INFLUENCE OF THE MICROSTRUCTURE ON FLOW STRESS AND DEFORMABILITY OF IRON-ALUMINIUM ALLOYS

Julius PEDDINGHAUS, Kai BRUNOTTE, Hendrik WESTER, Michael TILL, Christoph KOCK, Bernd-Arno BEHRENS

Leibniz University Hannover, Institute of Forming Technology and Machines, Garbsen, Germany, EU, peddinghaus@ifum.uni-hannover.de

https://doi.org/10.37904/metal.2022.4393

Abstract

Due to their higher weight-specific and high-temperature strength, iron-aluminium alloys have a high potential to replace steel in various applications. The good availability of the two materials, the excellent recyclability, lower density with increasing aluminium content and the high corrosion resistance in sulphide- and sulphur-rich environments are further advantages. However, with increasing aluminium content, ductility of FeAl alloys decreases due to hydrogen embrittlement at room temperature. As a result, iron-aluminium alloys have been excluded from potential applications, particularly structural ones. Investigations on powder metallurgical produced iron-aluminium alloys show that fine-grained microstructures can lead to significant improvement in ductility. Assuming equal grain diameters, higher toughness is expected in case of metallurgical ingot production followed by hot forming. The present work deals with the mechanical properties of fine-grained microstructure in iron-rich iron-aluminium alloys, pre-processed through Equal Channel Angular Pressing. In order to characterize the mechanical properties, compression tests with the alloys Fe9Al, Fe28Al and Fe38Al are carried out at different temperatures. The flow curves determined are then compared with those from as-cast state. In addition, deformation capacity is examined optically on slopes of external cracks. In conclusion, the results are discussed based on the microstructure.

Keywords: Iron-aluminium alloys, Microstructure, Equal Channel Angular Pressing (ECAP), Hot forming; Flow stress

1. INTRODUCTION

Due to their corrosion resistance in oxygen- and sulphur-containing environments, their high melting point and specific strength iron-aluminium alloys (Fe-Al alloys) are used, for example, as high temperature materials in the aerospace industry [1]. Depending on the composition, iron-aluminium alloys are also used for other industrial applications, e.g. brake disks for windmills and trucks, filter systems in refineries and fossil power plants, transfer rollers for hot-rolled steel strips as well as ethylene crackers and air baffles to burn high-sulphur coal [2-3]. Fe-Al alloys are categorised as the intermetallic compounds of the two elements aluminium and iron. This type of compound features completely different properties compared to its pure metallic alloy components. Depending on mixing ratio and temperature, the iron-aluminium alloys can exist in three essential intermetallic phases FeAl (α), FeAl (β') and Fe3Al (β') on the iron-rich side [4-6]. Due to their low price, the good availability and recyclability of iron and aluminium, Fe-Al alloys are also interesting for industrial use from an economic point of view [4]. Iron-aluminium alloys have however long been neglected in structural applications due to the very low ductility at room temperature caused by hydrogen embrittlement in humid environments and because of the rapid drop in strength when increasing temperatures above 600 °C [5].

With an increasing aluminium content, the fracture behaviour of iron-rich Fe-Al alloys changes from ductile to brittle at approx. 12 at.%-Al, when the trans-crystalline fracture occurs. The reason for the trans-crystalline fracture of Fe-Al alloys is hydrogen embrittlement [7]. The decreasing ductility with increasing aluminium content is an exclusion criterion for the large-scale use of Fe-Al alloys. Several studies deal with the increase in ductility and creep strength by alloying with chromium, molybdenum and other additives. As a result, elongations of up to 7 % could be achieved for the Fe3Al-phase of Fe-Al alloys, but this value is still below the required 10 % to make it suitable for structural component production [5,6].

Grain refinement is one of the few strengthening mechanisms that favours both strength and ductility on a micrometre scale [9]. Hall et al. demonstrated that a decreasing grain diameter of a metal structure causes an increase in yield strength and established the Hall-Petch equation [10,11]. Grain refinement also increases the grain boundaries. It is assumed that the interaction of the grain boundary atoms contributes to the ductility of metals [9]. In addition, studies have shown that grain boundaries are an obstacle to the movement of hydrogen, which could reduce the danger of brittle fracture due to hydrogen embrittlement of Fe-Al alloys [12].

