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High Temperature Deformation Behavior of Cr-containing Superplastic Iron Aluminide

Seok Hong Min, Woo Young Jung, and Tae Kwon Ha

Abstract**—**Superplastic deformation and high temperature load relaxation behavior of coarse-grained iron aluminides with the composition of Fe-28 at.% Al have been investigated. A series of load relaxation and tensile tests were conducted at temperatures ranging from 600 to 850^{o}C. The flow curves obtained from load relaxation tests were found to have a sigmoidal shape and to exhibit stress vs. strain rate data in a very wide strain rate range from 10^{-7}/s to 10^{-2}/s. Tensile tests have been conducted at various initial strain rates ranging from 3×10^{-5}/s to 1×10^{-2}/s. Maximum elongation of ~500 % was obtained at the initial strain rate of 3×10^{-5}/s and the maximum strain rate sensitivity was found to be 0.68 at 850^{o}C in binary Fe-28Al alloy. Microstructure observation through the optical microscopy (OM) and the electron back-scattered diffraction (EBSD) technique has been carried out on the deformed specimens and it has revealed the evidences for grain boundary migration and grain refinement to occur during superplastic deformation, suggesting the dynamic recrystallization mechanism. The addition of Cr by the amount of 5 at.% appeared to deteriorate the superplasticity of the binary iron aluminide. By applying the internal variable theory of structural superplasticity, the addition of Cr has been revealed to lower the contribution of the frictional resistance to dislocation glide during high temperature deformation of the Fe_3Al alloy.

Keywords**—**Iron aluminide (Fe_3Al), large grain size, structural superplasticity, dynamic recrystallization, chromium (Cr).

I. Introduction

STRUCTURAL superplasticity (SSP) has been usually exhibited in fine-grained materials (~10 μm) under the optimum conditions of strain rate and test temperature [1-3]. Some fine-grained intermetallic compounds, such as Ni_3Al, Ni_3Si, Ti_3Al, and TiAl, have been reported to show an excellent structural superplasticity [4]. Interestingly, the coarse-grained superplasticity has also been reported in Fe-Al based alloys with a grain size of 100~350 μm [5, 6] or even 700~800 μm [7, 8]. All the deformation characteristics such as a large value of strain rate sensitivity, a low flow stresses independent of strain, and high ductility have been exhibited in large-grained Fe-Al based alloys. The mechanism of large-grained superplasticity has, however, been reported and thought to be different from that of the conventional fine-grained superplasticity.

To understand the exact mechanism of a high temperature deformation behavior such as creep and superplasticity, wide range of flow data is necessary. It is well known that load relaxation test can provide a much wider range of strain rates, applying only a little amount of plastic strain to the specimen without an appreciable change in microstructures [9]. It has been attempted, in this study, to establish a better understanding of the mechanism for the large-grained superplasticity and the effect of Cr-addition in Fe-Al based alloys, by employing the high temperature load relaxation test and by applying the recently proposed internal variable theory of inelastic deformation [10].

W. Y. Jung is with the Department of Civil Engineering,, Gangneung-Wonju National University, 120 Gangneung -Daehangno, Gangneung, Gangwon 210-702, South Korea (phone: 82-33-640-2421; e-mail: woojung@gwnu.ac.kr)

T. K. Ha and S. H. Min are with the Department of Advanced Metal and Materials Engineering, Gangneung-Wonju National University, 120 Gangneung -Daehangno, Gangneung, Gangwon 210-702, South Korea (phone: 82-10-2367-1989; e-mail: tkha@gwnu.ac.kr)

II. Experimental Procedures

Iron aluminides with compositions of Fe-28at.%Al and Fe-28at.%Al-5at.%Cr were prepared by vacuum induction melting using the 99.99% purity electrolytic Iron, 99.99% purity Aluminum, and 99.9% purity Chromium. The ingots were homogenized at 1000°C for 5 h and then rolled from 30 mm to 9 mm in thickness, starting at 1000°C and finishing at 800°C. Rod type specimens with the gauge dimensions of 6 mm in diameter and 27 mm in length for load relaxation tests and plate type specimens with 5 mm in length and 3 mm in thickness for tensile tests, respectively, were machined from the hot rolled plates. These specimens were then recrystallized at 857°C for 1 h followed by an oil-quenching.

