase sustain large tensile stress without fracture, whereas the softer phase accommodate plastic deformation without failure.

Equal channel angular pressing (ECAP) is one attractive severe plastic deformation techniques to produce ultrafine grained (UFG) materials because it can economically produce bulk UFG materials that are high dense and contamination free. In the last decades, many metals and alloys, including Al, Cu, Ni and its alloys have been successfully processed by the ECAP technique at room temperature. However, only few literatures reported the mechanical properties of dual phase structure produced by ECAP technique <sup>6-8</sup>.

Recent research demonstrates that nanometals are ductile intrinsically, and it can be deformed enormously plastically during cold rolling or tensile test when they are formed as grain gradient materials <sup>9</sup>. As for DSS which consist of two ductile phases, the geometrically necessary dislocations near phase boundary induced by deformation compatibility would play critical role on the tensile ductility.

The major objective of this study is to investigate the mechanical properties of a duplex nanostructure produced by ECAP technique. The microstructure evolution and fracture mechanism were discussed in detail.

## Experimental methods

The DSS used in this investigation is a commercially billets with a diameter of 10 mm. A heat treatment (1100 °C for 2 hours, followed by oil quenching) was performed so as to obtain a dual phase microstructure.

The ECAP experiments were conducted by using a split die with two channels intersecting at inner angle of 90° and outer angle of 30°, which yields an effective strain about 1 by each single pass. All samples was pressed at room temperature by route B<sub>C</sub> in which the work piece was rotated 90° along its longitudinal axis after the first pass.

The as-pressed billets were then cut into dog-bone shape tensile specimen with a rectangular cross-section (2 mm×1 mm) and a gauge length 8 mm by electrical discharging. Tensile tests were conducted on an Instron 8871 test machine with an extensometer which has accuracy more than 0.5%, and the moving speed of crossbeam was controlled at 0.24 mm per minutes.

Samples were prepared for optic microscopy by grinding and then polishing. A special electrolytic etching technique with 40 pct vol. HNO<sub>3</sub> aqueous solution (1.1 V, 40 s) followed by Murakami's solution (30 g K<sub>3</sub>Fe(CN)<sub>6</sub>, 10g KOH and 100 ml H<sub>2</sub>O). TEM thin foils were prepared by jet polishing with a solution of 95% ethyl alcohol and 5% perchloric acid (HClO<sub>4</sub>) at -20°C, applying a voltage of 20 V.

The fracture of tensile specimens was investigated by scanning electron microscope (HITACHI S-570), and the microstructure was characterized by transmission electron microscope (JEM 200CX).

## Experimental results

**Mechanical properties.** The engineering tensile stress-strain curves are plotted in Fig. 1, where the annealing temperature is 700°C. Table 1 lists the tensile properties in detail. The results show that yield strength of four passed (P) sample is significantly increased from 403 MPa to 1461 MPa as compared to its coarse-grained counterparts. In contrast, the uniform elongation is decreased to only 2% together with elongation to failure of 9.8%, much lower than those of 25.4% and 42.6%.

The tensile ductility can be enhanced by annealing treatment due to annihilation of dislocations. After annealed at 700°C for 10 minutes, the uniform elongation increased to 5.3% with the yield strength of 1200 MPa. The yield-to-ultimate tensile strength ratio is an important parameter to reflect the plastic deformation ability. The lower the ratio value is, the stronger the strain hardening ability will be. The results exhibit that it's increased to 0.82 after four passes and decreased to 0.76 after annealing.

