

Materials Science and Engineering A 485 (2008) 643-650



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# Influence of processing temperature on the microstructures and tensile properties of 304L stainless steel by ECAP

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Received 11 May 2007; received in revised form 13 August 2007; accepted 23 August 2007

#### Abstract

304L austenitic stainless steel was successfully processed by equal channel angular pressing (ECAP) in the temperature range of 500-900 °C, and the influences of processing temperature on the microstructures and tensile properties were investigated. At temperature below 700 °C, the microstructures were characterized by lamellar structures and many bundles of deformation twins, which led to a high tensile strength but low elongation-to-failure. With increasing the processing temperature up to 800 °C, dynamic and also static recovery took place and more equiaxed subgrains with low dislocation density were obtained. Deformation twins were found to form only in some grains in the form of individual bands. A low strength and a high elongation-to-failure were achieved compared with those processed at low temperature. The best combination of both high strength and large elongation took place at the processing temperature of 800 °C.

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Keywords: Stainless steel; ECAP; Microstructure; Deformation twins; Tensile strength; Elongation

### 1. Introduction

Austenitic stainless steels (SSs) are widely used engineering materials mainly due to their excellent corrosion and oxidation resistance. However, their low yield strength is often a major drawback. To strengthen the stainless steel, microstructure/grain refinement is an effective approach [1,2]. It is now well established that processing through the application of severe plastic deformation (SPD), typically, equal channel angular pressing (ECAP), is especially effective in grain refinement for those bulk polycrystalline metals [3]. Up-to-date, this method has been successfully used to produce several ultrafine-grained (UFG) steels, such as low-carbon steels [4,5], ferrite–martensite dual-phase steels [6], etc. As a result, the UFG steels exhibited superior mechanical properties, such as high strength and sometimes good ductility, compared with their coarse-grained (CG) counterparts [5–8].

During the process of ECAP, many processing parameters can affect the deformation structures of materials. They are the die

angle, which determines the strain introduced into the material during each deformation pass [9]; the pass number, which corresponds to the total accumulated strain applied to the material; the processing route, which involves rotating the billet between passes [10,11]; the processing speed [12] and the processing temperature [13]. Among these parameters, the effect of processing temperature has been investigated in several materials, such as Al [14], Al-Mg alloys [13,15] and Cu [16]. Increasing the processing temperature (up to 300 °C) caused a fast dynamic recovery in Al and Al-Mg alloys, which resulted in an increase of grain size and the grain shape becoming more equiaxed-like [13–15]. Compared with Al deformed by ECAP at similar homologous temperature ( $\sim 0.32T_{\rm m}$ , 150 °C), the distinct difference in the deformation microstructure of Cu was the presence of dynamic recrystallization [16]. It is well known that dynamic recovery/recrystallization occurs easily in those materials with medium-high stacking fault energy (SFE) after large plastic strain. Reduction of SFE decreases the rate of dynamic recovery and facilitates the occurrence of deformation twinning. Recently, 316L SS was processed by ECAP and both dynamic recovery/recrystallization and deformation twinning were observed at temperature as high as 800 °C ( $\sim 0.6T_{\rm m}$ ) [17]. The occurrence of deformation twinning implies that twinning might be beneficial to the microstructure/grain refinement

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<sup>0921-5093/\$ –</sup> see front matter @ 2007 Elsevier B.V. All rights reserved. doi:10.1016/j.msea.2007.08.067

of austenitic SS at high processing temperature. Accordingly, the mechanical properties of 316L SS displayed different features, such as apparent tension–compression asymmetry at lower processing temperature and an increase of toughness at higher processing temperature [17].

By checking the materials processed by ECAP in literature, most of them are relatively soft, such as Cu [16], Al alloys [13–15], Fe [18] and low-carbon steel [4,5]. These materials can be processed at RT in order to obtain a good grain refinement effect. However, for hard material, such as SS [17,19], Ti alloys [19,20], Ni-Ti shape memory alloy [21] and W [22], they were commonly processed at elevated temperature due to the limitation of processing conditions. Therefore, the processing temperature becomes an important parameter that can affect the microstructure and related mechanical properties of these materials obviously. As to SS, though there have been several studies on the microstructure/grain refinement of SS processed by SPD [23–27], the application of ECAP to SS is still quite rare and the influences of the processing temperature on the deformation microstructures and corresponding mechanical properties are not adequately explored. In this work, 304L austenitic SS was selected to be processed by ECAP for one pass in the temperature range from 500 to 900 °C. The main goal is to investigate the influences of processing temperature on the microstructures and room-temperature (RT) tensile properties developed by ECAP.