Load relaxation tests were then carried out at the temperatures from 600 to 850°C by using a computer controlled electro-mechanical testing machine (Instron 1361 model) attached with a furnace capable of maintaining the temperature fluctuation within ± 1°C. It is well known that load relaxation test can provide a much wider range of strain rates, applying a little amount of plastic strain, 1.5 % in this study, to the specimen without an appreciable change in microstructures [9]. Loading strain rate was 5×10^{-2}/s in all cases. In the load relaxation tests employed in this study, the variation of load with time during the tests was monitored through a DVM and stored in a personal computer for the subsequent analysis. The flow stresses as a function of the inelastic strain rate were determined by following the usual procedure described in the literature [11]. A series of tensile tests were also carried out under the various strain rates ranging from 3×10^{-5}/s to 10^{-2}/s to examine superplastic deformation behavior.

Deformed microstructure was observed using the conventional optical microscopy (OM) and the electron back-scattered diffraction (EBSD) technique. EBSD has been carried out on the specimens deformed at 850°C under the strain rates of 3×10^{-5}/s and 1×10^{-3}/s, using a Philips XL-30 scanning electron microscope with a TSL automated orientation imaging microscopy (OIM) attachment. Specimens for EBSD were heated for deformation at the rate of 200°C/min using a radiant heating system to minimize the possible microstructural change during heating and cooling stages. Right after the tensile deformation of 100%, specimens have been cold-water-quenched in all cases. OIM scans were conducted in a hexagonal grid using 10-μm-step size and the orientations were corrected for the 70° tilt of the sample.

III. Results and Discussion

The grain sizes of the thermomechanically-processed materials were very large around 500 μm for binary and about 700 μm for ternary alloys, respectively. Each grain appeared to be equiaxed in both alloys.

The flow curves obtained from load relaxation tests conducted on the binary alloy at temperatures ranging from 600 to 850°C are summarized in Fig. 1. It is noted that flow data obtained are ranging from near 10^{-7}/s to 10^{-2}/s in strain rate. The flow curves are of the sigmoidal shape with maximum strain rate sensitivity, m, in the intermediate strain rate range, which appeared to shift into faster region with increase in testing temperature.

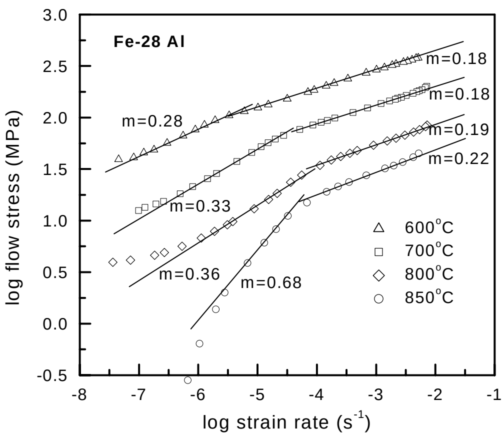

Fig. 1 Flow curves obtained from load relaxation tests on Fe-28 at % Al alloy conducted at various temperatures

Depending on test temperatures, m values varied from 0.28 to 0.68 in lower strain rate range ($\leq 10^{-4}$/s) and from 0.16 to 0.22 in higher strain rate range ($\geq 10^{-4}$/s). The m value in higher strain rate range obtained in this study is somewhat lower than that ($m \approx 0.3$) obtained by Chu et al. in Fe-27at.% Al at 800°C. [7] The difference in m value is thought to be attributed to the difference in grain size and the test method. Maximum value of m has been obtained as 0.68 in the binary (Fe-28at.%Al) alloy, which is the largest ever reported for binary Fe-Al alloys. Close examination of the flow curves reveals that there exist rate-insensitive portions in flow curves except at 850°C, at very low strain rate region around 10^{-7}/s. From the viewpoint of the internal variable theory of inelastic deformation, [10] frictional resistance to dislocation motion due to lattices is considered to be predominant mechanism in this strain rate region. Tensile test results of the binary alloy conducted at 850°C under the initial strain rates from 3×10^{-5}/s to 10^{-2}/s are summarized in Fig. 2 as engineering stress vs. engineering strain curves. With decrease in the initial strain rate, elongation to failure increased up to ~500 %, which is also the largest elongation ever reported for binary Fe-Al alloys. The whole gauge section of specimens appeared to be stretched and necking was developed severely.