Morris et al. summarized research results on the influence of grain size on the ductility of binary Fe-Al alloys produced by powder metallurgy [9]. As the grain diameter decreases, the ductility increases exponentially. In this way, elongations of up to 7 % could be achieved.

In contrast to the ultra-fine-grained structure produced by Morris et al. [9] this work deals with the generation of a fine-grained structure by means of hot bulk forming using the Equal Channel Angular Pressing (ECAP) process. For this purpose, the results of previous works were taken into account, where Fe-Al alloys were examined for their mechanical properties in the as-cast state, then for forming behaviour and finally the mechanical properties after forming in hot forging and hot impact extrusion [13,14]. The toughness of components made by powder metallurgy is generally lower than that of components produced by casting and forming due to the increased porosity caused by the manufacturing process. As a result, an improved elongation from the casting and forming of ultra-fine-grained Fe-Al alloys can be expected.

In previous works, the microstructures of the three alloys Fe9Al, Fe28Al, and Fe38Al in the as-cast state [14] and after an ECAP forming were examined [15]. In all three alloys, a grain refinement and homogenization of the structure could be observed after forming compared to the as-cast state. This study deals with the results of the mechanical investigations after ECAP in flow curves and the material's deformation capacity. These results are then compared with those from the as-cast state and discussed.

2. MATERIALS AND METHODS

In order to test the properties in forming behaviour tests, samples with the nominal compositions Fe9Al, Fe28Al and Fe38Al (at.%-Al) were selected to represent the three iron-rich phases A2, B2 and D03. These binary iron-aluminium alloys were cast to ingots (140 mm x 140 mm x 500 mm) and then wire-cut lengthwise to 20 mm flat samples using wire electrical discharge machining (EDM). Subsequently, incremental hot forming was performed to reduce the grain size without cracking and to eliminate possible casting defects such as internal pores. According to Huskic [14], Fe9Al was shaped at 1250 °C, Fe28Al at 1150 °C and Fe38Al at 1100 °C with a punch speed of 30 mm/s. Afterwards, the samples were heat treated at 750 °C in order to homogenize the microstructure and increase the deformability of the forged specimens. Sikka et al. [5] showed that a maximum increase in ductility of approx. 15 to 20 % in Fe3Al alloys with an Al content of 28 at.%-Al can be achieved after a thermo-mechanical process and a heat treatment of one hour at temperatures of 750 °C. Huskic's [14] heat treatment at the same temperature gave similar results for the three examined alloys. In the process, most of the formed grains recrystallized, which led to a grain refinement in comparison to the cast structure and an improvement in the deformability. The holding times determined by Huskic [14] (1 hour for Fe28Al and Fe38Al; 2 hours for Fe9Al) were used for the heat treatment. After incremental hot forming and heat treatment, the samples were wire-cut by means of wire EDM from a blank geometry of 40x40x240 mm to the required ECAP sample size of 14 mm x 14 mm x 75 mm.

The samples and the ECAP tool were heated to 1100 °C for 15 minutes (samples) and 45 minutes (tool) in a chamber furnace. To avoid premature cooling of the samples, they were placed in the preheated ECAP tool and heated together in the chamber furnace. Afterwards tool and sample were transferred into the press and formed with a constant punch speed of 5 mm/s. The angle Φ of the ECAP die was 120°, which corresponds to a true plastic strain of $\varphi = 0.7$ in the sample. After forming, the samples were cooled to room temperature in still air and then heat-treated at same parameters as after incremental hot forming (Fe28Al/Fe38Al for 1 hour and Fe9Al for 2 hours at 750 °C). The exact test implementation and tool structure of the ECAP process is described in [15].

3. RESULTS

3.1 Fe9Al

Figure 1 shows the results of the flow curves of cast and ECAP formed Fe9Al samples. It can be seen that the flow curves at all temperatures and forming speeds are higher in the ECAP-formed samples than in as-cast state. The lower the temperature, the higher the difference between the two states. The largest increase in the flow curves of ECAP formed Fe9Al can be observed at 700 °C, a deformation rate of 10 s^{-1} and a true plastic strain of $\varphi = 0.7$ where the flow stress has doubled from 250 MPa to 500 MPa. The flow stress of the ECAP formed Fe9Al alloys is already well above the flow stress of the cast Fe9Al alloys at the beginning of the forming process.

