MODEL OF STATIC RECRYSTALLIZATION IN THE COARSE-GRAINED B2 IRON ALUMINIDE

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Abstract

Microstructure of the laboratory castings made from Fe-40at.%Al-Zr-B alloy was homogenized by the multi-pass hot rolling in protective capsules and subsequent lannealing. Based on the isothermal hot compression tests and EBSD analysis, the sufficiently accurate model describing the static recrystalization kinetics of the such prepared coarse-grained B2 iron aluminide after strain 0.2 was developed. The independent variables in this model are temperature of deformation as well as annealing time equal to the deformation temperature. Unexpected effect of the previous strain value on the static softening was observed. Static recrystallization begins relatively easy in the studied alloy in the whole range of testing temperatures (900 – 1100 °C), but a very long-term annealing is quite necessary for the complete course of recrystallization.

Keywords:

Iron aluminide, hot compression test, microstructure, EBSD, static recrystallization.

1. INTRODUCTION

Iron aluminides are potential replacements for heat-resistant steels due to their excellent high temperature oxidation-corrosion resistance in aggressive environments and low density [1]. They usually offer low density and potentially lower cost than stainless steels. Larger application of iron aluminides is so far impeded by difficulties at their processing by conventional forming methods. From this perspective, the situation is better in the case of Fe_3Al type aluminides, enabling complex thermomechanical processing and annealing producing grain refinement [2]. The hot workability of B2 iron aluminides (typically with 40 at.% Al) is much more problematic. These alloys are extremely susceptible to crack formation in the surface areas cooled by contact with the forming tool [3]. These difficulties are in laboratory conditions eliminated for example by resistant heated anvils at plastometric compression [4]. Method of hot rolling in protective capsules, welded from ferritic stainless steel is very promising [5, 6].

2. HOMOGENIZATION OF THE AS-CAST STRUCTURE

The original objective was to study the kinetics of static recrystallization of the as-cast alloy Fe-40at.%Al-Zr-B using the isothermal plastometric experiments and structural analyses. Iron aluminide with the average chemical composition 24.6 Al – 0.04 Cr – 0.01 B – 0.18 Zr – 0.01 C – 0.14 Mn – 0.01 Mo (remainder Fe – all in wt.%) served as experimental material. In the first stage [7], the cylindrical samples with diameter 10 mm and height 12 mm were manufactured directly from central parts of the laboratory castings of the cross section of approx. 19.5 (thickness) x 33 (width) mm, prepared in the vacuum induction furnace. In spite of ultrasound application during casting and solidification [8] the resulting cast structure was quite heterogeneous and with the exception of thin surface areas also very coarse-grained, with distinct dendrites – see **Fig. 1**.

Heterogeneity of the initial structure caused significant problems at structural analysis of plastometric samples after their uniaxial compression and isothermal annealing, especially at quantification of the recrystallized grains fracture [7]. That's why we tried to homogenize the initial structure by high-temperature annealing of the hot-formed laboratory castings [6]. The heating temperature, as well as the inter-stage heating in the furnace after the second pass lasting 45 seconds, was 1200 °C. The samples were placed into special protective capsules which prevents cleavage of the rolling stock of the iron aluminide with extremely low plasticity [5]. Free cooling of the rolled products was followed by homogenization annealing of their cuts in a vacuum furnace heated to the temperature of 1200 °C. The structure after high temperature annealing lasting 7 hours (see **Fig. 2**) is the result of static recrystallization across the whole volume of the rolled product and of the subsequent coarsening of the fine recrystallized grains in the surface areas. We have thus succeeded in achieving the sufficiently homogenized structure, although at the cost of partial grain coarsening. The EBSD analysis revealed that average grain size was 0.77 ± 0.27 mm after such homogenization annealing of the studied alloy.

