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## OPTIMIZATION OF LABORATORY HOT ROLLING OF BRITTLE Fe-40at.%Al-Zr-B ALUMINIDE

### OPTIMALIZACJA PROCESU WALCOWANIA NA GORĄCO ALUMINIDKU Fe-40at.%Al-Zr-B NA PODSTAWIE PRÓB LABORATORYJNYCH

Use of the protective steel capsules enabled to manage the laboratory hot flat rolling of the extremely brittle as-cast aluminide Fe-40at.%Al-Zr-B with the total height reduction of almost 70 %. The hot rolling parameters were optimized to obtain the best combination of deformation temperature (from 1160°C up to 1240°C) and rolling speed (from 0.14 m·s<sup>-1</sup> to 0.53 m·s<sup>-1</sup>). The resistance against cracking and refinement of the highly heterogeneous cast microstructure were the main criteria. Both experiments and mathematical simulations based on FEM demonstrated that it is not possible to exploit enhanced plasticity of the investigated alloy at low strain rates in the hot rolling process. The heat flux from the sample to the working rolls is so intensive at low rolling speed that even the protective capsule does not prevent massive appearance of the surface transverse cracking. The homogeneity and size of product's grain was influenced significantly by temperature of deformation, whereas the effect of rolling speed was relatively negligible. The optimal forming parameters were found as rolling temperature 1200°C and the rolling speed 0.35 m·s<sup>-1</sup>. The effective technology of the iron aluminide Fe-40at.%Al-Zr-B preparation by simple processes of melting, casting and hot rolling was thus established and optimized.

*Keywords:* iron aluminides, as-cast structure, low plasticity, hot rolling, recrystallization, EBSD analysis, grain size, FEM simulation

Zastosowanie ochronnych stalowych kapsuł pozwoliło na przeprowadzenie laboratoryjnego walcowania na gorąco pasm z niezwykle kruchego odlewanego aluminidku żelaza (stopu na osnowie fazy międzymetalicznej) Fe-40at.%Al-Zr-B, które umożliwiło redukcję wysokości o prawie 70%. Parametry walcowania na gorąco były dostosowywane celem uzyskania korzystnego zakresu temperatury odkształcenia (od 1160°C do 1240°C) oraz prędkości walcowania (od 0.14 m·s<sup>-1</sup> do 0.53 m·s<sup>-1</sup>) dla otrzymania pasma bez pęknięć i rozdrobnienia silnie niejednorodnej mikrostruktury odlewu. Zarówno eksperymenty jak i matematyczne symulacje oparte na MES wykazały, że niemożliwe jest uzyskanie dobrej plastyczności badanego stopu przy niskich prędkościach odkształcenia podczas walcowania na gorąco. Strumień ciepła płynący z próbki na walce jest tak intensywny przy małej prędkości walcowania, że nawet kapsuła ochronna nie zapobiega pojawieniu się powierzchniowych pęknięć poprzecznych. Jednorodność i rozmiar ziarn w materiale silnie zależy od temperatury odkształcenia, podczas gdy wpływ prędkości walcowania jest relatywnie niewielki. Optymalne parametry kształtowania stopu to: temperatura 1200°C oraz prędkość walcowania 0.35 m·s<sup>-1</sup>. Skuteczna technologia przygotowania aluminidku żelaza Fe-40at.%Al-Zr-B w prostym procesie topienia, odlewania i walcowania na gorąco została w ten sposób ustalona i zoptymalizowana.

## 1. Introduction

Iron aluminides are of interest due to their excellent high temperature oxidation–corrosion resistance in aggressive environments, and they are potential replacements for high temperature steels [1]. The Fe-based composition ensures relative cheapness, while the high Al content leads to significant density reduction in comparison with commercial steels (6530 kg·m<sup>-3</sup> for Fe<sub>3</sub>Al, 5370 kg·m<sup>-3</sup> for FeAl composition). Iron aluminides are characterized by good

resistance to catalytic coking, sulphidation, carburization and wear. As a result, these alloys found their application as air deflector for burning high sulphur coal and transfer rolls for hot rolled steel strip [2]. Iron aluminides can become commercially attractive, if the hydrogen embrittlement problem in these materials can be overcome [3]. Troubles of low ductility at room temperature and poor high temperature strength have prevented significant commercial use [4]. Moreover Fe-Al alloys contain high concentrations of point defects, which adversely affects the mechanical properties