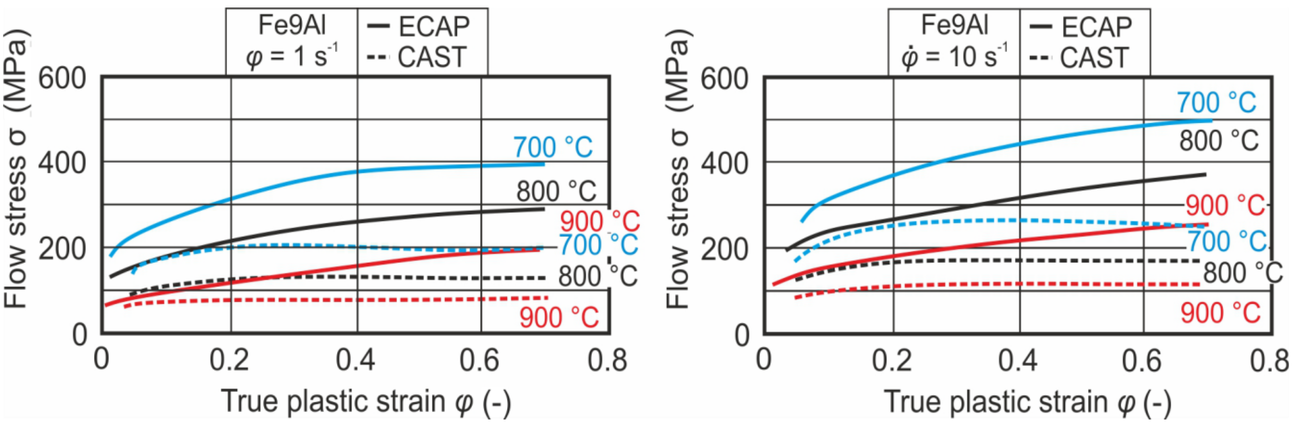

Figure 1 Comparison of the flow curves between casted [14] and ECAP formed Fe9Al upsetting samples at different temperatures, forming states and forming speeds of $\dot{\varphi} = 1\ s^{-1}$ (left) and $\dot{\varphi} = 10\ s^{-1}$ (right)

3.2 Fe28Al

Figure 2 shows the results of the flow curves of Fe28Al. Except at 800 °C and the forming speed of 10 s^{-1}, all samples start in a similar stress range, regardless of the state. At a true plastic strain higher than $\varphi = 0.1$, the flow stress of the ECAP formed samples does exceed the flow stress of the samples in the as-cast state. At the beginning of the forming process, stress peaks can be observed in both material states.

In the as-cast state at 700 °C and a deformation speed of 10 s^{-1}, fracture has occurred, which indicates a lower deformability. The flow stresses for Fe28Al are generally higher compared to Fe9Al, since the Fe28Al alloy has a higher strength.