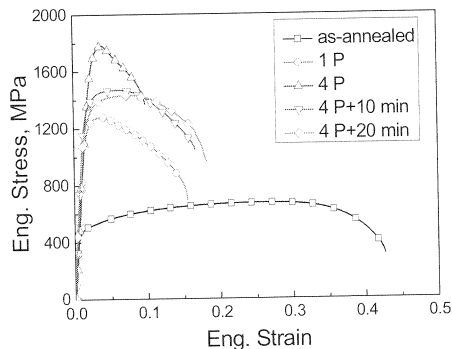


Fig. 1. Tensile engineering stress-strain curves of DSS

Table 1. Detailed summary of tensile properties of DSS. Yield stress  $\sigma_{0.2}$ , ultimate tensile strength UTS, uniform elongation  $\epsilon_u$ , elongation to failure  $\epsilon_{total}$

Samples	$\sigma_{0.2}$ (MPa)	UTS (MPa)	$\epsilon_u$ (%)	$\epsilon_{total}$ (%)	$\sigma_{0.2}/UTS$
as-annealed	403	671	25.4	42.6	0.60
1 P	1058	1278	2.7	15.6	0.83
4 P	1461	1781	1.9	9.8	0.82
4P+10 min	1215	1470	5.3	16.6	0.83
4P+20 min	1091	1433	6.5	18.1	0.76

**Microstructure.** Fig. 2(a) and (b) is the cross-sectional views about the as-annealed sample, on which the austenite phase is distributed on specific crystallographic direction. Two special plans called X (the cross-sectional plan of the billet) and Y (formed by pressure direction and extrusion direction) was investigated. The scales of austenite islands vary between several microns to hundreds of microns. They are featured by uniform distributed particles and needles at the vicinity of grain boundaries of primary ferrite.

Fig. 2(c) and (d) display the curved phase boundaries on X plane after one and four passes, respectively. Fig. (e) and (f) clearly show the curved, elongated and ribbon-like austenite phase due to shear stress during pressing.

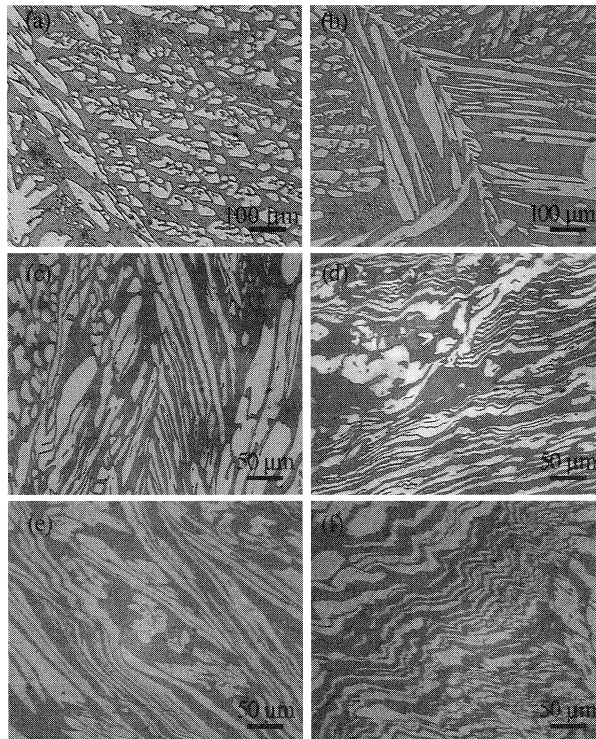


Fig. 2. OM micrograph of microstructures of DSS before and after ECAP (the white is austenite phase and gray is ferrite): (a) and (b) Cross-sectional microstructure of as-annealed sample; (c) and (d) Phase distributions on X plane after one and four passes; (e) and (f) Phase distributions on Y plane after one and four passes

Fig. 3(a) exhibits the curved phase boundary and deformation twin located at the kinks on boundary. Shear deformation is a common feature during ECAP process, which leads to the formation of kinks on grain boundaries<sup>10</sup>. The deformation twin with high density located the kinks revealed those places are strain-concentrated. Fig. 3(b) displays sub grain boundary evolved from dense dislocation walls (DDWs) in ferrite, which have a space size nearly 600 nm. It's noted that the direction of sub grain boundary is consistent with deformation twin, revealed they are both formed under the shear stress simultaneously.

Fig. 3(c) shows large number of microtwin are retained in austenite after four passes, which demonstrate the deformation twin is the main grain refine mechanism for austenite. Fig. 3(d) demonstrates one shear band in ferrite, in which some equiaxed grains were formed.

