Materials Science & Engineering A 639 (2015) 656–662

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

Strengthening mechanisms in nanostructured interstitial free steel deformed to high strain

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ARTICLE INFO

Article history:
Received 20 February 2015
Received in revised form
14 May 2015
Accepted 23 May 2015
Available online 2 June 2015

Keywords:
Nanostructured materials
Strengthening mechanisms
Accumulative roll bonding

ABSTRACT

In this study, the strengthening mechanisms in nanostructured IF steel deformed to high strain by four-layer accumulative roll bonding (ARB) process at room temperature in absence and presence of SiC particles were investigated. Microstructural observations were performed by scanning electron microscopy (SEM) and scanning transmission electron microscopy (STEM). The results indicated that the average grain size of the pure steel, composite, and nanocomposite was 95, 73, and 55 nm, respectively and the microstructures consisted of equiaxed grains. Also, with increasing the number of ARB cycles, the dislocation density of samples increased. The first cycle of ARB process had remarkable effect on the dislocation density. The value of dislocation density first rapidly increased, then dwindled, and finally saturated by further ARB cycles. The presence of SiC microparticles and nanoparticles in the IF steel matrix increased the dislocation density during the ARB process. On the other hand, dislocation density of the nanocomposite was higher than that of the composite. After first cycle, a significant increase observed in the yield strength, from 84 MPa to 609, 682, and 689 MPa for pure steel, composite, and nanocomposite, respectively, which was almost 7.3, 8.1, and 8.2 times greater than that of the initial sample. There was no perfect saturation in yield strength of the pure IF steel with increasing the number of ARB cycles. Finally, the contribution of individual mechanisms such as the grain refinement, dislocation, second phase, and precipitation in strengthening of the IF steel was evaluated. The contribution of grain refinement and precipitation to the improvement in yield strength was maximum (~67–72%) and minimum (~3.1–3.7%), respectively.

1. Introduction

Strong materials are a classical goal for materials research and development. Today there is focus on nanostructured metals since they are found to have a very high strength as well as other excellent mechanical properties [1–5]. According to the well-known Hall Petch equation, the strength increases with a reduction in the grain size. Therefore, fabricating materials with a grain size in the nanorange (less than 100 nm) or ultrafine range (between 100 and 1000 nm) is an effective approach to increase the strength of materials [1–7]. Nanostructured metals can be processed by a number of different techniques and one promising method is to apply plastic deformation to very high strains [1–7]. The processing of metals through the application of severe plastic deformation (SPD) provides the potential for achieving exceptional grain refinement in bulk metal solids [1–7].

There are techniques for producing nanostructure and ultrafine grain (UFG) materials such as equal-channel angular pressing (ECAP), high-pressure torsion (HPT), and accumulative roll bonding (ARB) [1–7]. The ARB process is one of the SPD that was proposed by Saito et al. [8,9]. The ARB consists of multiple cycles of stacking, rolling and cutting in which large strains are imposed into the material without any change in the cross section [8,9]. During ARB, the sample is assumed to be deformed in a plain strain condition. Therefore, the equivalent strain ε_{eq}, can be calculated using the following equation [4]:

$$\varepsilon_{eq} = \frac{2}{\sqrt{3}} n \ln \frac{h_0}{h} = \frac{2}{\sqrt{3}} n \ln \frac{1}{1-r} \quad (1)$$

where h_0 is the initial thickness of the stacked sheets, h is the thickness after roll bonding, r is the reduction in thickness per cycle and n is the number of ARB cycles. The reduction per ARB

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http://dx.doi.org/10.1016/j.msea.2015.05.075
0921-5093/

cycle is usually $r=50\%$, which results in an equivalent strain of about 0.8 per cycle.

The evolution of microstructures and the related mechanical properties during ARB process were studied for several metals such as commercial pure Al [4,8–12], Cu [13–15], Brass [16], Ti [17], Mg [18,19], and steel [20–23]. The ARB process is also applicable in the fabrication of multilayered composites [24–26] and metal matrix composites (MMCs) [27–33].

The density of dislocations is always difficult to determine experimentally. Traditionally there are some methods such as X-ray diffraction (XRD) [34–37], transmission electron microscopy (TEM) [38,39], electron backscatter diffraction (EBSD) [40,41], electron channeling contrast imaging [42], and hydrogen diffusivities [43]. Recently, the dislocation density has been estimated from hardness measurement [44,45]. Graca et al. [44] reported that it is possible to estimate the dislocation densities by the hardness indentation size using the Nix–Gao model [45].

In present study, the four-layer ARB process has been utilized at room temperature in presence of reinforcement particles to refine the grain size of interstitial free (IF) steel matrix. For a comparison, unreinforced pure IF steel was processed and characterized mechanically and microstructurally using the same methods. To quantify the microstructure, scanning transmission electron microscopy (STEM) is applied for fine-scale structures and scanning electron microscopy (SEM) is used for coarser-scale structures. Mechanical properties are determined by tensile testing at room temperature and are related to the structural parameters through the strength–structure relationship, which is discussed based on the operation of different strengthening mechanisms.

