

Strength, damage and fracture behaviors of high-nitrogen austenitic stainless steel processed by high-pressure torsion

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Received 21 July 2014; revised 4 September 2014; accepted 13 September 2014

Available online 4 November 2014

A high-nitrogen austenitic stainless steel (HNS) was processed by high-pressure torsion (HPT) for up to 5 turns to reveal the microstructural evolution and mechanical properties. The effects of the microstructure refinement and the increasing density of defects with imposed strain on the strength, damage and fracture behaviors of the HNS were investigated through the Ellipse criterion. The influences of these processes on the critical shear (τ_0) and normal (σ_0) fracture stresses determine the fracture mode.

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Keywords: Austenitic stainless steel; High-pressure torsion (HPT); Shear band; Strength; Fracture

For the last three decades, ultrafine-grained (UFG) materials produced by severe plastic deformation (SPD) have been of major interest [1,2]. Indeed, nanostructuring can improve the mechanical properties of metallic alloys due to grain size refinement [3]. Moreover, nanostructuring by SPD offers the possibility to produce non-porous bulk materials with good ductility [4]. Applications of SPD to multiphase microstructures have been attempted to explore the possibility of obtaining ultrafine composite nanostructures [5,6]. Among the numerous SPD techniques, high-pressure torsion (HPT) is especially attractive because it can produce materials with a relatively smaller grain size and higher strength due to the extremely high plastic strain applied [2]. To date, HPT has been used to process many different metallic materials; however, despite great commercial interest, very few studies have been conducted using steel.

In a different domain, an increase in grain boundary surfaces, which act as point defect sinks, can increase the number of deformation-induced porous defects [7,8]. Recent studies have revealed the formation of specific grain boundary types, so-called “non-equilibrium” or “high-energy” grain boundaries [9,10], which might be responsible for the unique properties of SPD-treated materials. In some cases of equal-channel angular pressing (ECAP)-deformed materials the formation of specific interconnected porosity channels in the UFG microstructure has been discovered [11]. Examples of such porosity channels were observed in

pure Cu [12] and Cu–0.3 at.% Pb deformed by ECAP at room temperature [13,14]. In a series of recent studies, Würschum and co-workers demonstrated that SPD processing did induce a significant amount of excess free volume in the form of vacancies, vacancy clusters, dislocation arrays and grain boundaries [15]. Nevertheless, the small grain size and saturated crystalline defects introduced during SPD improve the strength, though there is also an enhanced propensity for damage after the early stages of HPT deformation due to the significant increase in the number of potential microdefects. However, there are few in-depth studies on the influence of these microdefects on the fracture mechanism. Accordingly, the present investigation was initiated to provide information on the influence of the strengthening and damage process during SPD on the strength and fracture behaviors of SPD-treated materials.

The Fe–18Cr–16Mn–0.99 N samples used here have a mean grain size of 100 μm . Disks with a diameter of $d = 30$ mm and a thickness of $t = 2.5$ mm were processed by HPT using the parameters and procedures as described in Refs. [16,17]. The disks were processed through 5 turns under an imposed pressure of 6 GPa. The imposed strain ε is given by the relationship, $\varepsilon = 2\pi Nr/\sqrt{3}t$, where r and t are the radius and thickness of the disk, respectively, and N is the number of turns. The microstructures of the processed samples were characterized by scanning electron microscopy (SEM) undertaken at selected positions on transverse sections of the disks using a LEO Supra 35 microscope operated at 20 kV, and by transmission electron microscopy (TEM) using a FEI Tecnai F20 operated

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at 200 kV. Thin foils for TEM characterization, cut from the sectional areas at selected 10 mm distances from the center using spark cutting, were mechanically ground to ~ 50 μm thickness and then thinned by a twin-jet polishing method in an electrolyte consisting of 8% perchloric acid and 92% alcohol at -15 $^{\circ}\text{C}$. In addition, tensile tests were conducted at an initial strain rate of 10^{-3} s^{-1} using an Instron 5982 testing machine. Tensile dog-bone-shaped specimens with a gauge length of 8 mm, width of 2 mm and thickness of 1 mm were cut from the disks.

Tensile true stress–strain curves before and after HPT are shown in Figure 1a. The as-received disk exhibits high elongation but very low tensile strength, which is characteristic of coarse-grained materials. After 1/16, 1/4 and 1 turns, the yield strength increases significantly to 1500 MPa; however, the uniform elongation is extremely small, and the elongation to failure is also reduced drastically in contrast to the as-received sample, owing to a deterioration in the strain-hardening ability. When the disks are further strained to 5 turns, the tensile strength continues to increase, but the elongation becomes negligible, displaying a typical brittle fracture behavior. The tensile strength is as high as 2600 MPa after 5 turns, and this is comparable with the value in a lamellar-type nanostructured maraging steel subjected to ECAP for 12 passes [18]. According to the tensile stress–strain curves, the ultimate tensile strength (UTS) of the high-nitrogen austenitic stainless steel (HNS) steel specimens subjected to different turns of HPT is shown in Figure 1b. This demonstrates that with increase of the imposed strain of HPT, the strength slightly increases first and then basically remains unchanged, which is in line with the fact that strain hardening due to twinning is exhausted in the early stages of processing through an analysis of the evolution of strength with imposed strain as in Ref. [19].