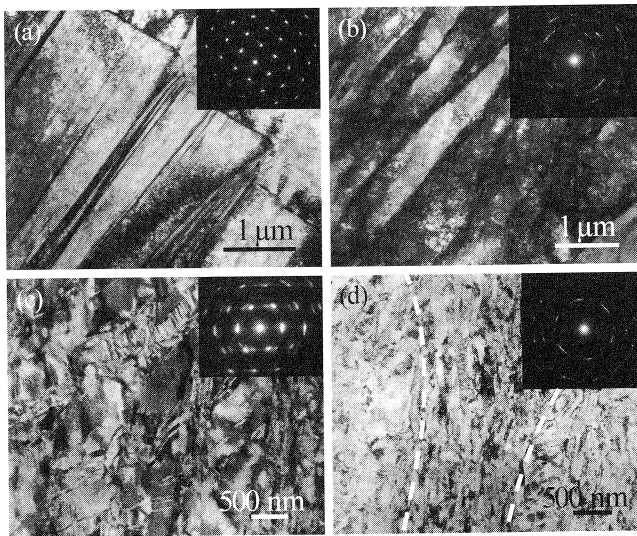


Fig. 3. TEM images on Y planes displaying the typical microstructure of DSS sample after one and four passes: (a and b) Bright-field images exhibiting deformation twin in austenite phase and DDWs in ferrite phase after one pass; (c and d) Retained microtwin in austenite and shear band in ferrite phase

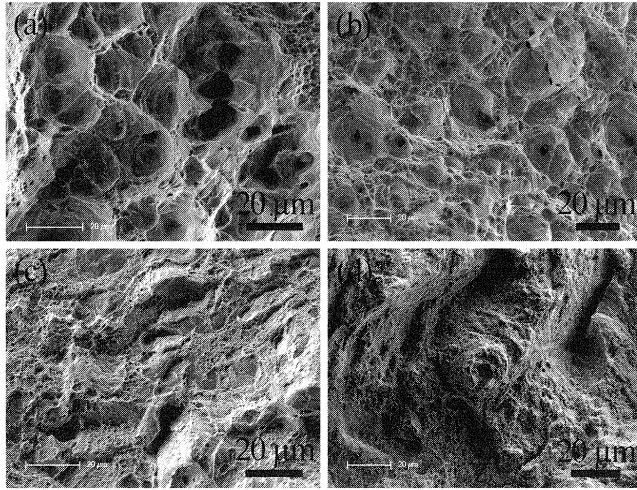


Fig. 4. SEM images of fracture surfaces: (a) Typical dimples of as-annealed samples; (b and c) Shallow dimples after one and four passes; (d) The fracture surface of samples suffered four passes ECAP plus annealing at 700°C for 20 minutes

**Fracture mechanism.** Fig. 4(a) displays the typical fracture surface of as-annealed sample which state large dimples on the fracture surfaces. Similar dimple-like fracture surface has been observed after one pass ECAP process. However, a comparison of Fig. 4(a) and Fig. 4(b) discloses that the dimples were shallower than former. It's impressive that the sample suffered four passes pressing exhibits different features from those suffered only one pass pressing. Fig. 4(c) exhibits this difference that smaller dimples with variant size and micro-cracks. The path of micro-cracks is consistent with the curved phase boundaries, stated the deformation incompatibility occurred on phase boundaries. This fracture features would be beneficial to fracture toughness due to increased fracture area. Fig. 4(d) demonstrates uniform dimples after annealing, but the micro-cracks along phase boundaries still exist.

### Summary

A nano-duplex structure obtained from a type of duplex stainless steel UNS S32304 suffered ECAP was investigated. The mechanical property test and microstructure investigation led to the following summaries:

1. The yield strength in a deformed nano-duplex structure was increased significantly from 402 MPa to 1461 MPa as compared to its coarse-grained counterpart, but tensile ductility was low. After annealing at 700°C for 10 minute, uniform elongation increases to 5.3% with the yield strength of 1200 MPa.
2. Deformed structure shows the deformed twin induced by shear stress is the main grains refine mechanism in austenite, whereas dislocation activities result in grain subdivision in ferrite.

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