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of their as well as significantly increases with the increase in aluminum content [5]. The additions of zirconium and boron improve tensile properties of the Fe-40at.% Al based intermetallic alloys at room and elevated temperatures. Increase of boron content changed the fracture mode from intergranular decohesion to cleavage, which correlates with significant increases in the fracture toughness. Boron was also found to increase grain boundary strength and modify the formation of the second phase particles [6, 7].

The aim of experimental works was to optimize the parameters of processing the extremely brittle alloy of type Fe-40at.%Al in the as-cast state by laboratory hot rolling, especially with utilization of the proved effect of special protective capsules.

The rolling procedure used for this material has been patented [8]. The sample is placed into a special welded capsule. The reason for its use is to prevent the nucleation of cracks on the surface of the hot rolled casting. Two factors are important:

- The walls of the capsule prevent the direct contact of the cold work rolls with the surface of the hot rolled casting. Cooling of the rolled material surface is thus minimized.
- The unfavourable shear stresses originate in this case on the contact surface of the capsule wall and not on the surface of the material with very low plasticity.

It is evident that a very close agreement of the deformation behaviour of both materials, i.e. of the protective capsule and of the rolled iron aluminide must exist. After some optimization experiments, the corrosion-resistant ferritic steel X6Cr17 was applied for the protective capsule [9], which is prepared by bending and welding of a sheet with thickness 2 mm. The capsule design also prevents from the undesirable spread of the rolled sample. Attention is paid to the appropriate location of the vent ports in the corners of the capsule, through which the hot air can safely escape during the heating and proper forming.

A successful rolling of the brittle as-cast iron aluminide, which is protected by the welded capsule, requires maintaining of the special procedure [10] that is, nevertheless, much less demanding than for example the methods based on the powders' sintering [11] or roll compaction of water atomized FeAl powder with a polymeric binder [12].

## 2. Experimental procedures

The material of type Fe-40at.%Al-Zr-B was obtained by melting in the laboratory vacuum induction furnace. It had the following chemical composition: 24.56 Al – 0.04 Cr – 0.01 B – 0.18 Zr – 0.01 C – 0.14 Mn – 0.01 Mo – Fe remainder (all in wt.%). The aluminium content corresponded to 40.3 at.%. The molten metal was cast into the divided cast-iron moulds. The rectangular samples (with thickness 19.4 mm, width 33 mm and length 100 mm) were obtained by melting and casting in the vacuum laboratory furnace, using ultrasound that improves the chemical and structural homogeneity of the casting [13]. The castings were protected by the above described capsules and

rolled on the laboratory four-high mill stand K350 by 7 reversible passes to the nominal thickness of 6 mm (without the steel layers). The work rolls with diameter of 67 mm rotated at the speed from 40 to 150 rpm. After heating to the rolling temperature (ranging from 1160 to 1240 °C), which corresponds to a range ordering decline [14] the material was rolled flatways to 1/3 of the thickness, using the relatively heavy height reductions (approx. 11 – 12 – 14 – 16 – 19 – 19 – 19 %). After every even pass the semi-finished product was put into the furnace heated to the rolling temperature, so that the decline of the surface temperature of samples could be eliminated. The intermediate pause was 45 seconds after the pass No. 2, 60 seconds after the pass No. 4 and 90 seconds after the pass No. 6. The finish rolling was always followed by cooling in free air to the room temperature. After cooling of the rolled down samples the protective capsules were mechanically removed (see the illustrative Fig. 1) and the structural analyses of the samples' upright longitudinal cuttings were carried out, preferentially by EBSD.