Since the mechanical properties are always closely related to the microstructures of materials, the characteristic microstructures of HNS after HPT are observed as presented in Figure 2. In the present study no martensite could be detected when carefully analyzing the deformed microstructures by TEM and X-ray diffraction. The first stage of plastic deformation in the austenitic steel is characterized as dislocation glide and dislocation multiplication. When the shear stress is sufficiently high, the deformation processes involve not only dislocation glide but also include microshear banding and mechanical twinning [20]. Most twins are parallel to each other in the form of bundles (Fig. 2a). Profuse thin deformation twins with a thickness of ~ 10 – 30 nm formed in most grains. By further increasing the shear strain strong intersections of profuse twins were observed which increase the twin density to accommodate the plastic deformation. Meanwhile, the subsequent plastic deformation is now realized by the formation of nanometer- and/or submicron-sized shear bands. As shown in Figure 2b, many microscale shear bands ~ 50 nm thick traverse the whole grain, implying that localized plastic deformation begins to accommodate severe shear strain on the grain-size level. Many dislocations were introduced into the twin-matrix lamellae in order to accommodate the plastic deformation. These dislocations may develop into grain boundaries which are responsible for the dissolution of both the coarse twins and the lamellae into equiaxed grains. The accumulation and interaction of these defects seem to play a crucial role in the formation of shear localizations. Both the high density of deformation twins and the

intersections of profuse twins may be one of the initial sources governing the localized shear deformation [21]. After 1/4 turn of HPT, as demonstrated in Figure 2c, the density of shear bands was greatly increased and the width of these shear bands quickly grew with a transverse size of ~ 1.3 μm . Moreover, the intersection of shear bands can also be found in many regions. Meanwhile, nanograins with relatively random orientations were found within shear bands. The image shows that the microstructure had been transformed into small fragments forming nanocrystallites. The estimated volume fraction of shear bands is $\sim 60\%$, which indicates that shear bands become the increasingly dominant deformation mechanism during 1-turn HPT. However, the coarse features show that structural refinement is not fully developed. Figure 2d shows representative images of the etched surfaces of the samples processed through 5 turns of HPT. The absence of coarse features suggests that structure refinement consumed the original coarse grains, and nanoscale grains cover almost the entire area, with a final average size of ~ 20 nm. It is clearly evident that a very fine grain and almost homogeneous crystal structure has been developed in most grains. The dynamic recovery rate of dislocations prevents dislocation boundaries from accumulating on smaller scales [22]. These changes in the microstructure can restrict the effect of shearing instability on the failure of materials, resulting in the material having a higher strength [23]. The structure evolution and grain refinement mechanisms are similar to those of several intensively studied 316L austenitic steels [24,25]. The different twin thicknesses and the microstructural sizes in the saturation region may mainly originate from two factors, i.e. the alloying concept and the deformation conditions applied.

Unfortunately, the deformation processing of the HNS steel by HPT may introduce additional free volume in the material in the form of non-equilibrium interfaces, vacancy clusters, nanovoids and micropores. Non-equilibrium vacancies are inevitably formed during SPD and may agglomerate near different internal sinks (presumably grain boundaries, triple lines or nanocracks formed by disclination or superdislocation activity). In this way, internal porosity may be produced even under the conditions of a high hydrostatic pressure. As illustrated in Figure 3e and f, pore- and crack-like defects were observed in the HNS steel subjected to the above 1-turn HPT process. Meanwhile, the volume fraction of these defects are increased with imposed strain. Considering microvoid accumulation, the accumulation of various defects is beneficial to the coalescence and aggregation growth of the cracks, which will adversely affect the mechanical properties of materials.

The refinement of the microstructure and the different degrees of damage of the HPT-processed steel described above should have different effects on the mechanical properties of the UFG or nanocrystalline (NC) materials. On the other hand, the different influences of the accumulation of various defects can be seen in their macroscopic tensile fracture behavior. After tension, the HNS specimens exhibited differences in shear fracture behaviors (Fig. 3a). The specimens processed through 1/16, 1/4, 1 and 5 turns ruptured by shearing at different shear angles of 48° , 50° , 60° and 72° , respectively, as revealed by further inspection. In recent reports on a Cu–Ag alloy processed by HPT and three Cu–Zn alloys processed by ECAP [23,26], the specimens macroscopically also failed through shear fracture at angles $>45^{\circ}$. All these phenomena exhibit similarities

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