Annealing and strain rate effects on the mechanical behavior of ultrafine-grained iron produced by SPD

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Abstract Thermal stability and strain rate sensitivity of ultrafine-grained (UFG) Fe produced by severe plastic deformation (SPD) were investigated. The UFG Fe was processed by equal-channel angular pressing (ECAP) via route Bc. After 6 passes, the grain size of UFG Fe reaches 600 nm, as confirmed by means of electron back scatter diffraction (EBSD). Examination of micro-hardness and grain size of UFG Fe as a function of post-ECAP annealing temperature shows a transition from recovery to recrystallization. The critical transition temperature is approximately 500 °C, and the material has a bimodal structure after annealing at this temperature. Deformation behaviors of ECAP Fe and ECAP + annealing Fe were studied under both quasi-static and dynamic compressive loadings. The UFG iron shows increased strength and reduced strain rate sensitivity compared with its coarse-grained counterparts. The appropriate post-ECAP annealing can increase strain hardening ability and cancel out thermal softening effect with only a small loss of strength under dynamic loading. (© 2011 The Chinese Society of Theoretical and Applied Mechanics. [doi:10.1063/2.1102102]

Keywords UFG/NC Fe, ECAP, EBSD, thermal stability, strain rate sensitivity

Severe plastic deformation (SPD), such as equalchannel angular pressing (ECAP), has been proven to be an effective method for the production of bulk and fully contamination-free dense metals with sub-micron grain sizes^{1,2}. The mechanical behavior of ultrafinegrained (UFG) Fe processed by SPD has been one of focuses of recent research due to their increased strength/hardness.^{3–7} However, most of reported works on UFG Fe were carried out under quasi-static loading, and few under dynamic loading.⁷ In the present study, both quasi-static and dynamic responses of ECAP Fe were investigated. The post-ECAP annealing effect of deformation behavior was studied by comparing as-ECAPed specimen results with those of post-ECAP annealed specimen. The thermal stability of UFG Fe was also investigated by examining its micro-hardness and grain size as a function of the post-ECAP annealing temperature. The results of the present paper will help not only understand strain rate and annealing effects on the deformation behavior of UFG Fe, but also provide insights for achieving optimal strength/ductility combination in such materials.

The materials used in the present study were received in the form of rods of 60 mm in diameter. The as-received materials have a composition of 0.004 C, 0.01 Si, 0.2 Mn, 0.013 P, 0.007 S, 0.15-0.5 Al, 0.1 Cr, 0.002 Ni, 0.2 Cu (in weight percentage), with the balance of Fe. The as-received rods with a length of 60 mm were extruded for six passes using route Bc of ECAP at room temperature. After ECAP, thermal stability and microstructure were examined by micro-

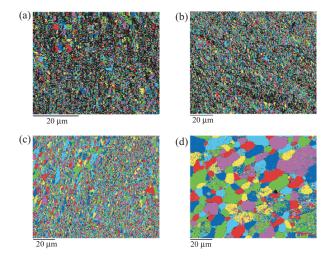


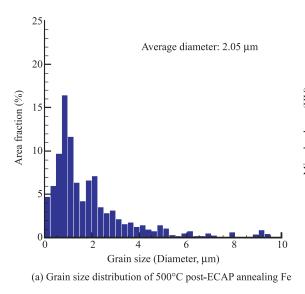
Fig. 1. EBSD maps of (a) ECAP-6 Fe; (b) $400 \,^{\circ}\text{C}$ post-ECAP annealing Fe; (c) $500 \,^{\circ}\text{C}$ post-ECAP annealing Fe; (d) $600 \,^{\circ}\text{C}$ post-ECAP annealing Fe.

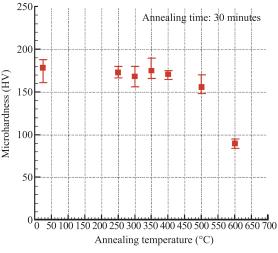
hardness testing and electron back scatter diffraction (EBSD). Small pieces were annealed for half an hour at different temperatures after ECAP in order to investigate the thermal stability of UFG Fe. The annealed sample surfaces were polished with 0.5 μ m diamond paste, and then were etched using 5% Nital to reveal the microstructure. For each annealing temperature, the Vickers micro-hardness was measured using a micro-hardness tester with a diamond indenter under a load of 10 g, for 15 s dwell time. All specimens for mechanical testing were machined from the extruded billets by wire saw with loading direction parallel to the direction of pressing. Following the ASTM standards, the specimens for quasi-static compression have a length

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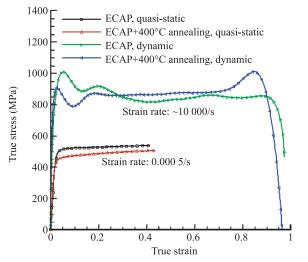
(b) Evolution of micro-hardness with annealing temperature

Fig. 2.

