STATIC RECRYSTALLIZATION OF INTERMETALLIC COMPOUND OF
THE TYPE Fe-40at.%Al-Zr-B

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Abstract
It was possible to form successfully the samples from aluminide of the type Fe-40at.%Al-Zr-B by uniaxial compression due to heating of forming tools. The initial material was obtained in the form of laboratory castings with highly heterogeneous structure, which made an objective evaluation of structure-forming processes difficult. It was necessary to roll the castings in protective capsules slightly and anneal them at the temperature of 1200 °C for the time that would ensure full recrystallization and homogenization of the formed structure. The samples with homogenized structure were subjected to deformation 0.2 at the temperatures from 900 to 1100 °C and to following isothermal annealing lasting from 0 to 240 s. Microstructure fixed by water cooling was then subjected to structural analyses, which led to obtaining of unique information on kinetics of static recrystallization of the investigated alloy.

Keywords: iron aluminides, as-cast microstructure, hot forming, annealing, static recrystallization

1. INTRODUCTION
The iron aluminides are considered as potential structural materials for many engineering applications. They usually offer a good corrosion resistance in many aqueous environments, low density and potentially lower cost than stainless steels. The amount of aluminum present in aluminides can range from 10 to 30 wt. % and is significantly higher than the aluminum concentrations present in conventional alloys and superalloys. The alumina layer formed on the surface of the materials is responsible for their excellent oxidation and carburization resistances even at temperatures over 1000 °C [1, 2]. Larger spreading 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 Fe3Al type aluminides, enabling complex thermomechanical processes comprising hot forging, hot rolling, warm rolling and annealing yielding to grain refinement [3]. There exists an effort to improve the hot workability through electroslag remelting in these alloys [4].

The hot workability of B2 iron aluminides (typically with 40 at. % or with about 24 wt. % Al) is much more problematic. These alloys are extremely susceptible to crack formation in the surface areas cooled by contact with the forming tool [5]. These difficulties, which are evident particularly in the as-cast condition, are in laboratory conditions eliminated for example by resistant heated anvils at plastometric compression [6]. Use of hot extruded alloy powders produced by expansive gas-atomization and water-atomization techniques and hot extrusion, accompanied by favourable state of stress, is more frequent [7, 8]. Milling of a Fe-40Al alloy powder in a high energy planetary ball-mill was applied by [9]. Interesting results were achieved by application of the shock-wave explosive deformation [2]. Method of hot rolling in protective capsules, welded from ferritic stainless steel with hot deformation behaviour, which is very close to the behaviour of aluminide Fe-40Al, is very promising [10]. The ability of this method to deform coarse-grained...
as-cast material and refine thus effectively the grain size and homogenize the microstructure led to the interest in studies of the kinetics of static recrystallization of coarse-grained aluminide Fe-40at.% Al-Zr-B. The first results of this method are subject of this article.

2. EXPERIMENTAL PROCEDURE

Iron aluminide with average chemical composition 24.6 Al – 0.04 Cr – 0.01 B – 0.18 Zr – 0.01 C – 0.14 Mn – 0.01 Mo (balance Fe – all in wt.%) served as experimental material. In the first stage, the cylindrical specimens with diameter 10 mm and height 12 mm were manufactured directly from central parts of laboratory castings of the cross section ca 19.5 (thickness) x 33 (width) mm, gained by means of the vacuum induction furnace.

After the uniform heating to the temperature of 1200°C the samples were subjected on the plastometer GLEEBLE 3800 to uniaxial compression at the strain rate of 4 s⁻¹. Logarithmic vertical strain 0.2 was achieved at 1200 or 1000°C. The samples were after deformation immediately quenched in water or annealed at the given deformation temperature for 5 to 100 seconds and then quenched. It was decided after evaluation of 8 tests results that a homogenization of the initial cast structure will be necessary before recrystallization annealing. This was done by rolling the castings in protective capsules by altogether 4 reductions with magnitude of deformation of 0.1. Before the 1st and the 3rd passage the castings were placed into the furnace with the temperature of 1200°C. The final thickness of the rolled products was 13.3 mm. The resulting structure was still not sufficiently homogeneous, that's why we applied their homogenization annealing in a vacuum furnace at the temperature of 1200°C. The samples for plastometric studies were again manufactured from the annealed rolled products that were conducted at the temperatures of deformation of 1100 - 1000 - 900°C followed by annealing at these temperature with a dwell of 0-240 seconds (total of 12 tests). Evaluation of structures was made metallographically or using the Electron Backscatter Diffraction (EBSD) analysis.

