Structure of rolled ferrite alloyed with 9.3 wt.% Al and 0.5 wt.% Zr

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Abstract

The previous experiments had shown that the rolling temperatures of iron aluminides based on Fe_3Al must be about 1200 °C to initiate the softening during and between individual passes. Material with lower content of aluminium (outside the phase field of iron aluminides) was tested in the laboratory rolling process, because the lower rolling forces are expected for the disordered lattice. Zirconium additions are expected to enhance the strength of the material for any practical use at higher temperatures.

K e y w o r d s: steel with Al and Zr, forming process, recrystallization, microscopy techniques

1. Introduction

Ordered intermetallic alloys exhibit generally good high-temperature strength, low density, and environmental resistance. In particular the corrosion resistance was reported (see e.g. [1–5]) as benefitial for high-temperature practical applications.

The precipitates formed during cooling from the annealing temperature of 1150 ℃ and remaining stable at the testing temperatures of 600–800 ℃ were suggested to be responsible for the strength increase. The main attention was paid to the influence of second phase particles formed in Fe_3Al-type iron aluminides alloyed with various additives, e.g. [6, 7]. The formation of Zr-rich particles depends strongly on the zirconium to carbon ratio. Recent experiments to improve Fe-Al alloys in ordered (aluminides) and disordered (ferrite) version by low amounts of Zr were reported by many authors, e.g. Alven and Stoloff [8, 9], Morris et al. [10, 11] and Kratochvil et al. [12].

The important technology used for initial working of casts from Fe-Al alloys is high temperature rolling. The present contribution aims to summarize the knowledge obtained during the laboratory hot rolling of the iron aluminide-like alloy, but with the lower content of Al. This situates the alloy into the field of ferrite in the binary Fe-Al phase diagram.

T a b l e 1. Composition of the investigated alloy

	Al	Cr	Zr	C	Fe
wt.%	9.20	3.90	0.52	0.03	balance
at.%	17.30	3.81	0.29	0.13	balance

2. Experimental methods

The composition of the rolled alloy is given in Table 1.

The alloy was prepared at the Institute of Modelling and Control of Forming Processes of the Technical University of Ostrava. Vacuum melting (1–10 Pa) was carried out in the induction furnace Leybold. The slabs having cross section of 20 × 32–35 mm were cast.

The slabs underwent two different rolling procedures:

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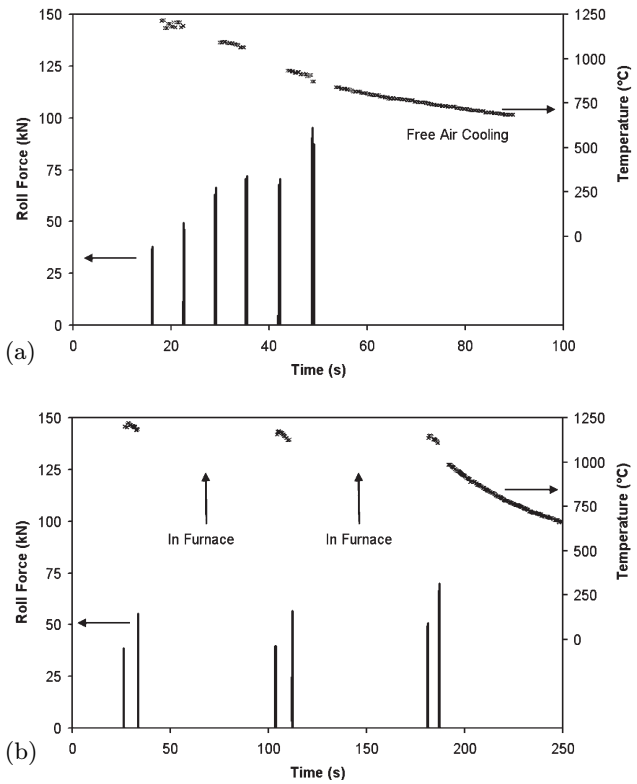

Fig. 1. The scheme of the rolling procedure: (a) rolling with free air cooling; (b) rolling with two additional heating at 1300 °C min^{-1}.

1. Rolling after heating to 1280 °C in several passes using laboratory four-high mill K350 down to the final thickness of 3.5 mm. The samples with a thickness of 3.5 mm were annealed at 1300 °C for 20 min and then cooled to the temperature of forming of 800, 900, 1100 and 1200 °C (temperature was homogenized 10 minutes in another furnace). Each sample was then single-pass rolled using the 32 % thickness reduction and quenched into oil either immediately or after 2.5 minutes hold in the furnace at the forming temperature. The deformation rate was 28 s^{-1}.

