Effect of equal channel angular pressing on the microstructure and mechanical properties of AISI type 304 austenitic stainless steel

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Abstract. The present paper describes the effect of severe plastic deformation on the microstructure and mechanical properties of AISI type 304 stainless steel by equal channel angular pressing (ECAP) at room temperature. The strain-induced martensitic transformation occurred in the specimens, and martensite phase increased with increasing strain up to 42% for three passes of ECAP. X ray diffraction was used to identify the strain-induced martensite phase and its volume fraction. The martensite phase, mechanical twins and micro hardness have increased with increasing passes of ECAP. Microstructures of specimens show that with increasing strain, subgrains less than microns are susceptible to be created by fragmentation of twins.

Introduction

Austenitic stainless steels (ASSs) usually have excellent corrosion resistance and good formability. However, they have relatively low yield strength about 200MPa in the annealed state. Therefore, they seem to be less suitable for structural applications. There are various strengthening mechanisms for ASSs, such as grain refining, transformation strengthening and work hardening. ASSs generally have a high strain-hardening coefficient, and therefore cold work is a suitable strengthening method [1]. As is well known, ASS is one material with low SFE in which deformation twinning occurs readily under plastic straining [2]. An important characteristic of these materials is their sensitivity to deformation-induced martensite transformation (DIMT) under plastic deformation due to the energetic instability of the austenitic structure [3]. This suggests that a refinement mechanism via DIMT is possible in those materials susceptible to DIMT, such as austenitic steels and some shape-memory alloy. There has been little investigation of the influence of deformation twinning upon grain refinement during ECAP. In contrast, the grain refinement process via deformation twinning has been revealed by other SPD methods. For instance, in Inconel 600 alloy (SFE=28 mJ m⁻² [4]) and 304 stainless steel (SFE=21 mJ m⁻² [4]), deformation twins and their interplays with dislocations dominate the grain refinement process during surface mechanical attrition treatment (SMAT) [5, 6]. The present work focused on the influence of ECAP processing on the phase transformation, grain refinement and hardness of the 304 SS.

Experimental materials and procedures

The material used in this study was a commercial 304 SS with chemical composition in Table 1. As received 304 SS was solution-treated at 1150°C for 2h and quenched in water to achieve homogeneity. Samples were cut with wire electro-discharge machining to 45 mm lengths and diameters of 6 mm. These samples were fitted in Fe sheaths with diameters of 20 mm and lengths of...
It should be noted that these Fe sheaths are in contact with the die walls during ECAP processing. All billets were processed by ECAP at room temperature using a die with an internal channel angle, $\varphi$, of 90° and an outer arc of curvature, $\psi$, of 20°. The pressing was conducted using route BC in which the sample is rotated around their longitudinal axis by 90° in the same direction between each consecutive pass. The process of ECAP was carried out for a total of three passes using a Molybdenum disulfide (MoS$_2$) lubricant at room temperature and a pressing speed of ~1 mm/s.

Table 1. Composition of 304 austenitic stainless steel (weight percent).

<table>
<thead>
<tr>
<th>Type</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Cr</th>
<th>Ni</th>
<th>Cu</th>
<th>Mo</th>
<th>P</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>304L</td>
<td>0.0063</td>
<td>0.525</td>
<td>1.79</td>
<td>18.51</td>
<td>8.00</td>
<td>0.667</td>
<td>0.339</td>
<td>0.036</td>
<td>Base</td>
</tr>
</tbody>
</table>

To study the phases present in the initial and ECAP specimens, X-ray diffraction (XRD) with Cu-Kα radiation was used in 10–110° range of the 2θ angle with a step size of 0.02 (2θ) and scan step time of 0.35 s. Also the volume fraction of phases was calculated from the peak intensities [8]. The minimum crystallite size of the three pass ECAP sample were calculated from the line broadening of fcc-$\gamma$ (111), (200), (220), (311) and (222) Bragg diffraction peaks using the Scherrer–Wilson method [9]. Microhardness measurements were taken with a load of 100 gr for 10 s and at least 7 measurements were used to determine the average hardness of each specimen after removing the highest and lowest values. The specimens were electroeched in a nitric acid at a voltage of 3 V for 30 second. The microstructures of the samples were observed by a Zeiss optical microscope (OM).

