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# Optimizing strength and ductility of austenitic stainless steels through equal-channel angular pressing and adding nitrogen element



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#### ABSTRACT

Two austenitic stainless steels were processed by equal-channel angular pressing to systematically investigate the influences of alloying nitrogen and severe plastic deformation on the strength and ductility. With increasing the number of ECAP pass and adding nitrogen element, the density of deformation twins increased. It is shown that the two steels exhibit a similar general trend that the strength increases and the ductility decreases with increasing the deformation strain, but an enhanced strength–ductility synergy can be achieved by adding nitrogen element.

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#### 1. Introduction

Austenitic stainless steels are one group of the currently favored structural multifunctional materials considered for many applications due to its attractive combination of excellent ductility and formability paired with high strength, stability of its austenitic structure, and superb corrosion and irradiation resistance [1-3]. However, their lower yield strength is a major drawback, which limits their technological applications. Microstructural refinement is an effective approach for strengthening, which can be induced by plastic deformation such as cold rolling (CR) [4], equal channel angular pressing (ECAP) [5], high-pressure torsion (HPT) [6] and dynamic plastic deformation (DPD) [7]. Among the techniques above, ECAP is superior to the other techniques in permitting the application of a large amount of strain without a significant change of sample cross section, control over the development of grain morphology and texture, and ease of process. Although microstructural refinement can result in a record-high strength, ductility and workhardening are decreased considerably due to the inability to accumulate dislocations with a saturation of dislocations [8,9]. This critical shortcoming of ultrafine-grained/nanostructure (UFG/NS) materials has restricted their practical applications, although several strategies have been proposed to improve their ductilities [10–13].

Except for microstructural refinement, alloying is another conventional strengthening mechanism by the interactions between

solution atoms and moving dislocations, which could potentially be beneficial to the promotion of ductility [14]. Thus, a simultaneous increase in strength and ductility was achieved recently in UFG/NS Cu and Cu–Al/Cu–Zn alloys processed by ECAP or HPT through appropriate tailoring of the stacking fault energy (SFE) [15–19]. Therefore, tailoring the SFE by alloying design may be a utilizable approach to upgrade the mechanical properties of UFG/NC materials.

Accordingly, some experiments were conducted on 316L and 316LN steels with chemical compositions listed in Table 1 using the ECAP to explore the influences of alloying and the SPD processing on strength and ductility. Thus, we expect to gain a relatively homogeneous microstructure in UFG/NC 316L(N) steels through the ECAP technique and critically discuss several questions in this study: how does the nitrogen element affect the microstructural evolution during ECAP? Can the strength and ductility be simultaneously improved by adding the nitrogen content in 316L steels? These essential issues are meticulously elucidated through systematic investigation and detailed analysis.

#### 2. Experimental procedure

The 316L and 316LN steels were produced by vacuum induction melting and electro-slag remelting. The nitrogen element in 316LN steel was added through the intermediate alloy of CrN in the process of melting. The as-received steels have been hot-forged and solution-heat-treated in a quartz tube under vacuum at  $1100\,^{\circ}\text{C}$  for 4 h followed by water quenching. Before the ECAP processing, some round

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**Table 1**Chemical compositions of the 316L and 316LN stainless steel.

Material	Composition (wt%)								
	Cr	Ni	Mo	С	N	Si	Mn	S	P
316L 316LN	17.2 15.3	14.61 12.54	2.16 2.50	0.01 0.01	0.01 0.14	0.24 0.21	1.28 1.48	0.005 0.003	0.007 0.008

cylindrical billets (8 mm in diameter and 45 mm in length) were cut from the sheet by means of electrical discharge machining (EDM).

The ECAP procedure was performed using a die fabricated from tool steel with two channels intersecting at an inner angle of  $120^\circ$  and an outer angle of  $30^\circ$ , accordingly yielding an effective strain of  $\sim\!0.628$  for each pass. All the rods coated with a MoS $_2$  lubricant were processed at room temperature (RT).

Microstructures of the as-ECAP and the annealed samples were characterized by using transmission electron microscopy (TEM, FEI Tecnai F20) operated at 200 kV. Thin foils for TEM observations, cut from the Z plane in the center of the pressed rods using spark cutting, were mechanically ground to about 50  $\mu m$  thick and then thinned by a twin-jet polishing method in an electrolyte consisting of 8% perchloric acid and 92% alcohol at  $-15\,^{\circ}\text{C}.$ 

Tensile specimens with a dog-bone shape were cut into a gauge length of 8 mm, with a width of 2 mm and a thickness of 1 mm from the processed billets along the extrusion direction of ECAP. In the absence of any effects of the microstructure inhomogeneity on the tensile behavior, the specimens for comparation are selected in the same place through the transverse section. Tensile tests were carried out at RT with an Instron 5982 testing machine operating at a strain rate of  $1\times10^{-3}~\text{s}^{-1}$  for the as-received and ECAP-processed specimens.