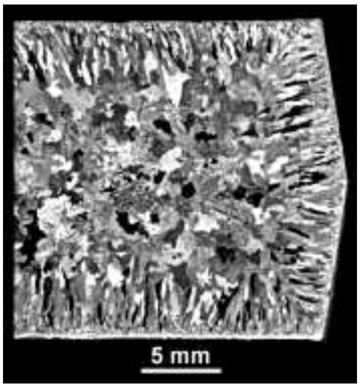


Fig. 1 Heterogeneous macrostructure of the initial casting (partial cross section)

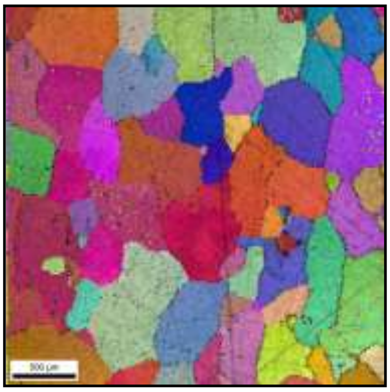


Fig. 2 Microstructure after homogenization annealing at 1273 °C for 7 hours (EBSD)

3. INFLUENCE OF PREVIOUS DEFORMATION ON THE STATIC SOFTENING PROCESSES

Homogenization of the initial structure made it possible to obtain much more reliable information on the kinetics of static recrystallization of the investigated B2 iron aluminide, when applying isothermal annealing and quenching of the plastometric samples after the specific deformation. The cylindrical samples with diameter 10 mm and height 12 mm were manufactured from the central parts of the homogenized rolled products and tested by the isothermal uniaxial compression in the hot deformation simulator Gleeble 3800. The aim of the preparatory stage of experiments was to set the optimal deformation (i.e. strain value) after which the static softening processes have to be evaluated. The samples were deformed by the height strain of 0.10, 0.20 or 0.35 and subsequently annealed at 1100 °C (with a dwell of 5 s before the water quenching), or at 900 °C (with a dwell of 19 s before the water quenching).

The samples annealed at 900 °C show the unambiguous acceleration of static recrystallization by the increasing deformation – see **Fig. 3** and **Fig. 4**. After strain of 0.10 and annealing, the new grains occur at the initial grain boundaries only. Strain of 0.35 yields to the massive progress of recrystallization. To the contrary, annealing at 1100 °C gives the more complicated results. After strain of 0.10 the recrystallized fraction X_R = 0.22; X_R = 0.40 after strain of 0.20, but X_R = 0.28 only after strain of 0.35 (see **Fig. 5**). The higher strain thus always leads to the more pronounced flattening of grains, but not always to the faster static recrystallization.

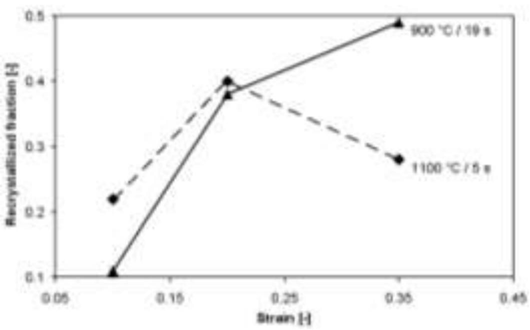
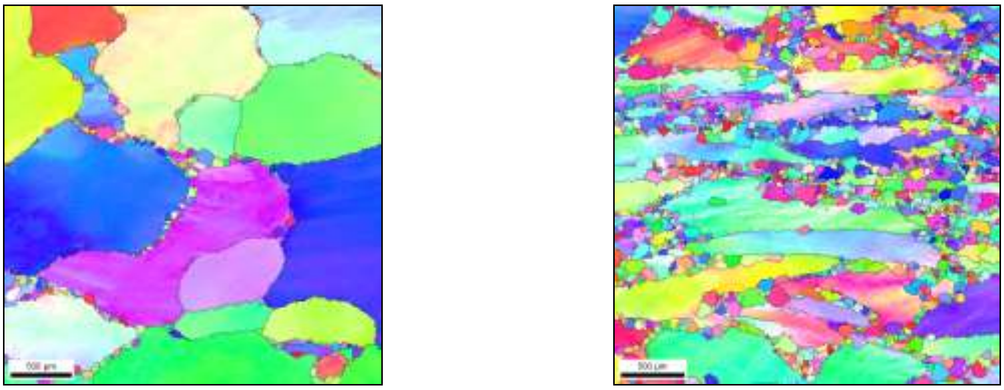
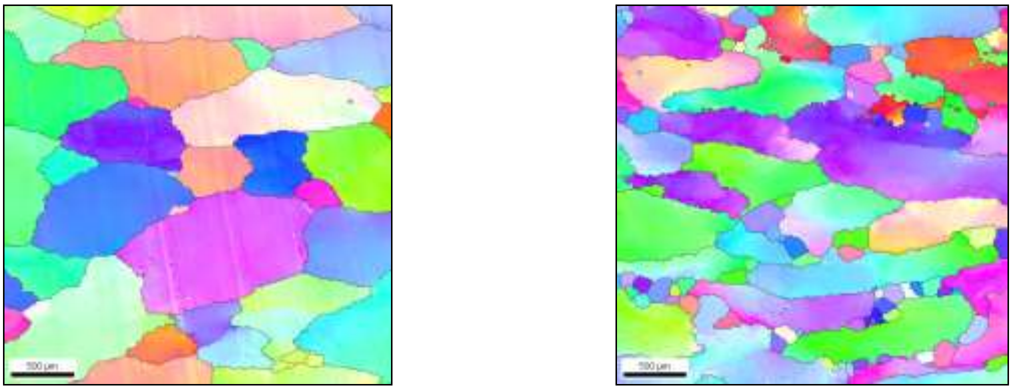