2. Heat treatment at 1250 or 1300 °C for 20 min and rolling by 6 reverse reductions of 18–21–22–21–21–19 % in the mill K350 to the final thickness of 5 mm. The interpass time was set to reach various finishing temperatures. During these interpass intervals the slab was either air cooled or heated in the furnace. The finishing temperatures were in the range of 1133–816 °C. The rolled specimens were free air cooled. The deformation rate was 10–19 s^{-1} depending on the individual reductions.

The rolling process is illustrated in Fig. 1. In this figure, the effect of temperature on the roll force is obvious.

The structure was studied using the following experimental techniques:

a) light optical microscopy (LOM) with the application of Nomarski contrast, etching by emulsion OP-S (Struers) and by Rollason solution (100 ml H_2O + 50 ml 38 % HCl + 5 g $FeCl_3$),

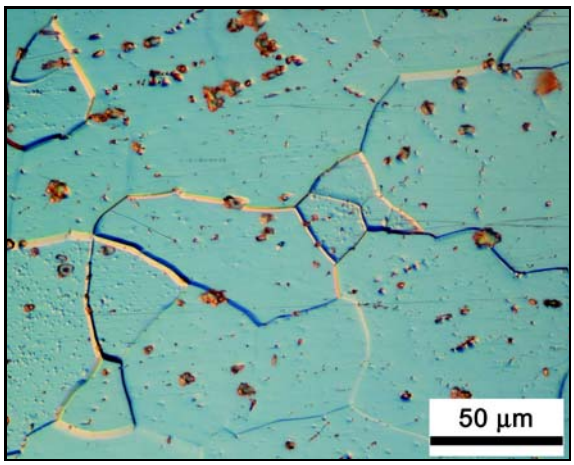

Fig. 2. Grain refinement under the surface of the sample rolled at 1200 °C.

b) X-rays diffraction (XRD) with XRD7 – FPM for the phase identification from the cut parallel to the rolling direction. The investigated area was 3.5 or 5.0 × 20 mm,

c) transmission electron microscopy (TEM) at 200 kV. Samples were prepared by spark cutting and the foils prepared by electrolytic double jet polishing at −40 °C using 10 % solution of HNO_3 in methanol,

d) electron microanalysis (EDAX) for the phase identification.

The samples for tensile and creep tests (the gauge length of 25 mm, the sample diameter of 5 mm) were prepared with the axis parallel to the rolling direction. The tensile tests were performed at temperatures ranging from 400 to 800 °C in air using the deformation machine INSTRON 1186. The initial strain rate was 1.5 × 10^{-4} s^{-1}. The creep experiments were carried out under constant load at a temperature of 800 °C. The temperature was kept with the accuracy of 3 °C during both tests.

3. Experimental results and discussion

The effect of rolling temperature and hold after single pass rolling on the grain structure and substructure was studied. Static recrystallization (SRX) took place during the hold at 1300 °C and the grains elongated during the subsequent rolling at 800 °C, 900 °C, 1100 °C and 1200 °C.

At hold temperatures of 1100 °C and 1200 °C, where SRX may be expected, the grain refinement in the surface layer was observed – see Fig. 2.

Moreover, different structure in the samples rolled at 1100 °C and then quenched with and without the

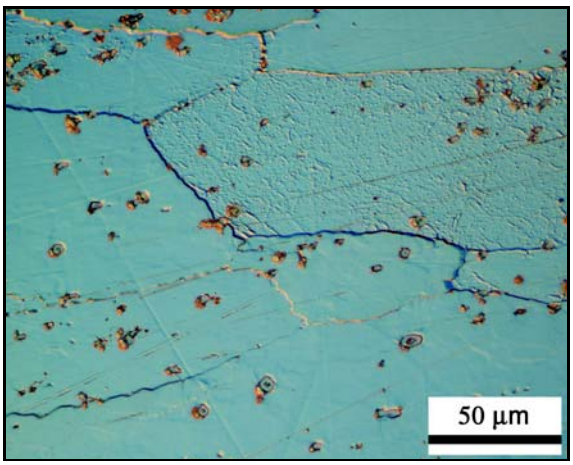

Fig. 3. Structure of the slab rolled at 1200 °C and oil quenched.