#### 3. Results and discussion

#### 3.1. Microstructure observations

The typical microstructures of the two materials after 1- and 4passes are displayed in Fig. 1. After ECAP for one pass, the microstructures mainly consist of elongated cells separated by geometrically necessary boundaries (GNBs), with a high density of dislocations in the two materials (Fig. 1(a) and Fig. 2(a and b)). Moreover, in some grains, some deformation twins can be found, as illustrated in Fig. 1(b and c) and Fig. 2(c). Substantially different microstructural features were observed in different areas of the samples. This can be explained by the orientation dependence of the deformation mechanisms (slip versus twinning). After ECAP for 4 passes, more deformation twin lamellas and some shear bands are found to coexist for both 316L and 316LN steels, as shown in Fig. 1(d-f) and Fig. 2(d-f), in which the arrows point to some shear bands. It is indicated that with an increase in the number of ECAP pass and the addition of nitrogen atoms, the density of deformation twins is obviously increased as illustrated in Fig. 3. The microstructure evolution can be mainly attributed to the stress state during ECAP and the effects of nitrogen addition and will be discussed in detail below.

Recently, it is shown that the nucleation of deformation twins in austenitic steels strongly depends on the SFE and applied stress level [20]. Byun [21] reported that during deformation of 316 stainless steel, a variety of deformation structures was produced depending on the applied stress level: (1) dislocation tangles were dominant at low equivalent stress of 400 MPa; (2) isolated stacking faults smaller than about 1  $\mu m$  were formed in the stress range from about 400–600 MPa; and (3) twin bands became dominant

at stress exceeding 600 MPa at RT. In the present work, during the first pass of ECAP through the die at RT steady extrusion occurred at a relatively low applied normal stress, which may not exceed the critical stress for twinning ( $\sigma_t$ ) for most grains. The applied normal stress increases with the increase of the ECAP pass. Therefore, deformation twins develop most rapidly after the first pass of ECAP through the die. The density of twins increases with the increase of the ECAP pass because the applied normal stress in most grains exceeds the critical stress  $\sigma_t$ .

When the material strength is increased such as by the addition of interstitial atoms or by applied high strain level, deformation twinning will occur because of the effect of stress on the partial dislocation separation [21]. Meanwhile, the propensity of twining is also promoted by solutes raising the shear stress in the slip plane, reducing SFE and increasing the planarity of slip. It was argued that the nucleation stress for twinning is directly proportional to the SFE [22]. The SFE in austenitic stainless steels of 300 series varies broadly from low to high values: 9.2–80.7 mJ/m² [23]. Depending on the chemical composition, the SFE of the two steels can be estimated by the following empirical relation [24]:

SFE [m] 
$$m^{-2}$$
] = 25.7+2Ni+410C-0.9Cr-77N-13Si-1.2Mn (1)

From the alloy chemistry (Table 1), Eq. (1) yields a medium SFE of 34.8 mJ/m<sup>2</sup> for the 316L steel and 25.8 mJ/m<sup>-2</sup> for the 316LN steel under investigation, respectively. The decrease in SFE leads to a decrease in the twinning stress and thus to the formation of deformation twins at early stage of deformation in the 316L(N) steels [22], which is similar to the results in Cu–Al and Cu–Zn alloys [15–19].

Therefore, on the one hand, the introduction of N atom may lead to an interaction between dislocations and solute atoms because they can reduce the dislocation mobility and limits dynamic recovery [25], which plays a crucial role in annihilating dislocations and rearranging them in a lower energy configuration of cell walls. Whereas, on the other hand, the addition of N element into the austenitic (fcc) matrix lowers the SFE and thereby restricts the occurrence of cross-slip [25,26]. Both the addition of solute atoms and decrease in the SFE may significantly enhance the propensity to deformation twinning through restricting the dislocation motion to form the planar-type dislocation configuration or a decrease in twinning stress [22].

#### 3.2. Tensile properties

Having deciphered the effects of ECAP and N element on the microstructures and the corresponding deformation mechanisms, it is necessary to investigate their mechanical properties with respect to these microstructures. Typical tensile engineering stress-strain curves of the 316L(N) steels subjected to different passes of ECAP and the as-received CG sample are presented in Fig. 4(a) and (b). It can be seen that the ECAP processing can significantly improve the tensile strength in both 316L and 316L(N) steels, but there is an obvious loss in ductility. The mechanical properties, including yield stress (YS), ultimate tensile strength (UTS), uniform elongation (UE) and total elongation (TE) as a function of the number of ECAP passes are summarized in Fig. 4(c and d) and Table 2. In order to reveal the work hardening behavior of the present samples, the engineering stress-strain curves in Fig. 4(a and b) were converted into the true stress–strain curves. Work-hardening rate  $\Theta$  vs. true strain e (a) and vs. true stress are shown in Fig. 5.

On the macroscale, the Considère criterion governs the onset of localized deformation in tension:

$$\Theta = d\sigma/d\varepsilon \le \sigma \tag{2}$$

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