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Improvement of formability of ultrafine-grained materials by post-SPD annealing

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ABSTRACT

Ultrafine-grained (UFG) IF-steel, as an example of an essentially single phase UFG microstructure, was annealed at different temperatures and time intervals in order to improve its formability by achieving a good strength–ductility–formability balance. In general, annealing increased the ductility and formability of UFG steel. Annealing at temperatures inside the recovery region caused a limited improvement in the formability of UFG steel due to the relief of internal energy without considerable grain coarsening. As the grain size increased to above 4 μm by annealing at temperatures inside the partially recrystallization region, the formability of UFG steel in the uniform region increased considerably, and localized deformation with early necking changed to a homogeneous mode as revealed by increased uniform thinning and enlargement of the membrane straining regime. Further grain coarsening resulted in a slight increase in uniform elongation both in uniaxial and biaxial tests. The UFG microstructure reduced the roughness of the free surface of biaxially stretched samples by decreasing the non-uniform grain flow, which leads to the so-called orange peel effect. Annealing of UFG microstructure did not degrade this positive effect due to the formation of sharp recrystallization textures although the annealed microstructures have relatively coarse grains. It can be concluded that a good balance between strength and uniform formability without an orange-peel effect can be achieved in UFG microstructures by well-design annealing processes.

1. Introduction

Ultrafine-grained (UFG) materials produced by severe plastic deformation (SPD) methods have received considerable attention due to their substantially increased strength as compared to their coarse grained (CG) counterparts [1–5]. However, their small uniform elongation under uni-axial loading, which is not exceeding a few percentage, is considered as the major drawback of these materials [4,6,7]. The UFG materials feature a sub-micron grain sized microstructure containing a high amount of strain-induced boundaries (SIB) formed by accumulation and rearrangement of dislocations during severe deformation [7–9]. This in turn, however, also results in limited strain hardening capability, and thus, small uniform elongation. Specifically, dislocation trapping effects at the SIBs reduce dislocation interaction during plastic deformation, and consequently decrease the strain hardening capacity by causing early deformation localization [10]. In order to improve the ductility without a significant decrease in strength of the UFG microstructures, post-SPD annealing is considered to be the most promising approach to obtain a good combination of strength and ductility [11–18]. By controlling the recovery and/or recrystallization fraction and the recrystallized grain size, a bi-modal grain size distribution can result [9,12–21].

UFG or nano-structured (NS) materials are also considered as promising candidates for micro/macro-forming applications for producing small parts to be used in electronics/micro electromechanical systems and the medical sector [22,23]. Hence, it is important to characterize and enhance the formability of UFG materials under multi-axial loading conditions. So far, only few studies have addressed the formability of UFG materials under multi-axial loading condition. Previous studies showed that UFG Al alloys produced by accumulative roll bonding (ARB) feature lower formability compared to the CG counterparts in deep drawing tests [24,25]. Similar results were also observed for UFG Cu produced by equal-channel angular extrusion/pressing (ECAE/P) [26]. The formability of UFG IF-steel produced by ECAE via various strain paths was investigated by Saray et al. [4]. This study showed that grain refinement by ECAE decreased the formability and increased the

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required punch load of UFG steel regardless of the applied strain paths. Moreover, the stretch formability was affected considerably by the grain morphology of the UFG microstructure. Specifically, an UFG microstructure with heavily elongated grain morphology resulted in low formability, whereas UFG microstructures with equiaxed grains resulted in significantly better formability [4]. Still, the forming capability of the equiaxed UFG microstructure is fairly low because of the limited strain hardening capability leading to a small uniform elongation [23].

For an appropriate formability, a reasonable combination of high strength and good ductility for UFG materials is needed [4]. Some earlier reports have shown that the ductility of UFG materials can be enhanced without considerable strength loss by appropriate annealing treatments [4,9,10,12–18]. The formability of UFG IF-steel was investigated by Yoda et al. [24] after ARB, and they reported that both elongation to failure and the Erichsen value increased with increasing recrystallized grain size [24]. In the present study, this approach was extended to obtain a better formability of UFG microstructure under biaxial stretching. For this purpose, a Ti-stabilized IF-steel as a model material was chosen, and different microstructures were produced by ECAE and post-ECAE annealing. Also, the effect of annealing on the deformation behavior of the UFG materials under biaxial loading conditions was investigated.

2. Experimental procedures

The material in this study was Ti-stabilized IF-steel with a chemical composition of 0.004 wt% C, 0.012 wt% Si, 0.2 wt% Mn, 0.012 wt% P, 0.009 wt% S, 0.1 wt% Ti and balance Fe. The received material was hot-rolled to a thickness of 25 mm after casting. A multi-pass ECAE was applied to the steel billets with dimensions of 25 mm × 25 mm × 150 mm in order to obtain a UFG microstructure with sub-micron grain size. The billets were processed using an ECAE die system with a sharp 90° channel cross-section angle up to 8 passes following route-E where the billets were rotated by +180° and +90° between successive passes. More information about ECAE processing can be found in [3]. For post-ECAE annealing, three routes were employed: The first set of the UFG steel samples was annealed at 500 °C for 60 min in the recovery region to obtain a recovered but not recrystallized microstructure. The second set was annealed at 600 °C for various time periods from 12 min to 60 min in order to obtain partially recrystallized (bi-modal) microstructures. Finally, some UFG samples were annealed at 650 °C for 60 min in order to result in a fully recrystallized microstructure. These annealing conditions were determined based on our earlier research on the recrystallization behavior of the UFG IF-steel [9].