Fig. 3 Influence of previous deformation on the static recrystallization kinetics



a) after strain 0.10 b) after strain 0.35

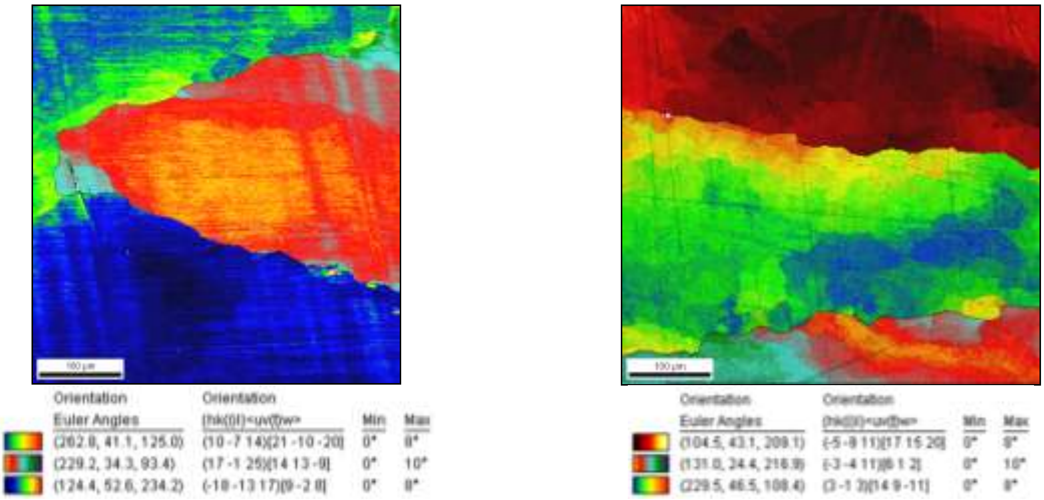
Fig. 4 Microstructure of the samples annealed at the temperature of 900 °C after different deformation (EBSD)



a) after strain 0.10 b) after strain 0.35

Fig. 5 Grain size maps of the samples annealed at temperature 1100 °C (EBSD)

The Forward Scatter Detector (FSD), which enables to distinguish even the subgrains, was used to explain this apparent anomaly. It was proved that high deformation at 1100 °C led to the noticeable progress of polygonization (see **Fig. 6**). Such static recovery competed with static recrystallization and reduced its driving force. The effect of static recovery was minority after low strains.