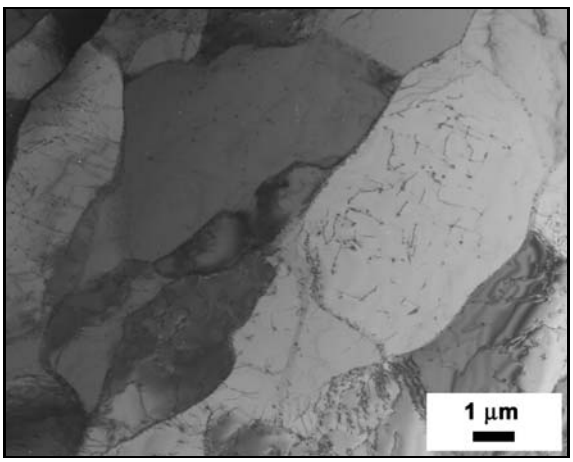

Fig. 4. Subgrains in the samples rolled at 1100 °C and oil quenched.

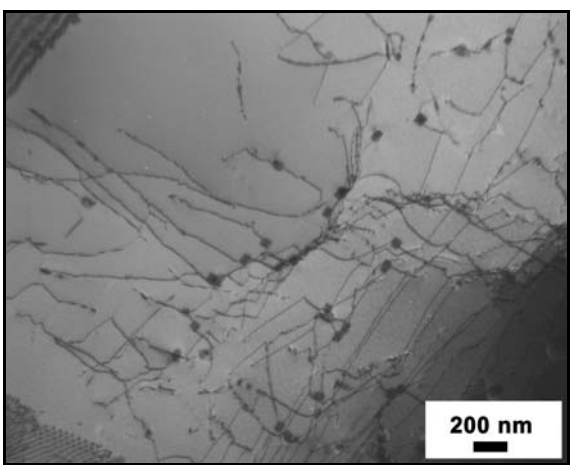

Fig. 5. Dislocations and particles in the subgrain in the samples rolled at 1100 °C and oil quenched.

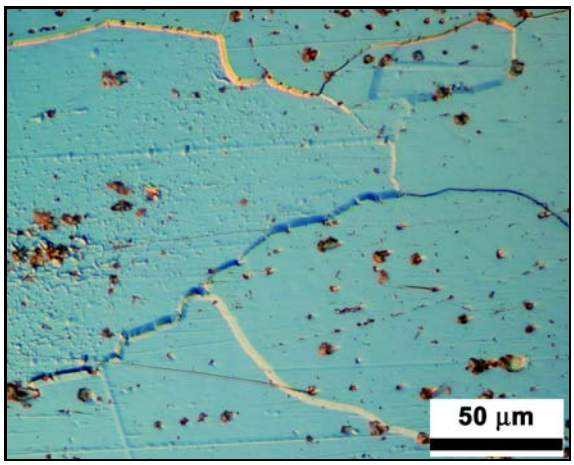

Fig. 6. Structure of the slab rolled at 1100 °C and hold at 1100 °C before oil quenching.

hold at 1100 °C was observed. Grain boundary migration takes place during the hold and bulging is observed (Fig. 3). Fe_2Zr and ZrC particles acting as obstacles for the movement of grain boundaries were identified by EDAX and XRD. SRX resulting in equiaxed grains is completed only in sample after the hold at 1200 °C. The recrystallized structure has its origin in the dynamic recrystallization (DRX).

The process operating during the high temperature deformation at 1100 °C and 1200 °C was studied using TEM. In both cases the typical features of dynamic softening were observed, in particular, subgrains with moderate density of dislocations inside the subgrains (Fig. 4). The particles are situated mostly on dislocations and in the subgrain boundaries (Fig. 5). The size of the particles is about 100 nm.

The temperature of the finishing pass influences the structure and substructure after multipass rolling. The structure of slabs heated initially at 1300 °C or 1250 °C differs only slightly. Moreover, holds between individual interpass cooling or heating have no observable effect on the structure of the final rolled product. Individual experiments differ only in the finish rolling temperature (1133–816 °C). At temperatures of 1133 °C and 1107 °C grain boundaries were found in the state of the interrupted migration. Grain boundaries are pinned by particles (Fig. 6).

For lower finish rolling temperatures the observed structure corresponds to the plastic deformation of a set of grains at this temperature. The observed shape of the grain boundary depends both on the orientation of the grain boundary with respect to the direction of the applied forming force, and on the orientation of the slip plane (slip direction) with respect to the rolling direction.