Results and discussion

**Microstructural study.** Deformation twinning is an important deformation mechanism in stainless steels, especially at large plastic strains. At the beginning of the ECAP process, severe plastic shear strain produced a high density of deformation twins (Fig. 1). These twin boundaries subdivided the original grains into thin twin-matrix lamellae and/or submicron-sized rhombic blocks (Fig. 2). With subsequent pressing, the twin-matrix lamellae were successively cut up due to the shear deformation from different directions, because the sample was rotated for 90 in the next pass (Fig 1b–d). Tao et al. [10] and Wang et al. [11] have shown the micro-mechanism of twin fragmentation during the formation of nanostructures in the top surface layers of Inconel 600 alloy and Cu samples subjected to surface mechanical attrition treated (SMAT). Their observations demonstrated that a lot of dislocations and/or dislocation walls rather than micro-twins were introduced into the thin twin–matrix lamellae in order to accommodate plastic deformation. These dislocation walls were eventually developed into GBs, cutting the two-dimensional twin–matrix lamellae into three-dimensional equiaxed nanograins. In the case of ECAP processing, nanocrystalline $\gamma$ austenite can also be achieved via the twin fragmentation mechanism. In the three-pass sample, minimum crystallite with a size of 35 nm can be measured from peak widening of XRD pattern. Figure. 2 shows the typical microstructure of austenite in the three-pass sample, which is characterized by many fine fragments of twin lamellae. These fragments formed ultra fine grains (UFG). A low SFE is also beneficial to twinning for fcc metallic materials, the predominant deformation mechanisms at high plastic strain change gradually from dislocation slip to deformation twinning with decreasing SFE [12,13]. Accordingly, the grain refinement mechanisms are also transformed from the dislocation-subdivision mechanism to the twin fragmentation mechanism.
Deformation-induced martensite transformation (DIMT) during ECAP. Figure 3 shows XRD patterns of the annealed and ECAP specimens at different passes. The microstructure of the solution-treated specimen was approximately fully austenitic. The diffraction peaks of ε-martensite cannot be easily found, because, a weak ε(1011) peak can be detected at lower strain and the ε phase is only an intermediate phase during the formation of α'-martensite [14]. With increasing the strain, the intensity of austenite peaks is gradually decreasing and martensite peaks appear and their intensity increases in the spectrums. Finally the microstructure is changed to 42% α'-martensite due to the ECAP processing.

Figure 4 shows the volume fraction of strain-induced α'-martensite in the AISI 304 stainless steel ECAP at room temperature after different passes. It can be seen that the austenitic microstructure is transformed to α'-martensite during ECAP processing. The resulting transformation curve shows that the slope is increased by increasing passes of ECAP. This behavior shows that in the primary stages of deformation instead of increasing the α'-martensite content, new sites for strain-induced martensite such as twins were created [14] (Fig. 1). The region of twins is etched dark and by increasing strain they are increased.

It is well-known that shear band intersections can be very effective strain-induced nucleation sites. The shear bands can be in the form of ε-martensite, mechanical twins, or dense stacking fault bundles [15].

Traditionally, the γ → α' transformation occurs via two shear strains of specific defects (mostly characterized by the type of a/6 < 112 > Shockley partial dislocation) on the {111}_γ planes [16]. Depending on the crystallographic directions of the second shear deformation, the K-S or the Nishiyama–Wessermann orientation relationship could result. In K-S relationship, the {111}_γ is converted into the {110}_α' with the <110>_γ parallel to the <111>_α' [17]. This mechanism could be reasonable for deformation-induced martensite transformation (DIMT) in large-sized γ grains.
Fig. 3. X-ray diffraction patterns of the ECAP and annealed AISI 304L specimens at room temperature.

Fig. 4. The volume fraction of $\alpha'$-martensite in the ECAP austenitic stainless steel AISI 304L after different passes at room temperature.

**Hardness tests.** The effect of ECAP processing on the Vickers hardness is shown in Fig. 5. The hardness values were found to increase with strain. The cause of the change in hardness with deformation may be attributed to the effect of the increased dislocation density [14]. The Vickers hardness increases from 200 to 470 due to the applied cold working.

![Hardness Test Graph](image)

Fig. 5. Effect of ECAP processing on the Vickers hardness of AISI 304L.

**Conclusion**

1. Ultra fine grain (UFG) microstructures could be obtained via the twin fragmentation mechanism in an ultra-low carbon SS by means of ECAP at room temperature.
2. The metastable austenite was transformed to the strain-induced α'-martensite by ECAP processing. With increasing the strain, more than 40 vol.% of austenite was transformed to α' martensite by 3 passes of ECAP.

3. Hardness of the ECAP specimens was increased with increasing strain up to 470 Vickers for 3 pass specimen, which was two times higher than primary material.

References