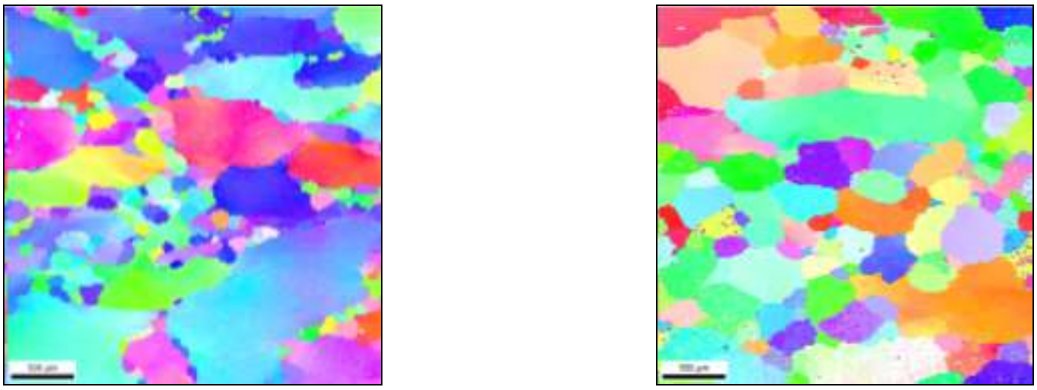
a) strain 0.10 b) strain 0.35

Fig. 6 Detection of the small angle grain boundaries after annealing at 1100 °C (EBSD+FSD)

The SEM and EDX Spectroscopy excluded the possibility of influencing the static softening processes by the precipitation processes acting at the several annealing temperatures.

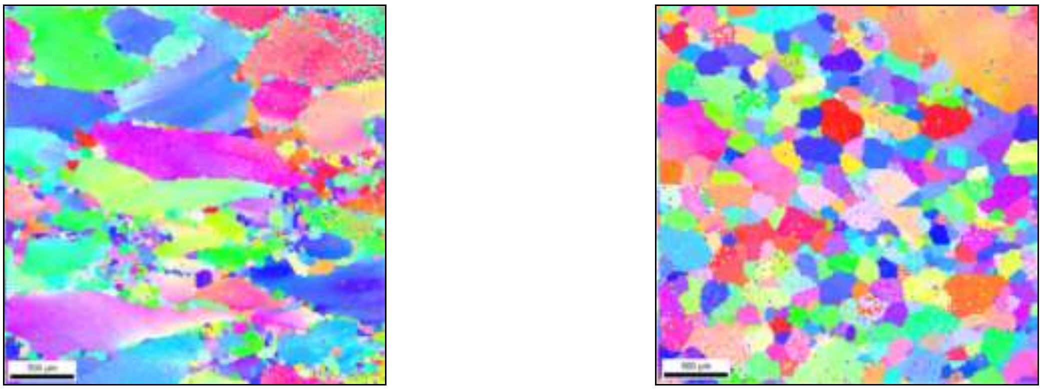
4. DETERMINATION OF THE RECRYSTALLIZED GRAINS

For the above mentioned reasons it was decided to realize the fundamental stage of experiments with the uniform strain 0.20. The plastometric tests were conducted at the deformation temperatures of 900 – 1000 – 1100 °C followed by annealing at these temperatures with a nominal dwell of 0 – 600 s (total of 20 tests). Evaluation of the water-quenched microstructures was made using the EBSD analysis. Course of static recrystallization at high annealing temperature of 1100 °C is surprisingly fast (see **Fig. 7**). At annealing temperature of 1000 °C chains of new grains are evident at the initial grain boundaries after annealing lasting 5 s, annealing lasting 180 s leads to almost complete recrystallization (see **Fig. 8**).



a) annealing time of 5 s　　b) annealing time of 120 s

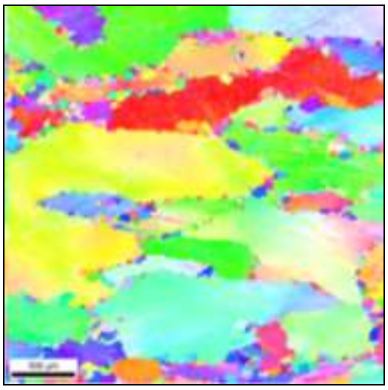
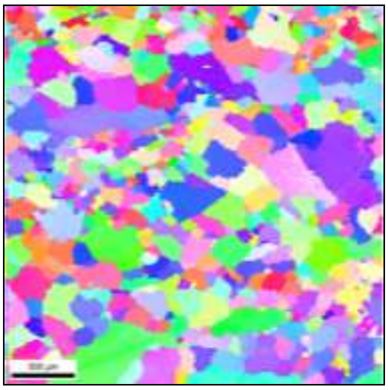
Fig. 7 Microstructure of the samples annealed at the temperature of 1100 °C (EBSD)



a) annealing time of 5 s　　b) annealing time of 180 s

Fig. 8 Microstructure of the samples annealed at the temperature of 1000 °C (EBSD)

