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Interplay between grain structure, deformation mechanisms and austenite stability in phase-reversion-induced nanograined/ultrafine-grained austenitic ferrous alloy

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Abstract—The concept of phase reversion involving severe cold deformation of austenite at room temperature to generate strain-induced martensite, followed by annealing when martensite reverts to austenite via a diffusional mechanism, was used to obtain a "high strength, high ductility" combination in nanograined (NG) austenitic stainless steel. Using this concept, the objective of the study is to elucidate the dependence of grain size on deformation mechanisms and deformation-induced microstructural changes. The objective was accomplished by combining depth-sensing nanoindentation experiments conducted at strain rates spanning two orders of magnitude and post-mortem analyses of deformed Fe–17Cr–7Ni austenite alloy using transmission electron microscopy. The strain-rate sensitivity of the NG structure (0.13) was about twice the coarse-grained (CG) counterpart (0.06) and the activation volume was about one-third ($16b^3$) of the CG structure ($48b^3$), where b is the magnitude of the Burgers vector. In the high strength NG steel, deformation twinning contributed to excellent ductility, while in the low strength CG steel, ductility was also good, but due to strain-induced martensite, implying clear distinction and fundamental transition in the deformation behavior of NG and CG Fe–17Cr–7Ni austenitic stainless steels. In the NG structure, there was marked increase in stacking faults and twin density at high strain rates, which led to a decrease in the average spacing between adjacent stacking faults, converting stacking faults into twins. The plastic zone in the NG structure resembled a network knitted by the intersecting twins and stacking faults. The observed change in the deformation mechanism with change in grain size is attributed to increased stability of austenite with a decrease in grain size, and is explained in terms of the austenite stability–strain energy relationship.

Keywords: Nanograined structure; Deformation mechanism; Austenitic stainless steel; Electron microscopy; Twinning

1. Introduction

There is a continued and significant interest to pioneer advanced high strength steels, including stainless steels, characterized by nano/ultrafine grains with a high strength, high ductility combination for lightweight construction. This interest has led to the development of newer processing routes. Grain refinement is considered as a practical approach to improve the strength of engineering metals and alloys [1,2]. Thermomechanical controlled processing (TMCP) is one of the primary methods of grain refinement. For example, in body-centered cubic (bcc) ferrous alloys, TMCP in conjunction with appropriate microalloying elements results in a ferrite grain size less than 5 μm [3–6]. The grain refinement limits imposed by conventional TMCP can be overcome by the application of severe plastic deformation, leading to submicron or ultrafine grain (UFG) structures in metallic materials [7,8].

In the context of obtaining nanograined (NG) structure, we have recently obtained a high strength, high ductility combination in NG austenite stainless steels using the innovative concept of a phase-reversion-induced nano/submicron-grained structure [9–12]. The controlled deformation-annealing approach to obtain NG material with a high strength, high ductility combination involved cold deformation (~60–80%) of metastable face-centered cubic (fcc) austenite (γ) to strain-induced body-centered cubic (bcc) martensite (α'). Upon annealing at 700–800 °C for a short duration of ~10–100 s, martensite transforms back to austenite via a diffusional reversion or martensitic shear mechanism, depending on the chemical composition of the steel, without affecting the texture [9–12]. The success of this approach in obtaining NG structure depends on the predominance of dislocation cell-type structure in the severely deformed austenite [10]. The concept enables us to explore deformation mechanisms in a single material from the NG

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to the coarse-grained (CG) regime, depending on the processing parameters (cold deformation and annealing temperature–time sequence). It is also capable of producing a large volume of defect-free material.

The understanding of deformation behavior of NG alloys continues to be fragmented [13–15]. Deformation mechanisms in NG/UFG metals can be dramatically different from those operating in CG metals. The higher strength of NG metals in relation to the CG counterpart has led to the suggestion that the dominant mode of plastic deformation of ductile CG materials mediated by the grouped activity of dislocations inside the grains (e.g., the formation of dislocation pile-ups and cells) is suppressed in NG materials, where partial dislocation emission from grain boundaries may operate [14,16]. Thus it is widely believed that the decrease in grain size and the consequent increase in yield strength must be accompanied by a change in the deformation mechanism.

