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Ultrastrong nanocrystalline stainless steel and its Hall-Petch relationship in the nanoscale

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ABSTRACT

An ultrastrong nanocrystalline stainless steel with a yield strength of ~2.0 GPa was reported and the Hall-Petch equation of the stainless steel in the nanoscale was determined. The effects of the grain refinement and element segregation on the mechanical behaviors of the nanocrystalline stainless steel were analyzed and discussed. The validated Hall-Petch equation for the stainless steel in the nanoscale regime could provide a guideline for Hall-Petch strengthening of the stainless steel with grain refinement.

Nanocrystalline metallic materials exhibit extraordinary mechanical, biological and chemical properties. The mechanical strength of the metallic materials could be adjusted by modulating their grain structure and element segregation at the grain boundaries. According to the classical Hall-Petch equation [1, 2], the yield strength is inverse to the square root of the grain size of the materials. Grain boundary can block the dislocation movement and result in dislocation pile-up when the materials undergo plastic deformation. 316L stainless steel (316L SS) is widely used in the fields of biomedical engineering, structural engineering and civil engineering, et al. due to its good biocompatibility, excellent corrosion resistance and good formability. However, one of the drawbacks of the 316L SS is the low mechanical strength. The yield strength and ultimate strength of the 316L SS is about 250 MPa and 515 MPa, respectively [3]. Grain refinement has been used for the mechanical strengthening of the 316L SS for decades.

For example, Chen et al. developed a nanocrystalline 316L SS (NC-316L SS) with an average grain size of 40 nm. The nanohardness and yield stress of this NC-316L SS is 4.5 GPa and 1.45 GPa, respectively, which are 5 times higher than that of the coarse-grained 316L SS (CG-316L SS) [3]. Roland et al. also developed a NC-316L SS whose strength is comparable to titanium alloys. The developed NC-316L SS could have a yield stress varying from 1.4 GPa to 1.8 GPa [4]. Wang et al. fabricated a NC-316L SS with an average grain size of 62 nm through the high-pressure torsion (HPT) and the yield strength of this NC-316L SS is 1360 MPa. Furthermore, they found the post-deformation annealing could introduce further strengthening to the NC-316L SS. The yield strength of the annealed NC-316L SS can be 2230 MPa [5]. The extra strengthening of the NC-316L SS after annealing could be attributed to the element segregation and nanoscale grain boundary stabilization. Abramova et al. directly observed the Mo-Cr-Si rich grain boundary segregation in the NC-316L SS deformed at elevated temperature (673 K). They demonstrated that the grain boundary segregation can lead to significant enhancement of the yield stress in the NC-316L SS. In their study, the average grain size of the NC-316L SS fabricated at the room temperature and elevated temperate (673 K) is 40 nm and 90 nm, respectively. Both of the NC-316L SS have an enhanced yield strength about 1700 MPa [6]. However, the mechanical behavior of the NC-316L SS with an average grain size smaller than 30 nm has not been reported yet. Investigation of the Hall-Petch relationship of the NC-316L SS in the nanoscale regime is meaningful for the future grain refinement and mechanical strengthening of the austenite 316L SS.

In this study, the NC-316L SS with the average grain sizes varying from 10 nm to hundreds of nanometers was successfully fabricated by surface mechanical attrition treatment (SMAT) at room temperature

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[7–14]. Fifty steel shots with the diameter of 3 mm were accelerated by a vibrating solid surface driven by high frequency ultrasonic signal in an enclosed chamber. The multidirectional impacts of the shots on the 316 L stainless steel surface resulted in the grain refinement of the materials. The working distance and peening time is 10 mm and 600 s, respectively. A NC-316L SS (~25 nm) structure with a yield strength of 1.91 GPa, which is the highest ever reported in the literatures for the NC-316L SS deformed at room temperature and without post-heat treatment, was identified by micro-pillar compression test and validated by the nanohardness tests. Even higher yield stress, namely 2.0 GPa, is guaranteed on the NC-316 L SS with the average grain size of 15 nm at the topmost surface according to the nanohardness measurements.

Fig. 1(a) is the Scanning Electron Microscope (SEM) characterizations of the etched NC-316L SS from the cross-sectional direction. The NC-316L SS specimen was polished by using diamond pastes firstly, and then etched at room temperature in a solution of 20 ml nitric acid, 20 ml hydrochloric acid and 20 ml distilled water. Fig. 1 (b) illustrates the gradient NC-316L SS surface layer characterized by means of the Focus Ion Beam (FIB) channeling contrast image technique [15–17]. The average grain size of the NC-316L SS increases gradually with the increment of the depth from the topmost surface. The extremely fine NC-316L SS structure was characterized by Transmission Electron Microscope (TEM) as shown in the Fig. 1(c). The average grain size of this surface layer (~3 μm thick) is around 10 nm according to the statistical analysis of the characterization results. The TEM sample was prepared by the FIB and lift-Out method by using the FEI QUANTA 3D FEG SEM/FIB equipped with the Omniprobe AutoProbe 200 lift-out system and characterized by using an FEI Tecnai TEM operated at 200 kV. With the increment of the depth from the topmost surface to 15 μm, the average grain size of the gradient NC-316L SS surface layer increases from 10 nm to 115 nm gradually as illustrated in the Fig. 1 (c)–(e). It should be noted that there is no observation of twin boundaries in the nano-structures closing to the topmost surface as illustrated in the Fig. 1(c)–(d). Nevertheless, there are many twins in the large grains deep in the matrix as shown in the Fig. 1(a). The twin structure may be further refined into extremely fined nanocrystalline structure because of the continuous high strain-rate impacts.