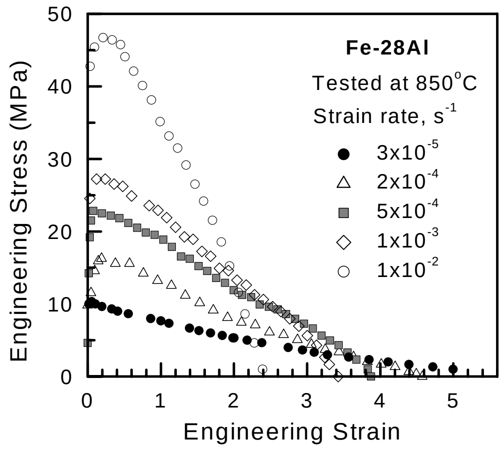

Fig. 2 Tensile test results of Fe-28 at.% Al alloy obtained at 850°C given as engineering stress vs. engineering strain curves

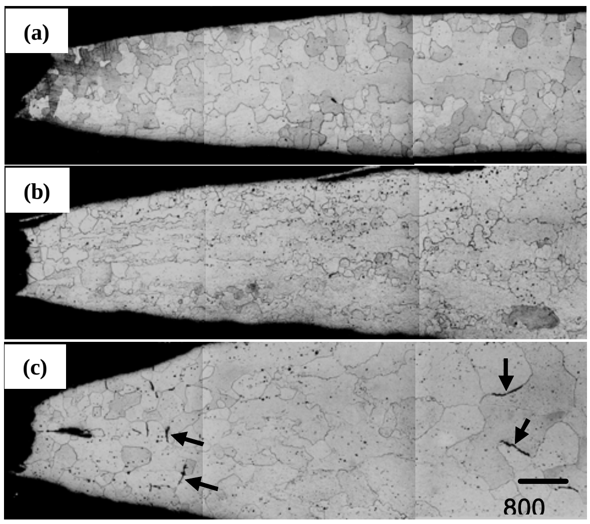

Fig. 3 Optical micrographs taken from surfaces of specimens fractured at 850°C under the strain rate of (a) 2×10^{-4}/s, (b) 1×10^{-3}/s, and (c) 1×10^{-2}/s, respectively

In order to examine the microstructure evolution, longitudinal optical micrographs were taken from fractured specimens along the gauge section and they are shown in Fig. 3. Some grain boundary cavities were found in the specimen tested at the initial strain rate of 10^{-2}/s as marked by arrows.

Despite large elongations to failure, elongated grain structure is not prominent but irregularly curved grain boundaries, evidence for possible grain boundary migration, were observed at the strain rates of 10^{-2}/s and 10^{-3}/s. Partially and fully recrystallized structures can be noted in the specimens deformed at 10^{-3}/s and 10^{-4}/s, respectively. Grain refinement can be observed in all cases at the tip region of fractured specimens. As the strain rate decreased, grain refinement appeared more severe over the whole gauge region. The aspects of grain boundary migration and dynamic recrystallization during superplastic deformation in iron aluminides are exactly consistent with other researchers' observations. [5, 6] Recently, for example, Chu et al. [8] have summarized the microstructure evolution during superplastic deformation of coarse-grained iron aluminides as the sequence of three stages, i.e. subgrain-boundary formation, grain-boundary migration, and formation of recrystallized grains. It can be confirmed from the electron back-scattered diffraction (EBSD) investigation as given in Fig. 4, in which a large number of small-angle boundaries near the gauge region have been observed.

