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An investigation into the room temperature mechanical properties of nanocrystalline austenitic stainless steels

Mostafa Eskandari*, Abbas Zarei-Hanzaki, Hamid Reza Abedi

School of Metallurgy and Materials Engineering, College of Engineering, University of Tehran, Tehran, Iran

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ABSTRACT

The present work has been conducted to evaluate the mechanical properties of nanostructured 316L and 301 austenitic stainless steels. The nanocrystalline structures were produced through martensite treatment which includes cold rolling followed by annealing treatment. The effect of equivalent rolling strain and annealing parameters on the room temperature mechanical behavior of the experimental alloys have been studied using the shear punch testing technique. The standard uniaxial tension tests were also carried out to adapt the related correlation factors. The microstructures and the volume fraction of phases were characterized by transmission electron microscopy and feritscopy methods, respectively. The results indicate that the strength of nanocrystalline specimens remarkably increases, but the ductility in comparison to the coarse-grained one slightly decreases. In addition the strength of nanocrystalline specimens has been increased by decreasing the annealing temperature and increasing the equivalent rolling strain. The analysis of the load–displacement data has also disclosed that the universal correlation of linear type (UTS = $m\tau_{max}$) between shear punch test data and the tensile strength is somehow unreliable for the nanocrystalline materials. The results suggest that the actual relation between the maximum shear strength and ultimate tensile strength follows a second order equation of type UTS = $a\tau_{max}^2 - b\tau_{max} + c$.

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1. Introduction

The austenitic stainless steels are one of the most commonly used stainless steels characterized by favorable ductility and excellent overall corrosion resistance used in biomedical applications. However their relatively low yield strength (about 170 MPa) confines the application of this grade of steels [1]. To resolve this shortcoming, the strengthening of stainless steels through grain refinement up to the nano/submicron-grained scale has been recently proposed for structural applications, especially in lightweight components [2]. The martensite-treatment is one of the most effective thermo-mechanical processing routes to produce nanocrystalline stainless steels [3–6]. This consists of a severe cold rolling path followed by a proper annealing treatment.

The austenitic stainless steels exclusively consist of metastable austenite at room temperature, which may transform to martensite by deformation below the M_d temperature. Accordingly the martensite crushes during deformation, thereby increasing the concentration of lattice defects inside the matrix. Finally, the martensite is retransformed to austenite during subsequent annealing, leading to noticeable grain refinement. To this end as was already reported, the nanocrystalline 316L stainless steels could possess a highly enhanced combination of mechanical properties (e.g. tensile strength of 1385 MPa and elongation of 5.5%) [5].

In particular conditions where only a small amount of material is available, the small specimen testing methods such as shear punch testing technique (SPT) are considered to reduce the costs and difficulties of the standard tensile specimen preparation [7,8]. Hence, SPT is a promising method to resolve the situations where conventional mechanical testing techniques are practically not possible such as weld joints [9], coatings, failed components, very small volumes of irradiated materials [10], biomaterials, composites and metallic glass [11]. The load-displacement curve obtained during the SPT is very similar to that of a conventional uniaxial tensile test one [12]. It shows a linear region, followed by non-linear increase of load with displacement up to a maximum and then the load starts decreasing. This eventually ends to separation of the punched out piece. The strength and ductility parameters obtained by analyzing the SPT curve may well be correlated with the corresponding conventional tensile properties [13].

The nature of tensile-shear correlations obtained from SPT has been a subject of debate over a period of years [14–16]. Early studies showed that the relation between the shear and the uniaxial tensile is linear in nature. However, the shear punch-tensile correlations equations for the strength and ductility parameters are





^{*} Corresponding author. Tel.: +98 21 61114167; fax: +98 21 88006076. *E-mail address:* m.eskandari@ut.ac.ir (M. Eskandari).

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mostly empirical in nature due to the complex stress states in the clearance zone. Karthik et al. [14] have reported the linear tensile-shear correlation (σ_{ys} = 1.73 τ_{sys}) for the coarse grain 316 stainless steel.

To the best of authors' knowledge, no work has been reported so far on the room temperature mechanical properties of nanograined 316L and 301 stainless steels by SPT method. Therefore, the present study has been aimed to *investigate* the mechanical properties of nanostructured 316L and 301 austenitic stainless steels using SPT technique. Moreover, the tensile–shear correlations obtained from SPT technique has been discussed.

2. Experimental procedure

2.1. Alloys preparation and thermomechanical treatment

The experimental materials were 316L and 301 stainless steels, the composition of which are given in Table 1. To achieve nanocrystalline microstructures, the martensite treatment was conducted to the hot-rolled specimens with the thickness of 7 mm (Fig. 1). A set of multi-pass cold rolling was performed at temperatures of -196, -15 and 0 °C under the strain rate of 0.5 s⁻¹ down to the thickness reduction of 10–95%. The subsequent annealing treatment was done in the temperature range of 750–850 °C for 60–1800 s.

2.2. Microstructure characterization

The microstructures were analyzed using scanning electron microscopy (SEM Philips X230) after electro-etching in 65% nitric acid solution. Transmission electron microscopy (TEM) was performed by a JEOL 2010 one operated at 200 kV. The TEM specimen was prepared by mechanical grinding of the sliced samples to a thickness of approximately 100 μ m. The disk of 3 mm in diameter was then punched out from them. The disks were dimpled on both sides using a Gatan dimple grinder with 1 μ m diamond suspension. Electron transparency was obtained by twin-jet electro-polishing using 24 V DC at a temperature of -30 °C. A solution of 5 vol.% perchloric acid in methanol served as the electrolyte. The Clemex software was used to calculate the grain size. The amount of magnetic phases was calculated using a Feriteoscope (Fischer MP30) device.

2.3. Tensile and hardness tests

The tension tests were performed at room temperature according to ASTM E8 standard [17] using a tensile machine (Hounsfield H50ks) at a crosshead speed of 0.2 mm/min. The tensile direction was parallel to the rolling direction. The hardness was measured by Vickers method applying a 10 kg force.

2.4. Shear punch testing

The shear punch tests were performed at room temperature using a special designed fixture (Fig. 2) [8]. The test fixture consists of a flat punch of 3.00 mm diameter made of the hardened tool steel (H13) and a set of dies between which the specimen is clamped. The diameter of the receiving hole in the lower die is

Anneal 750-850 °C 1-30 min Cold roll Red. 95% Time

Fig. 1. The schematic diagram of the martensite process to obtain nanocrystalline austenitic stainless steels.



Fig. 2. The schematic demonstration of shear punch test setup.

3.04 mm. The specimens were sliced to the approximate thickness of 500 µm through wire cutting method. In order to ensure removing any surface oxide, each slice was mechanically polished. Finally, minimum three punches were carried out on each slice. The stroke movement speed was set to 0.2 mm/min for all punch tests. As the strain and strain rate are in angular form in SPT, a similar

 Table 1

 Chemical composition of 316L and 301 stainless steels used in this study (wt%).

| Steel | С | Si | Mn | Ni | Cr | Мо | Ti | V | Р | S | Cu | Nb |
|-------|------|------|------|------|-------|------|------|------|------|------|------|-------|
| 316L | 0.01 | 0.36 | 1.54 | 11.4 | 18.20 | 1.68 | 0.08 | 0.14 | 0.03 | 0.03 | 0.28 | 0.09 |
| 301 | 0.11 | 0.67 | 0.65 | 6.91 | 16.20 | 0.27 | 0.02 | 0.06 | 0.02 | 0.03 | 0.53 | 0.003 |

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