Fig. 2(a) shows the nanohardness of the gradient NC-316L SS at the location of 2 μm, 4 μm, 8 μm, 12 μm, 16 μm and 20 μm depth from the topmost surface with their corresponding average grain sizes of 15 nm, 25 nm, 42 nm, 58 nm, 115 nm and 168 nm, respectively. The average grain size of the materials at different location was measured based on the FIB channeling contrast imaging and TEM characterization as illustrated in the Fig. 1. The nanohardness of the NC-316L SS was measured by using the Agilent Technologies Nanoindenter G200 with a standard Berkovich diamond indenter. Mechanical properties including nanohardness and elastic modulus were obtained from force-displacement curves by standard Oliver and Pharr method [18]. The maximum displacement of the indentations is 500 nm. The loading, holding, and unloading times were 20 s, 5 s, and 20 s, respectively. The maximum nanohardness of the NC-316L SS is 6.093 GPa located at the layer of around 2–3 μm depth from the topmost surface with the average grain size of 15–20 nm. The nanohardness of the NC-316L SS with the average grain sizes of 25 nm, 42 nm, 58 nm, 115 nm and 168 nm is 5.875 GPa, 5.77 GPa, 5.57 GPa, 5.44 GPa and 5.356 GPa, respectively. Eq. (1) reflects the relationship between the grain size and hardness of the materials with regard to the well-established Hall-Petch Eq. (4):

$$H_v = H_0 + \frac{K_h}{\sqrt{d}} \tag{1}$$

where, H_v is the hardness of a materials; H_0 and K_h are the materials constants; d is the average grain size of the materials. Also, the empirical relationship between the hardness and yield stress of the material can be seen in the Eq. (2):

$$H_v = 3\sigma_y \tag{2}$$

where the σ_y is the yield stress of the materials and could be expressed as follows:

$$\sigma_y = \sigma_0 + \frac{K_y}{\sqrt{d}} \tag{3}$$

where σ_0 is the friction stress and K_y is a yield constant.

Furthermore, to get the yield strength and flow behavior of the NC-316L SS, micropillar compression tests were performed. Micropillars with the diameter of ~3 μm and length of ~6 μm were fabricated by using the FIB in the FEI Nova 200 SEM/FIB Dual beam system. The

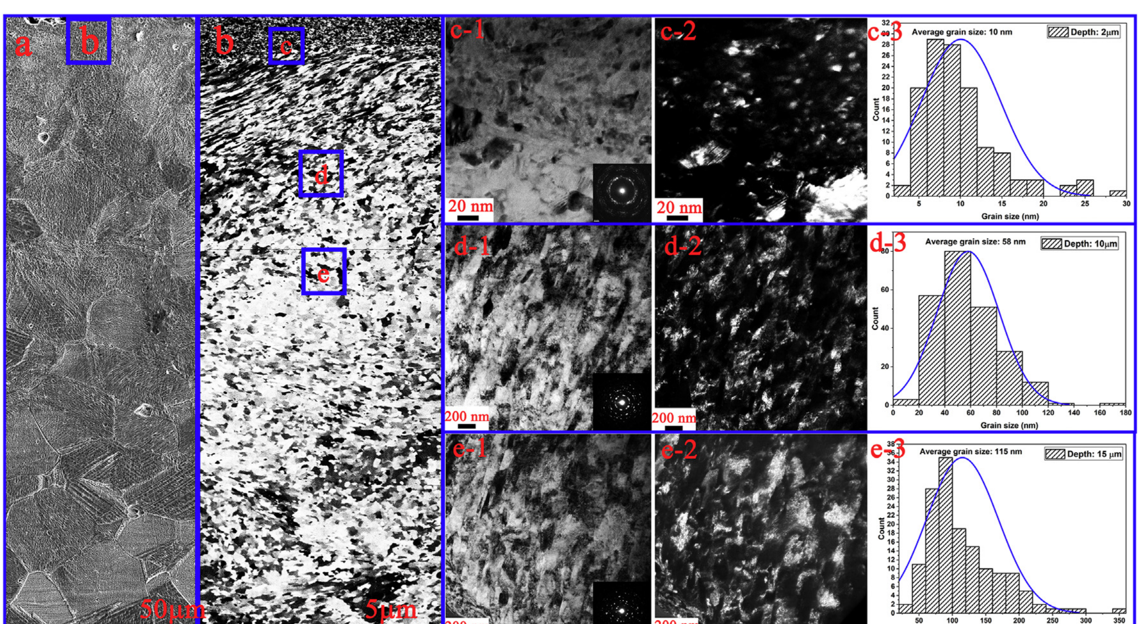

Fig. 1. Characterizations of the gradient nanocrystalline 316L stainless steel and the grain size distribution along the cross-sectional direction in the nanoscale.