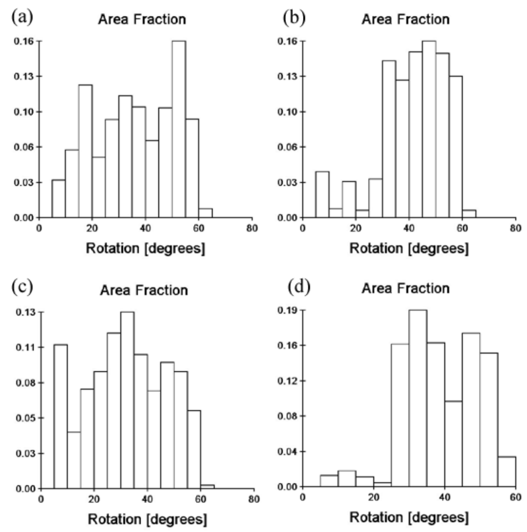

Fig. 4 Frequency histograms of grain or subgrain misorientation taken from the specimens deformed at 850°C under the strain rate of 1×10^{-3}/s, (a) gauge and (b) grip parts, and that of 3×10^{-5}/s, (c) gauge and (d) grip parts, respectively

In the strain rate range of high strain rate sensitivity, however, neither the irregularly curved grain boundaries nor recrystallized grains could be observed, suggesting that a different deformation mechanism could be operated. The result of EBSD given in Fig. 4, however, reveals that the basic deformation mechanism has not changed with the decreasing strain rate at all, since in Fig. 4, both specimens deformed at 3×10^{-5}/s and 10^{-3}/s show the similar distributions of boundary orientation in the frequency histograms.

From the flow data given in Fig. 1, activation energy Q can be calculated by plotting the ln σ against 1/T at a given strain rate. [6] The strain rate of 10^{-4}/s was chosen and the Q value was obtained as 387 kJ/mol in this study as illustrated in Fig. 5. It is interesting to note that the activation energy obtained in the present study is very similar to that for creep deformation of Fe-27at.%Al alloy, [12] suggesting that the mechanism operating during the superplastic deformation of the binary alloy in the strain rate range higher than 10^{-4}/s is likely controlled by the lattice diffusion.

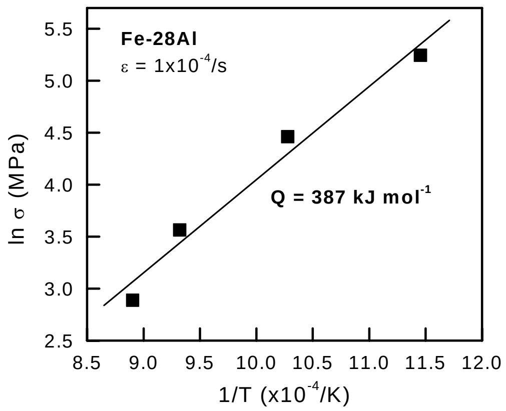

Fig. 5 The plot of ln σ vs. 1/T at the initial strain rate of $\varepsilon = 1\times10^{-4}$/s, providing a means to determine the activation energy

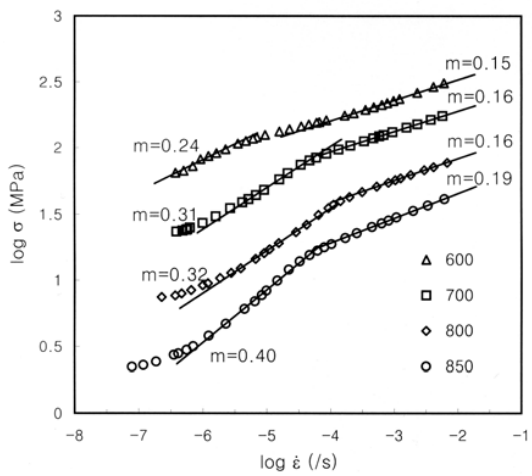

Fig. 6 Flow curves obtained from load relaxation tests on Fe-28 at.% Al-5 at.% Cr alloy conducted at various temperatures

The deformation behavior and the microstructure evolution of the ternary alloy were very similar to those of the binary alloy. The flow curves obtained at the temperature range from 600 to 850 °C are shown in Fig. 6 and the maximum value of strain rate sensitivity was evaluated as 0.40, which is somewhat lower than that of the binary alloy. Elongation to failure was also found to be comparable to but slightly lower than the binary alloy. The maximum elongation of the ternary alloy was obtained as about 400 % at 850°C as shown in Fig. 7. It is, also, noted from this figure that the peak stress of the ternary alloy is higher than that of the binary alloy. Applying the internal variable theory of inelastic deformation, [10] the level of frictional stress due to lattice structure of the ternary alloy was higher than that of the binary alloy. This implies that the ternary alloy is expected to show higher resistance to high temperature creep deformation than the binary alloy.