2. Experimental procedure

The materials used in this study were fully annealed sheets of interstitial free steel (specifications are given in Table 1) and SiC microparticles (50 μm) and nanoparticles (50 nm) (Fig. 1). Four sheets of 150 mm × 50 mm × 0.7 mm were degreased via acetone and scratch brushed with a stainless steel wire brush 0.25 mm in diameter. For fabrication of steel-based composite (containing microparticles) and nanocomposite (containing nanoparticles), after the surface preparation, SiC micro/nano-particles were uniformly dispersed between the four sheets. To achieve a uniform dispersion of SiC particles between IF steel sheets, an acetone-based suspension was prepared. After surface preparation, the SiC particles in acetone were sprayed between the four sheets with an atomizer. Then, SiC particles were deposited and the acetone evaporated in air, so that the brushed surfaces of sheets were uniformly covered with SiC particles. The sheets were then stacked over each other and fastened at both ends by steel wires. Attention was also paid to proper alignment of the four sheets surfaces prior to rolling. The roll bonding process was carried out with no lubrication and with an amount of thickness reduction equal to 75% corresponding to a von Mises equivalent strain e_{vM} of 1.6 per cycle (first step). Then, the roll bonded sheets were cut into four pieces. Then, to achieve a uniform distribution of SiC particles in the IF steel matrix, the above procedure was repeated again up to fourth cycle without adding particles (second step). The schematic illustration of the ARB process for fabrication of steel-based composite and nanocomposite samples is shown in Fig. 2.

Table 1
Chemical composition of IF steel (wt%).

C	N	Si	Mn	Cu	Ni	Ti	Fe
0.002	0.004	0.01	0.14	0.01	0.018	0.055	Bal.

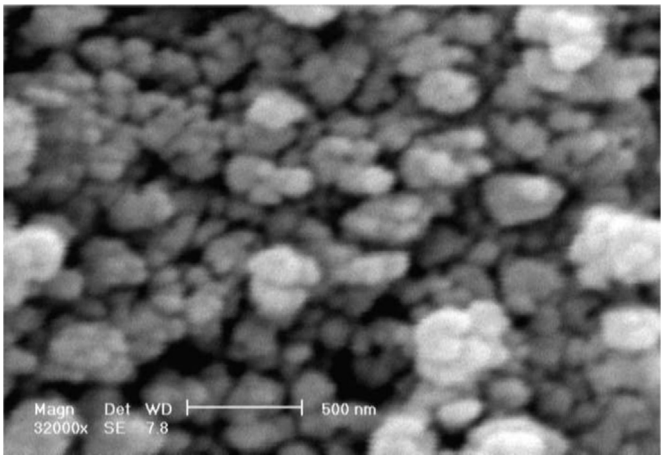

Fig. 1. SEM micrograph of the SiC nanoparticles.

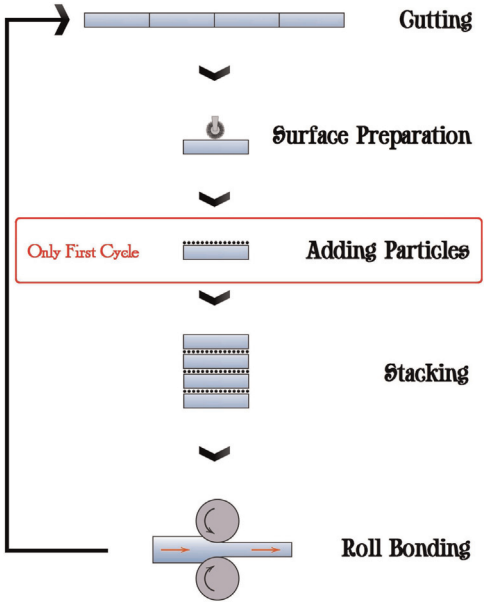

Fig. 2. Schematic illustration of ARB process.

The samples for scanning electron microscopy (SEM) observations were cut from the sheets and this was mounted in bakelite. Then, the samples were polished using 80–4000 grit water-proof SiC paper. Finally, the polishing was finished on a cloth using alumina paste of 3 μm. Scanning electron microscopy PHILIPS XL30 was used.

The microstructural observations were performed using scanning transmission electron microscopy (STEM). Thin foils were prepared with electropolishing conducted at −30 °C using 60 V in a 5% perchloric acid/95% methanol solution. Multiple disc samples with 3 mm diameter were separated from thin foils. Then, the discs were prepared using a low angle Ion Milling System from Fischione Model 1010 with 5 kV operating voltage, 5 mA current, 2.5 h time duration, and angle of 10° conducted at −40 °C. A Hitachi S-4800 field emission scanning electron microscope was used.

Vickers microhardness was measured according to the ASTM: E384-11e1 standard. The surfaces used for indentation testing were ground with SiC papers and polished with a sequence of alumina particles suspensions. Vickers indentation tests were performed using loads in the range 0.01– 2 N.

The tensile test samples were machined from the ARB-processed sheets, according to the ASTM: E8M tensile sample,