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## Formability of ultrafine-grained interstitial-free steel fabricated by accumulative roll-bonding and subsequent annealing

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The miniaturized Erichsen test was successfully developed for evaluating the stretch formability of the ARB and annealed IF steel sheets. The Erichsen value tends to decrease as the average grain size becomes small. However, it should be emphasized that even in the specimen with ultrafine grain, a high Erichsen value is obtained. This result indicates that the ultrafine grained IF steel sheet could be highly deformed under multiaxial stress.

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Severe plastic deformation (SPD) processes, which involve subjecting bulky metallic materials to large equivalent strains of over 4.0, have been developed in order to produce ultrafine-grained (UFG) structures [1–9]. These processes make it possible to fabricate UFG metals composed of grains with an average grain size much smaller than 1  $\mu$ m. UFG metals show superior mechanical properties—for example, their strength is 2–4 times higher than that of conventional metals with average grain sizes of over 10  $\mu$ m [1,2].

It is very important for practical applications to know the formability as well as the ductility of this new class of materials. It is known that UFG materials with single-phase structure show limited tensile ductility, especially uniform elongation, due to the early plastic instability that occurs in UFG structures [10]. On the other hand, the formability, e.g. bendability, deep drawability and stretch formability, of UFG materials has so far rarely been studied [11]. Miyata et al. [11] reported, based on a Hemming's test, that UFG low-C steel possesses a degree of local ductility, which plays an important role in formability, e.g. bending.

In the present study, the formability under multiaxial stress conditions of an UFG material was studied, using Ti-added interstitial-free (IF) steel fabricated by accumulative roll-bonding (ARB), which is a SPD process that can fabricate sheet materials with UFG structures [12].

The material used in this study is an ultra-low-carbon IF steel sheet (C 0.0020, Si 0.01, Mn 0.10, P 0.005, S 0.006, s-Al 0.032, Ti 0.039 mass%, N 27 ppm), coldrolled to 2 mm in thickness and then fully annealed. The ARB was conducted for seven cycles (an equivalent strain of 5.6) with lubrication under the conditions same as those reported previously [13]. The final dimensions of the ARB processed sheet were 40 mm in width, about 200 mm in length and 1 mm in thickness. The ARB processed sheets were then annealed at various temperatures for various periods as described below, in order to obtain specimens with various average grain sizes: 500 °C for 0.5 h, 600 °C for 0.5 h, 600 °C for 20 h and 850 °C for 20 h.

The cross-section perpendicular to the transverse direction (TD) of the ARB processed sheet was observed by electron backscatter diffraction (EBSD) measurements using an Orientation Imaging Microscopy

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(OIM<sup>TM</sup>) system made by EDAX/TSL, which was attached to an FEI XL30 Sirion field emission scanning electron microscope (FE-SEM,) operated at 15 kV. The specimens were prepared by electropolishing using a solution of 10% perchlorate acid +90% acetic acid to obtain a smooth and flat surface. The measured areas were at the thickness center of the ARB processed sheet, and each scan area was selected to include a sufficient number of grains.

Uniaxial tensile deformations were performed on the starting sheet, the ARB processed sheet and the ARB processed and annealed sheets. The tensile specimens 5 mm in gage width and 10 mm in gage length were machined from the sheets so that the loading direction was parallel to the rolling direction (RD) of the sheets. This size of specimen corresponds to one-fifth of the size of JIS (Japanese Industrial Standards) No. 5 type tensile specimen. The tensile deformations were carried out at an initial strain rate of  $8.3 \times 10^{-4} \text{ s}^{-1}$  at room temperature using a Shimadzu Autograph (AG-100kNI). The nominal stress vs. nominal strain curves were calculated from the load–displacement relationship.