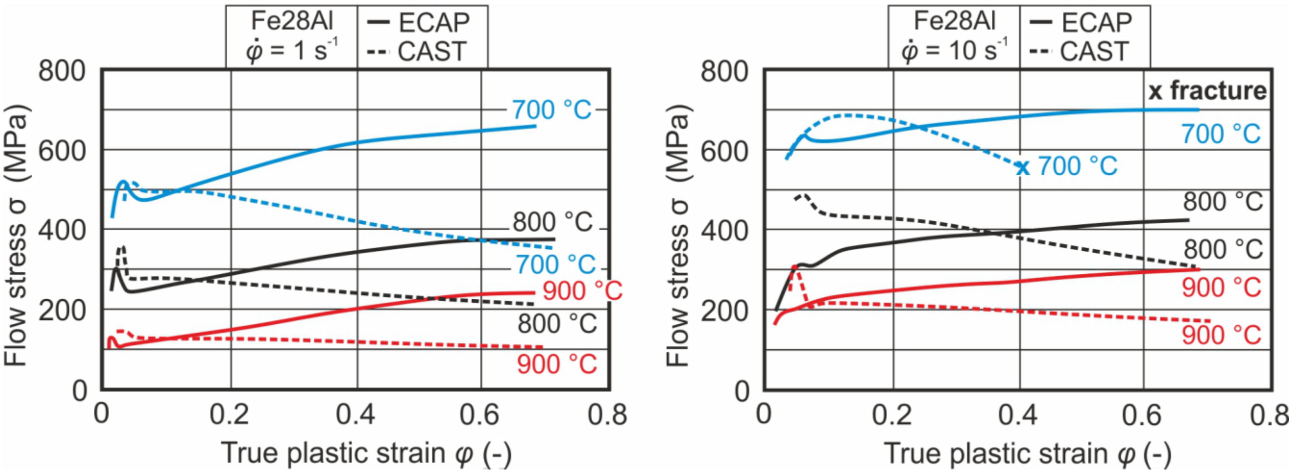

Figure 2 Comparison of the flow curves between casted [14] and ECAP formed Fe28Al upsetting samples at different temperatures, forming states and forming speeds of $\dot{\varphi}$ = 1 s^{-1} (left) and $\dot{\varphi}$ = 10 s^{-1} (right)

3.3 Fe38Al

The Fe38Al alloy showed the highest flow stresses of all three alloys, since a higher aluminium content leads to higher strength. However, as this also reduces the deformation capacity, the temperature was adjusted accordingly, so that a range from 800 °C to 1000 °C was investigated. As seen in **Figure 3**, the Fe38Al samples from the ECAP process also have a higher flow stress than those in the as-cast state. The clearest difference is shown by results at 800 °C and a forming speed of 1 s^{-1}. Here, the flow stress of approx. 350 MPa in the as-cast state is significantly lower than the 700 MPa of the ECAP formed samples. At the deformation speed of 10 s^{-1}, as with Fe28Al, an improvement in the deformation capacity of ECAP formed samples compared to the samples in the as-cast state can be observed. At 900 °C a fracture occurs in the sample in the as-cast state, whereas the ECAP formed sample can be formed over the full path. Only at 800 °C the ECAP sample fails with a similar true plastic strainlike the sample in as-cast state.

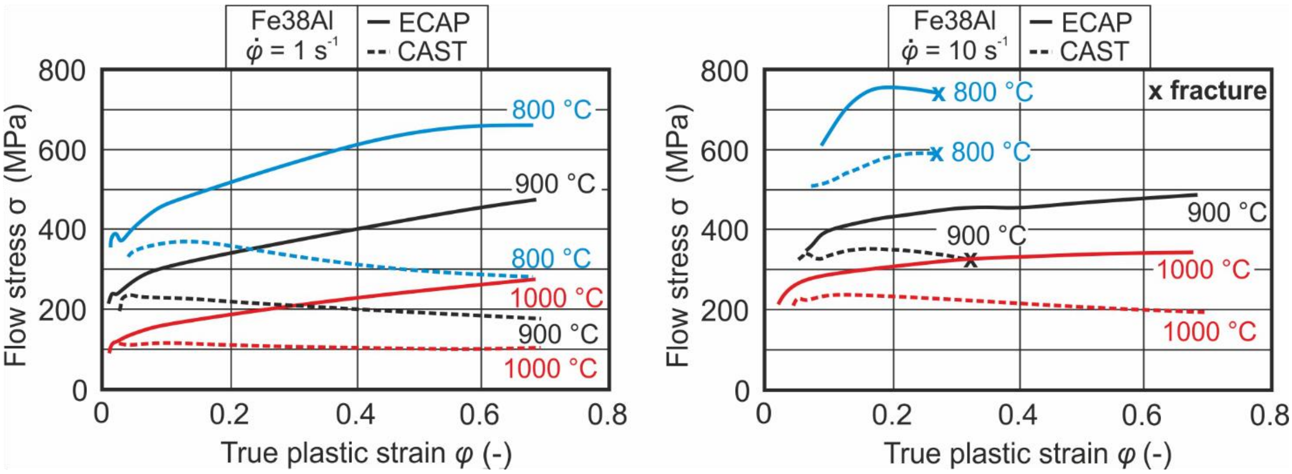

Figure 3 Comparison of the flow curves between casted [14] and ECAP formed Fe38Al upsetting samples at different temperature, forming state and forming speed of $\dot{\varphi}$ = 1 s^{-1} (left) and $\dot{\varphi}$ = 10 s^{-1} (right)

4. DISCUSSION

Regardless of the alloy, all samples formed using ECAP show a higher flow stress than the as-cast samples. **Figure 4** shows the microstructure of the three alloys in the as-cast state and reshaped after the ECAP forming. The mean grain diameter in the as-cast state is over 2 mm for all three alloys. In contrast, the mean grain diameter of the ECAP formed samples is significantly smaller (average grain size of Fe9Al approx. 0.5 mm, Fe28Al approx. 0.4 mm and Fe38Al approx. 0.3 mm).