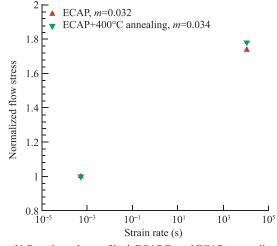
to width aspect ratio of 2.5, and the specimens for dynamic compression (rectangular shape) have a length to width aspect ratio of 0.6. The quasi-static compression tests were performed at a strain rate of 5×10^{-4} /s by MTS 810 universal testing machine, while the dynamic compression were performed at strain rates of 1×10^4 /s by the Hopkinson-bar technique.

Figure 1 shows the evolution of microstructure and grain size after annealing for half an hour at different temperatures. The grains have experienced severe shear deformation, and the average grain size is approximately 600 nm after pressing for 6 passes (Fig. 1(a)). At annealing temperature 400 °C, because only recoverv occurs, there is not much difference in microstructure from that of ECAP-6 Fe, and the average grain size slightly increases to 900 nm (Fig. 1(b)). At annealing temperature 500 °C, the microstructure consists of both ultra-fine grains and newly recrystallized larger grains (Fig. 1(c)). The grain size distribution after $500 \,^{\circ}\text{C}$ post-ECAP annealing is shown in Fig. 2(a). The bimodal structure has an average grain size of approximately 2 μ m, and this bimodal grain size distribution could provide potential for achieving simultaneously good yield strength and fairly large uniform elongation.⁸ At annealing temperature 600 °C, because recrystallization is complete, the microstructure consists of mostly larger grains with an average grain size of approximately $12 \ \mu m$ (Fig. 1(d)). The evolution of micro-hardness of ECAP-6 Fe after annealing for half an hour at different temperatures is shown in Fig. 2(b). The micro-hardness decreases with annealing temperature, revealing a transition from recovery to recrystallization. The critical transition temperature is approximately 500 °C, which has a good correlation with EBSD results.

Figure 3(a) presents true stress-strain curves obtained during both quasi-static and dynamic compression loading for both ECAP Fe and post-ECAP annealing Fe (half an hour). Under quasi-static loading, the ECAP Fe behaves like a nearly elastic-perfectly plastic material. The yield strength is found to decrease slightly during the annealing process, while the ECAP + annealing Fe shows some strain hardening due to recovery effect. Under dynamic loading, the flow stress of ECAP Fe is increased by a factor of 1.8 compared with quasi-static case, while some flow softening behavior is observed. Provided 90% of plastic work is converted into heat, the temperature rise during dynamic loading is estimated to be about 200 K, so the flow softening behavior could be explained by the adiabatic heating and subsequent thermal softening during dynamic loading. Under dynamic loading, the initial flow stress is found to decrease slightly after post-ECAP annealing, however the flow softening behavior is vanished for post-annealed sample because the increased strain hardening ability after annealing cancels out the thermal softening effect during dynamic loading. The rate dependence of the ECAP Fe and ECAP + annealing Fe is shown in Fig. 3(b). The flow stresses of all strain rates at a strain of 0.2 were normalized by that of strain rate of 5×10^{-4} /s. By using a power law with the form of $\sigma/\sigma_0 = (\dot{\varepsilon}/\dot{\varepsilon}_0)^m$, the strain rate sensitivity was derived as the slope of a linear regression fit of double log-log plots of the stress vs. strain rate curve. Although the strain rate sensitivity calculation using flow stress at 20 % plastic strain tested under different strain rates is only an approximation, the values can be compared with those of its coarse grained counterparts calculated in a similar way. The strain rate sensitivities are found to be 0.032 and 0.034 for ECAP Fe and ECAP + annealing Fe, respectively. These results indicate that the UFG iron shows increased strength and reduced strain rate sensitivity compared with its coarse grained counterparts.^[7] Appropriate post-ECAP annealing can increase strain hardening ability and cancel out thermal softening effect with only a small loss of strength under dynamic loading.







(b) Rate dependence of both ECAP Fe and ECAP + annealing Fe

Fig. 3.

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- Y. Iwahashi, Z. Horita, M. Nemoto, and T.G. Langdon, Acta Materialia 46, 3317 (1998).
- 2. R. Valiev, Nature Materials 3, 511 (2004).
- N. Tsuji, Y. Ito, Y. Saito, and Y. Minamino, Scripta Materialia 47, 893 (2002)
- B. Q. Han, E. J. Lavernia, and A. Mohamed, Metallurgical and Materials Transactions A 34A, 71 (2003).
- 5. B. Q. Han, E. J. Lavernia, and A. Mohamed, Metallurgical and Materials Transactions A **35A**, 1343 (2004).
- Y. Ding, J. h. Jiang, and A. d. Shan, Journal of Alloys and Compounds 487, 517 (2009).
- Q. Wei, L. Kecskes, T. Jiao, K. T. Hartwig, K. T. Ramesh, and E. Ma, Acta Materialia 52, 1859 (2004).
- Y. M. Wang, M. W. Chen, F. H. Zhou, and E. Ma, Nature 419, 912 (2002).