Miniaturized Erichsen tests were carried out in order to evaluate the stretch formability of the ARB and annealed sheets. Three pieces of square specimens,  $20 \text{ mm} \times 20 \text{ mm} \times 1 \text{ mm}$  in size, were cut from each sheet, one side of which was parallel to the RD of the sheet. The miniature Erichsen test device was designed so that its size was one-third that of the device used for the standard Erichsen test [14]. The device was attached to the Shimadzu Autograph. The Erichsen value was evaluated as the point where the load discontinuously dropped in the load–stroke diagram. The stroke was calculated by measuring the movement of the using a CCD camera.

The cross-section perpendicular to the TD of the specimens was measured by EBSD for microstructural observation. Grain boundary maps obtained from the EBSD measurements for the starting material, the specimen ARB processed by seven cycles with lubrication and the specimens ARB processed and then annealed at various temperatures for 0.5 or 20 h, are shown in Figure 1. In this figure, the green and red lines represent high-angle grain boundaries (HAGBs) having misorientations larger than 15° and low-angle grain boundaries (LAGBs) having misorientations of 2-15°, respectively. The average interval of the HAGBs in the normal direction (ND) of the sheets was defined as the average grain size  $(d_t^{HAGB})$  in each specimen. The average grain sizes of the starting material, the specimens ARB processed, and the specimens ARB processed and then annealed at 500 °C for 0.5 h, 600 °C for 0.5 h, 600 °C for 20 h and 850 °C for 20 h were 16.0, 0.24, 0.44, 1.15, 7.20 and 13.5 µm, respectively.

The starting material has a fully recrystallized microstructure slightly elongated in the RD ( $d_t^{HAGB} =$ 16.0 µm). The ARB processed sheet has a lamellar structure elongated in the RD—typical of microstructures obtained by ARB. The average thickness of the grains is 0.24 µm, while the length parallel to the RD is over 10 µm in some grains. The microstructures of the sheets ARB processed and annealed at 500 or 600 °C are mostly composed of elongated grains, similar to the



Figure 1. Grain boundary maps obtained from the EBSD measurements for the starting material, the specimen ARB processed for seven cycles with lubrication, and the specimens ARB processed and then annealed at various temperatures for 0.5 or 20 h: (a) starting material; (b) ARB processed for seven cycles; ARB processed for seven cycles and then annealed at (c) 500 °C for 0.5 h, (d) 600 °C for 0.5 h, (e) 600 °C for 20 h and (f) 850 °C for 20 h.

as-ARB processed sheet. However, grain growth is observed, so that the grain size increases with increasing annealing temperature. On the other hand, the microstructures of the sheets ARB processed and annealed at 600 or 850 °C show nearly equiaxed grains and the grains coarsen with increasing annealing temperature.

The nominal stress vs. nominal strain curves of the specimens studied are shown in Figure 2. Tensile strength, yield strength (0.2% proof stress), uniform elongation and total elongation of the starting material  $(d_t^{\text{HAGB}} = 16.0 \,\mu\text{m})$  are 276 MPa, 151 MPa, 27% and 58%, respectively. The ARB processed sheet ( $d_t^{\text{HAGB}}$  $= 0.24 \ \mu m$ ) has a tensile strength of 783 MPa, which is 2.8 times higher than that of the starting material, and the total elongation is only 3%. The deformation stress increases rapidly at the beginning of deformation, and macroscopic necking starts immediately after reaching maximum stress, leading to rupture. These are typical stress-strain behaviors of ARB processed materials [10,15,16]. The limited uniform elongation is understood in terms of the early plastic instability that occurs in single-phase UFG structures [10]. For the sheet ARB processed and annealed at 500 °C for 0.5 h ( $d_t^{HAGB}$  $= 0.44 \,\mu\text{m}$ ), the flow stress also increases rapidly, but the maximum strength is lower than the as-ARB sheet  $(d_t^{\text{HAGB}} = 0.24 \,\mu\text{m})$ . In the case of the sheet ARB processed and annealed at 600 °C for 0.5 h ( $d_t^{\text{HAGB}}$  $= 1.15 \,\mu\text{m}$ ), the tensile strength decreases to 408 MPa,

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