The high ductility of conventional CG austenitic stainless steels is associated with the gradual transformation of austenite to martensite, which increases the strain-hardening rate and delays the onset of localized necking [17–19]. However, in striking contrast to the behavior of CG austenite, the present study underscores that the grain size of austenite affects its mechanical stability such that the NG austenite is resistant to transformation to martensite. In this regard, we utilize the concept of phase reversion to fundamentally explore the deformation mechanism in a high strength, high ductility combination NG material as a function of strain rate and compare with that of the CG counterpart. To accomplish the aforementioned objective, we have conducted nanoscale deformation experiments using a nanoindenter for the following reasons [20–25]. In nanoindentation, when the indenter tip (20 nm radius in our case) is too small to produce a highly stressed volume beneath the indenter, the fundamental processes underlying discrete deformation in a small volume of the material is envisaged to have a low probability of encountering pre-existing dislocation prior to the commencement of plastic deformation. Furthermore, the tested volume is scalable with respect to the microstructure [20,21]. The distinct transition in deformation mechanism from strain-induced transformation to martensite in CG austenitic stainless steel to nanoscale twinning in NG is explained in terms of the austenite stability–strain energy relationship.

2. Experimental: materials and methods

The starting material was Type 301LN austenitic stainless steel of ~1.5 mm thickness and having nominal composition (in wt.%) of Fe–0.017C–0.52Si–1.3Mn–17.3Cr–6.5Ni–0.15Mo–0.15N. The strips were cold-rolled in a laboratory rolling mill to ~62% thickness reduction and subsequently annealed at 800 °C for 10 s in a Gleeble 1500 thermomechanical simulator to obtain reverted NG austenite structure. The annealing experiments were carried out on strips of 120 × 25 mm (thickness ~0.6 mm). The phase reversion process is described in detail elsewhere [9–12]. Room temperature tensile properties were determined using specimens that were machined to a profile of 25 × 25 mm with a 20 mm gage length. Tensile tests were conducted at a strain rate of 3×10^{-3} s^{-1}.

We adopted two methods to measure the grain size from the point of view of grain size distribution – average grain size and weighted average grain size. In the first approach, the average austenite grain size was determined by analyzing at least 100 grains in the micrographs to determine the mean linear intercept grain size, $\bar{d}$. In the second method, with focus on grain size distribution, weighted average grain size, $\bar{d}_w$ was measured. Here, ~100 grains were distributed in bins 250 nm (0.25 μm) in size. A bin of 250 nm was selected to optimize the statistical data. A small bin size results in poor statistical accuracy, while a large bin size masks the effect of small grains. Based on the range of grain size present, an optimum bin size of 250 nm (0.25 μm) was selected. The bins were defined as $B = (b_1, b_2, \ldots, b_N)$, where $b_1 = 0$–250 nm (0.25 μm), $b_2 = 0.25$–0.50 μm, …, $b_N = 24.75$–25.00 μm. Subsequently, the number of grains belonging to each of the ith bin was counted from a sample of 100 grains. Denoting the number of grains in the ith bin as n_i and dividing it by the total number of grains, N, the weight of the ith bin is given by [26]:

$$w_i = \frac{n_i}{N} \tag{1}$$

Moreover, the square root of the areal mean of n_i grains in the ith bin gives the average grain size, $\bar{d}_i$, for the ith bin. Knowing $\bar{d}_i$ and w_i, the weighted average grain size of the sample is calculated by [26]:

$$\bar{d}_w = \sum_{i-1}^{N} w_i \bar{d}_i \tag{2}$$

Given that the nanoindents were to be subsequently examined by transmission electron microscopy (TEM) for studying deformation mechanisms and associated microstructural evolution, the following procedure was adopted. First, 3 mm disks were punched from the experimental steels. To ensure that the nanoindents were distributed along the thin area of the disk for examination via TEM, a modification of twin-jet electropolishing and a new design of sample mounting for nanoindentation were developed. The disks were partially jet-electropolished in a refrigerated electrolyte of 10% perchloric acid in acetic acid at 25 V for ~30 s to obtain a shining surface in the center part of the 3 mm disk. The procedure of placing horizontally partially electropolished disks of ~30 μm thickness inside the ~4 mm diameter pits drilled on the polished basal surface of an aluminum block of diameter ~25 mm was as follows. In brief, the ~4 mm diameter pits were partially filled with the solder. Next, the surface was polished prior to placing the partially polished disk on top of the solder. This was followed by placing a transparent tape around the disk (foil specimen) to prevent its movement during nanoindentation experiments. This carefully designed approach fully supported the specimen to withstand the applied force of the nanoindenter and also prevented the specimen from bending during nanoindentation experiments.

Nanoindentation experiments were carried out in displacement-controlled mode at strain rates in the range of 0.05–1 s^{-1}. The maximum displacement was set to 200 nm. The nanoindenter system (MTS XP) consisted of a Berkovich three-sided pyramidal diamond indenter with a nominal angle of 65.3° and indenter tip radius of 20 nm. An array of indents of matrix 12 × 12 was defined with the indent gap of 10 μm. After the indentation experiments, the disks were removed from the mount and final electropolishing was carried out only from the side opposite to the indented surface. Using this procedure, the area surrounding the indents, which is present along the final jet-polished hole, was electron-transparent to study the deformation