The microstructures of the differently annealed UFG samples were investigated using electron backscattering diffraction (EBSD) technique in a scanning electron microscope (SEM) operated at a nominal voltage of 20 kV. The mechanical properties under uniaxial loading were evaluated with tension tests using dog-bone-shaped samples with gauge section dimensions of 8 mm × 3 mm × 1.5 mm. These tensile samples were cut from the ECAE-processed billets with their tensile axis oriented parallel to the extrusion direction and tested at room temperature with a strain rate of $5 \times 10^{-4}\,s^{-1}$. The formability of the IF-steel with CG, UFG and annealed UFG microstructures was evaluated using a miniaturized Erichsen test system having a die with half dimensions as given in the ISO 20482 standard. The formability samples with dimensions of 25 mm × 25 mm × 0.9 mm were sectioned from the transverse plane of the extruded billets using a wire-EDM. Prior to the formability tests, the surface of the samples were mechanically ground using emery paper down to grid size of 1000 and then polished with a 1 μm alumina solution. The tests were performed with a punch speed of $0.01\,mm\,s^{-1}$ without lubrication. The Erichsen index (Ei) and the load (F_{Ei}) corresponding to this index were evaluated from the load–displacement curves obtained during the Erichsen tests. The tested samples were investigated with a JEOL JSM 6400 SEM operated in the secondary electron mode at 15 keV in order to reveal the surface features and deformation characteristics. Cross-sections of the stretched samples were cut, and dome thickness measurements were carried out using an optical microscope. Textures of the different microstructural conditions were determined using a Bruker AXS D8 Discover Diffractometer with Cu-K_α radiation at 40 kV. The popLA (preferred orientation package – Los Alamos) [27] texture analysis software package was used to visualize pole figures of annealed UFG samples in selected conditions.

3. Results and discussion

3.1. Microstructure

As expected, 8E ECAE processing significantly refined the microstructure and transformed the CG microstructure (average grain size: 30 μm [3,4]) into an UFG one as shown in Fig. 1(a). The grain size histograms show that the mean grain size of UFG microstructure after ECAE is about 0.90 μm, and the grains have a size in the range from 0.30 μm to 3.40 μm throughout the microstructure (Figs. 1(a) and 2(a)). The UFG grains are mostly separated by high-angle grain boundaries (HAGBs) with a fraction of about 54% when considering 15° as of the lower limit of HAGBs (Fig. 2(a)). Annealing of UFG steel at 500 °C for 60 min slightly increased the mean grain size to about 1.0 μm (Figs. 1(b) and 2(b)). Also, the grain size distribution and grain boundary type of the UFG microstructure did not change considerably after this annealing (Fig. 2(b)), i.e. only recovery took place. Recovery of the UFG microstructure slightly increased the fraction of HAGBs from 54% to about 55% (Fig. 2(b)). Annealing of UFG steel at 600 °C for various time intervals led to progressive recrystallization of the UFG microstructure (Figs. 1(c)–(e), 2(c) and 3(e)). Annealing at 600 °C for 12 min resulted in a partially recrystallized microstructure with a mean grain size of 4.2 μm (Fig. 2(c)). Some recrystallized grains of this microstructure are in the 4–10 μm range, while some grains with sizes smaller than 1 μm are still evident in the microstructure with a fraction of about 15% (Fig. 3(c)). Thus, this annealing brought about a bimodal grain size distribution (Figs. 1(c) and 2(c)), and the area fraction of the HAGBs in this microstructure increased up to 62% (Fig. 2(c)). Increasing the annealing time to 30 min at the same temperature almost fully eliminated the UFG microstructure and resulted in new grains within a size in the range from 3 μm to 15 μm (Figs. 1(d) and 2(d)). Mean grain size and fraction of the HAGBs of this microstructure were determined to be 6.3 μm and 65%, respectively (Fig. 2(d). An increase in annealing time to 60 min at 600 °C further promoted recrystallization (Figs. 1(e) and 2(e)), and the mean grain size and fraction of the HAGBs were 7.5 μm and 68%, respectively. Annealing of the UFG IF-steel at 650 °C for 60 min resulted in full recrystallization of the UFG microstructure (Figs. 1(f) and 2(f)) and onset of the grain growth period. The microstructure after this annealing consisted of coarse grains mostly separated by HAGBs, with a mean grain size and fraction of HAGBs of 13 μm and 72%, respectively (Fig. 2(f)).

3.2. Uniaxial tension behavior

The stress–strain curves of the CG, UFG and annealed UFG IF-steel samples are shown in Fig. 3. The CG steel features low strength but high elongation with a large strain hardening region. Formation of the UFG microstructure drastically increased the