At the lowest annealing temperature of 900 °C the chains of new grains are observed at annealing time of 19 s, whereas very long-term annealing leads to selective coarsening of new grains (**Fig. 9**). Mean size of recrystallized grains decreases with the decreasing annealing temperature.

a) annealing time of 19 s b) annealing time of 600 s

Fig. 9 Microstructure of the samples annealed at the temperature of 900 °C (EBSD)

5. DESCRIPTION OF THE STATIC RECRYSTALLIZATION KINETICS

We assume that the process of static recrystallization can be described in the terms of the Johnson, Mehl, Avrami and Kolmogorov (JMAK) model [9-11]:

$$X_R = 1 - \exp\left(-B \cdot t^n\right) \tag{1}$$

where t [s] is annealing time. The constant B depends on the mechanisms of nucleation and growth, and n is an Avrami exponent. According to the former experiences [12], the coefficients in Eq. (1) were evaluated by multiple nonlinear regression on experimental data. Independent variables were the temperature T [K] and the nominal annealing time t [s]. The effect of the annealing temperature was introduced into Eq. (1) by a modification of the quantity B. The resulting phenomenological model is:

$$X_R = 1 - \exp\left[-28.7 \cdot \exp\left(\frac{-7265}{T}\right) \cdot t^{0.66}\right] \tag{2}$$

Good agreement of experimental values with values obtained using Eq. (2) is obvious in **Fig. 10**.

Comparison of the obtained results with those obtained by other authors is difficult due to significantly different experimental conditions and the nature of the initial material. Bystrzycki [13] annealed a similar alloy at the maximum temperature of 800 °C and he observed full recrystallization after approx. 10 s, but the previous shock-wave deformation at room temperature accumulated in the material much higher density of defects than hot forming, which was used here. Problems with final structure homogeneity were mostly recorded. Two distinct grain structures were observed in annealed B2 FeAl samples by Samajdar et al. [14] – small equiaxed grains and large columnar grains.

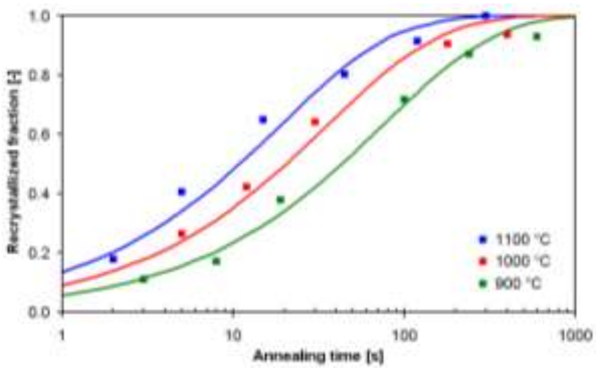


Fig. 10 Demonstration of the accuracy of Eq. (2) (solid lines) in comparison with experimental data (points)

6. CONCLUSIONS

Microstructure of the laboratory casting made from Fe-40at.%Al-Zr-B alloy was homogenized by hot rolling and thermal processing. Based on the isothermal plastometric tests and EBSD analysis, the sufficiently accurate model describing the static recrystalization kinetics of the such prepared coarse-grained B2 iron aluminide after strain 0.2 was developed. The independent variables in this model are temperature of deformation as well as annealing time equal to the deformation temperature. Unexpected effect of the previous strain value on the high-temperature static softening was observed, with the noticeable competition acting between recrystallization and recovery processes.

Static recrystallization begins relatively easy in the studied iron aluminide in the whole range of testing temperatures (900 – 1100 °C), but a very long-term annealing is quite necessary for the complete course of recrystallization. Heterogeneous course of recrystallization in the coarse-grained initial structure yields in growth of some new grains. High-temperature thermal processing after deformation has to take several hours for obtaining the homogeneously recrystallized microstructure, which is unserviceable in practice, e.g. from point of view of real inter-pass times at hot rolling.

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