During rolling at high temperatures due to the stress distribution in slip planes plastic deformation occurs in all grains. In two neighbouring grains plastic

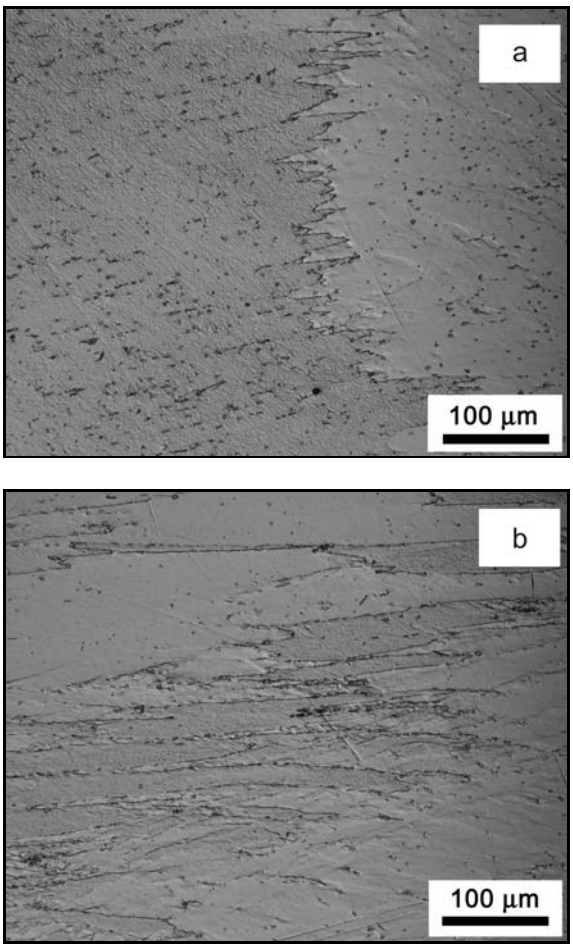

Fig. 7. The grain boundary originates during its migration (finish rolling temperature 920 °C). It is stopped in a zig-zag position (a) or the migration of the grain boundary proceeded further (b) and the elongated grains penetrate into the neighbouring grain.

Table 2. The yield stress $\sigma_{0.2}$ and the fracture strain $\varepsilon_{\mathrm{pl}}$ at 400–800 °C

Temperature (°C)	$\sigma_{0.2}$ (MPa)	$\varepsilon_{\mathrm{pl}}$ (%)
400	370	12.2
500	375	30.5
600	244	12.6
700	131	26.3
800	47	64.2

deformation occurs preferentially in the grain with higher stress in the slip plane. The grain boundary migrates and expands in the neighbouring grain. Consequently toothed, zig-zag grain boundary is formed (Fig. 7a). Grain boundary migration (glide deformation) between two obstacles stops quickly or is blocked by next obstacle after the pass is completed. During the extension of the teeth of the zig-zag boundary deep in neighbouring grain separate new grains appear (Fig. 7b). The observed phenomena are very similar to those observed during the grain boundary migration in copper [13].

Table 3. Example of values of MCR and TTR at 800 °C

Load	MCR	TTR
5 MPa	5.0×10^{-11}	9850 hours
10 MPa	4.1×10^{-9}	3350 hours

In order to evaluate material properties at high temperatures [14] the characteristic parameters were determined from mechanical tests, in particular the offset 0.2 % yield strength and the plastic strain to rupture at higher temperatures (strain rate was $10^{-4}\,\mathrm{s}^{-1}$). These results are summarized in Table 2. The creep tests had shown that the material was applicable for the use at high temperatures (up to 800 °C). As an example the data of minimum creep rate (MCR) and the time to rupture (TTR) at 800 °C are given in Table 3.

4. Conclusions

1. The experiment with single pass rolling has shown that at least the temperature of 1100 °C must be reached to initiate the softening process (either dynamic or static). In order to guarantee the recrystallized structure for the start of any rolling operation heating at 1200 °C is therefore sufficient.

2. The temperature of 1200 °C is high enough for the multipass rolling to guarantee sufficient softening for the rolling process. Even at finish temperatures substantially below that of recrystallization (either SRX or DRX), e.g. 816 °C, the rolling is possible.

3. After low finish temperature special shapes of the grain boundaries appear. Non-recrystallized structure does not hinder the practical use of sheets prepared using this procedure. Before any handling the sheet has to be reheated to start softening. The sheets of this material are intended to be employed as high temperature protection of less quality materials in corrosion environments.

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