#### 2. Experimental procedure

A hot-rolled commercial AISI 304L type austenitic SS was used in this investigation. The chemical composition of the material is in weight percent of 0.025 C, 18.75 Cr, 10.96 Ni, 0.005 S, 0.0068 P, 0.36 Si, <0.5 Mn, and the balance Fe. The hot-rolled billet was annealed at 1150 °C for 2 h. Samples for ECAP processing were cut from the as-annealed billet with dimension of  $\emptyset8 \text{ mm} \times 45 \text{ mm}$ .

The ECAP experiments were conducted using a split die with two channels intersecting at inner angle of 90° and outer angle of 30°, which yields an effective strain of  $\sim$ 1 by a single pass. Before extrusion, the die was preheated to 300 °C and the samples were heated up to the deformation temperature and held for 10 min. Samples were pressed for only one pass at a pressing speed of  $\sim$ 9 mm/s and at high temperatures of 500, 600, 700, 800, and 900 °C, respectively. After pressing, the samples were cooled in air to RT.

The microstructures on the transverse cross-sections of the as-pressed samples were observed by both optical microscope (OM) and JEM-2000FXII transmission electron microscope (TEM, operating at 200 kV). For OM, samples were ground on SiC paper, polished and etched in a solution of 10% Cr<sub>2</sub>O<sub>3</sub> + 90% ethanol. While for TEM, thin foils were first mechanically ground to about 40  $\mu$ m thick and then finally thinned by a twinjet polishing facility using a solution of 10% perchloric acid and ethanol at room temperature.

Tensile specimens with a dog-bone shape were cut from the as-pressed billets with the tensile axes oriented parallel to the extrusion direction. The gauge dimension of the tensile specimens is  $1.5 \text{ mm} \times 3.0 \text{ mm} \times 15 \text{ mm}$ . Tensile experiments were



Fig. 1. Optical microstructure of the initial state.

performed on an Instron 8871 testing machine at a constant crosshead speed of 1 mm/min at room temperature.

#### 3. Results and discussion

#### 3.1. Microstructure observations

### 3.1.1. Optical microstructures

Fig. 1 is the optical microstructure of the stainless steel after hot-rolling and annealing at 1150 °C for 2 h. The microstructures are characterized by equiaxed grains with grain sizes in a range of 40–120  $\mu$ m. Additionally, there are some annealing twins with orientations varying from grain to grain. Inclusions with sizes of several micrometers can only be detected occasionally.

Figs. 2(a)–(f) present the typical optical microstructures of the samples after ECAP processing at different temperatures. It can be seen that for the samples processed at low temperature, below 800  $^{\circ}$ C (Fig. 2(a)–(d)), the deformation microstructures mainly consist of severely elongated grains, as well as very high density of lamella in the interior of some grains. One of the detailed deformation structures for the sample processed at 500  $^{\circ}$ C is enlarged and shown in Fig. 2(b). As indicated by the white arrows, many parallel thin lamellae with alternative dark-bright contrast were formed and curved together along certain crystallographic habit plane. Similar deformation structures were also observed in other austenitic steels during tension and are believed to be deformation twins [28]. With increasing the processing temperature, the microstructures display the following characteristics (see Fig. 2(e) and (f)): (a) the shape of grains became equiaxed-like; (b) the density of lamella in grain interior decreased significantly. Fig. 2(g) is a high magnification image of the sample processed at 900 °C, showing that the grains are almost lamella-free. These deformation features suggest that deformation twinning is not prevalent any more at such high temperature.

#### 3.1.2. TEM microstructures

TEM observations were carried out in order to characterize the deformation microstructures in detail. In most cases, two kinds of deformation microstructures were observed, i.e. deforDownload English Version:

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