The activation energy of the ternary alloy was calculated from Fig. 6 and obtained as 414 kJ/mol, somewhat higher but very close to that of the binary alloy. Surface observation of deformed specimens revealed that the dynamic recrystallization appeared to be retarded in Cr-containing ternary alloy as illustrated in Fig. 8. The reduced elongation of the ternary alloy, compared with the binary alloy, seems to be mainly attributed to the less active dynamic recrystallization.

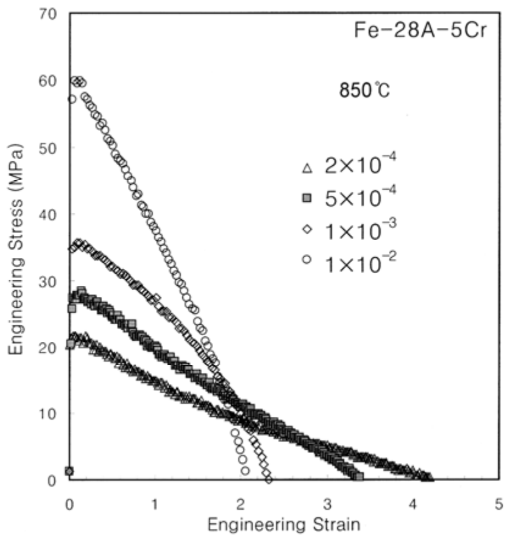

Fig. 7 Tensile test results of the Fe-28 at.% Al-5 at.% Cr alloy obtained at 850°C

IV. Conclusion

The flow curves of Fe_3Al based alloys were sigmoidal shape and the strain rate range of maximum m value, appeared to shift into faster region with increase in testing temperature. A large elongation of about 500 % was obtained in Fe-28at.%Al alloy with the maximum m value of about 0.68, which is the largest ever reported for binary Fe-Al alloys. Microstructure observation revealed the evidences for the strain-induced grain boundary migration and dynamic recrystallization during the deformation at 850°C. The addition of Cr seemingly retarded the dynamic recrystallization. The activation energy Q of 387 kJ/mol for binary and 414 kJ/mol for ternary alloys suggested that the mechanism operating during the superplastic deformation of Fe-28at.%Al alloy in the strain rate range higher than 10^{-4}/s is likely controlled by the lattice diffusion. The addition of Cr to the binary alloy appeared to increase the high temperature strength but decrease the elongation.

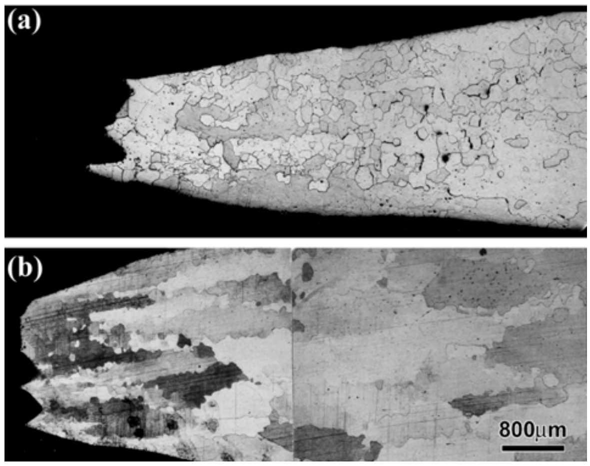

Fig. 8 Optical micrographs taken from surfaces of specimens of (a) binary and (b) ternary alloys fractured at 850°C under the strain rate of 5×10^{-4}/s

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