3. EVALUATION AND DISCUSSION OF RESULTS

In spite of ultrasound application during casting and solidification [11] 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. Non-homogeneity of the initial structure was reflected negatively at investigation of kinetics of static recrystallization in the first stage of the experiment – see Fig. 2.

![Structure of the laboratory casting](image-url)
Fig. 2 Results of EBSD analysis of annealed samples – initial cast structure.
This is particularly evident at the annealing temperature of 1200°C, when it is impossible to remove the columnar grains by re-crystallization (Fig. 2b), and after longer time the structure is partially formed also by grains that became coarsened after recrystallization (Fig. 2c). The static recrystallization starts at the borders of original grains (Fig. 2d) at the annealing temperature of 1000°C and the dwell of 10 s. The share of recrystallized grains increases with the increasing duration of annealing (Fig. 2e), but even after a period of 100 s not all grains were transformed (Fig. 2f). The lower annealing temperature leads to the formation of finer recrystallized grains, but the heterogeneity of the structure and grain size are significant in the case of both temperatures. The necessary homogenization annealing was performed on several rolled samples. It was established that dwell at the temperature of 1200°C for 7 hours leads to sufficient homogenization of structure by static recrystallization by and subsequent coarsening of recrystallized grain – compare the photos shown in Figs. 3a and 3b. Investigation of static recrystallization in the second stage of the experiment, i.e. with initial homogenized structure of samples provides significantly more representative results.

At high temperature of 1100°C annealing process is surprisingly fast course of static recrystallization at high annealing temperature of 1100°C is surprisingly fast (Fig. 4a), after 120 s of annealing the structure is formed by recrystallized and coarsened grains (Fig. 4b).

At mean annealing temperature of 1000°C chains of new grains are evident at the initial grain boundaries after annealing lasting 2 seconds (Fig. 5a), annealing lasting 180 seconds leads to almost complete recrystallization. At the lowest annealing temperature of 900°C almost perfect course of static recrystallization was recorded already after 44 s of annealing (Fig. 6a), long-term annealing leads to selective coarsening of new grains (Fig. 6b). Mean size of recrystallized grains decreases with the decreasing annealing temperature and their size distribution within individual samples is much more favorable than in the case of the samples with initial cast state.
At the lowest annealing temperature of 900°C almost perfect course of static recrystallization was recorded already after 44 s of annealing (Fig. 6a), long-term annealing leads to selective coarsening of new grains (Fig. 6b). Mean size of recrystallized grains decreases with the decreasing annealing temperature and their size distribution within individual samples is much more favourable than in the case of the samples with initial cast state. 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 [2] annealed a similar alloy at the maximum temperature of 800°C and he observed full recrystallization after approx. 10 seconds, 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. Yang and Baker [12] achieved in the alloy Fe-40Al by its annealing at the temperature of 900°C the size of recrystallized grain of approx. 230 μm, which is analogous with the result presented in Fig. 6a (900°C / 44 s). For comparison – a much longer duration of approx. 600 s was needed for achieving of the complete course of static recrystallization of the disordered intermetallic Fe-17.3Al-3.8Cr-0.3Zr-0.13C (at.%) alloy [13]. Problems with final structure homogeneity were mostly recorded. Two distinct grain structures were observed in annealed B2 FeAl samples by Samajdar et al. [8] – small equiaxed grains and large columnar grains. The large columnar grains favored the [111] fiber texture. Possible selection of [111] directions for growth was due to grain boundary character distribution.
4. SUMMARY

It was shown that in the as-cast alloy Fe-40at.%Al-Zr-B with initial coarse-grained structure it is relatively easy to initiate static recrystallization by high-temperature forming and annealing, but it was very difficult to achieve a uniform grain size by this process. For achievement of sufficient structure homogeneity it was necessary to apply annealing at 1200°C for 7 hours, after which all recrystallized grains got uniformly coarsen. A complete recrystallization after isothermal dwell of the order of 100 s was achieved by forming and annealing of the samples with homogenized initial structure at the temperatures of 900 – 1100°C. Susceptibility to coarsening of new grains in the investigated alloy is comparatively high. The recrystallized shares and grain size for all 13 samples (including the initial homogenized state) will be determined after more detailed analysis of the results of the second stage of the experiment, which should lead to the possibility to quantify the parameters of the Avrami equation, describing the kinetics of static recrystallization of the alloy Fe-40at.%Al-Zr-B, depending on the temperature and duration of annealing after hot deformation.

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REFERENCES