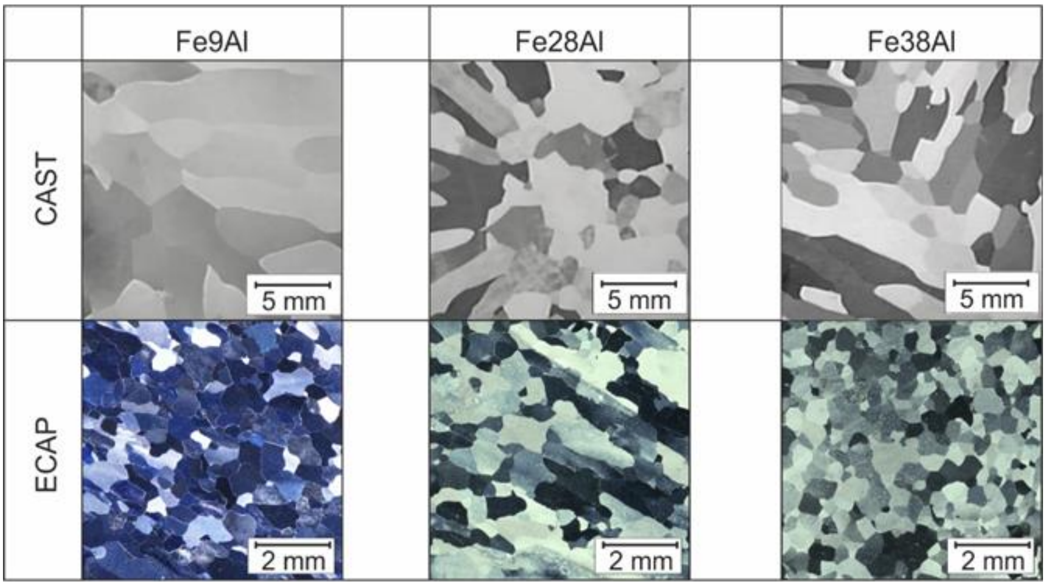

Figure 4 Microstructure of the casted samples [14] (top) and ECAP formed samples (bottom) of the alloys Fe9Al, Fe28Al and Fe38Al

Since the dislocations in smaller grains accumulate faster at the grain boundaries, the strength of the ECAP formed grains increases significantly compared to the as-cast samples. Huskic [14] justifies the partial decrease in the flow stress of the upset samples from the casting in his work through the interaction of dynamic recrystallization and hardening by dislocations. The hardening mechanism in the case of smaller grain sizes seems to be more pronounced in the ECAP formed upset samples, which increases the flow stress.

In the case of the Fe28Al and Fe38Al samples, the higher deformation speed resulted in fracture formation. Firstly, the Fe38Al sample failed at 900 °C (as-cast condition), the Fe28Al sample failed at 700 °C (as-cast state). In addition to the improved flow stress of the two alloys, the higher deformation capacity is particularly noteworthy. The ECAP formed Fe28Al sample at 700 °C and high forming speed as well as the Fe38Al sample at 900 °C could be reshaped completely and without cracking compared to the as-cast sample. It can be assumed that the enlargement of the grain boundaries due grain size refinement leads to an increase in the grain boundary bond.

5. CONCLUSION

This work shows that the microstructure refinement of iron-aluminium alloys through the ECAP process leads to an increase of flow stress and deformation capacity. The 4 to 6 times smaller grain diameters as the cast of the three alloys Fe9Al, Fe28Al and Fe38Al achieve a doubling of the flow stress compared to the cast state. In addition, due to the grain refinement, the enlargement of the grain boundaries of the two brittle alloys Fe28Al and Fe38Al have apparently led to an improvement in the grain boundary bond. The critical forming temperature that leads to fracture was lowered in the upsetting tests. The results demonstrate a significant potential to broaden the application spectrum for FeAl alloys through ECAP processing.

ACKNOWLEDGEMENTS

The authors would like to thank the German Research Foundation (DFG) for the financial support [funding number: 142849202].

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