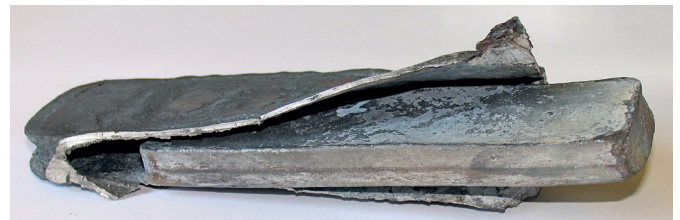


Fig. 1. Example of the rolled semi-product (with thickness of 12.7 mm) with partly removed protective capsule

## 3. Discussion of results

### 3.1 Structure of castings

The cardinal task of the hot rolling was to refine the initial structure by the repeated static recrystallization and thus to improve technological formability of the examined brittle alloy. The very coarse-grained and heterogeneous structure of the as-cast material is demonstrated in Figs. 2 and 3. The grains with the size over 2 mm are not exceptions.

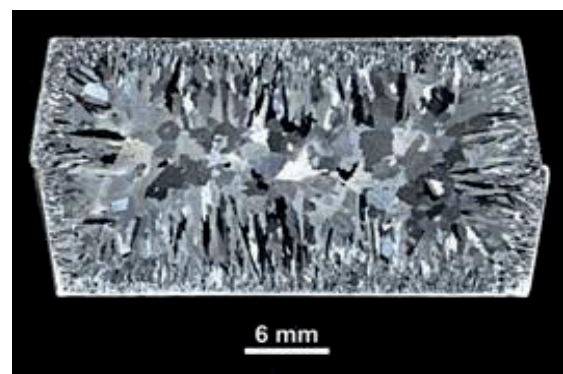
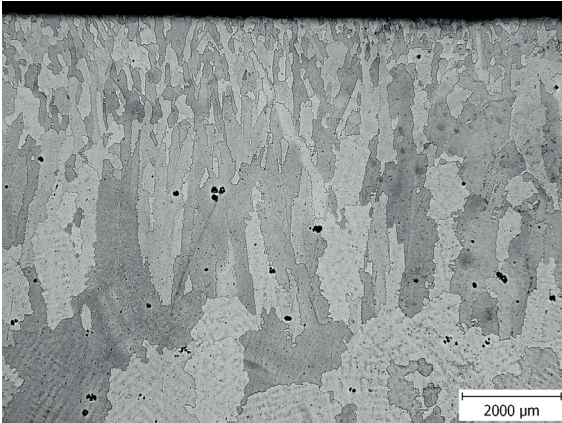
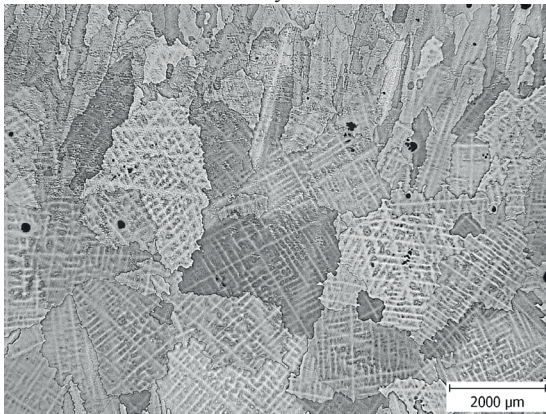


Fig. 2. Macrostructure of the laboratory casting (cross section)





a) Surface area with columnar crystals



b) Central area with dendrites

Fig. 3. Microstructure of the laboratory casting (cross section)

### 3.2 Occurrence of cracking

We can see the attempt to roll the casting without the protective capsule at 1200°C on Fig. 4. The first pass (with height reduction of 11%) already led to massive cross cracking. Some cracks propagated even on the sides of the product. It is obvious that the given material's formability is so low that application of the common rolling methods would not be possible.

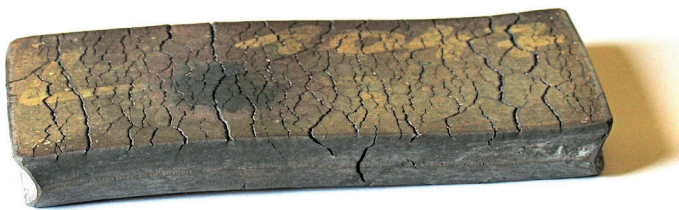
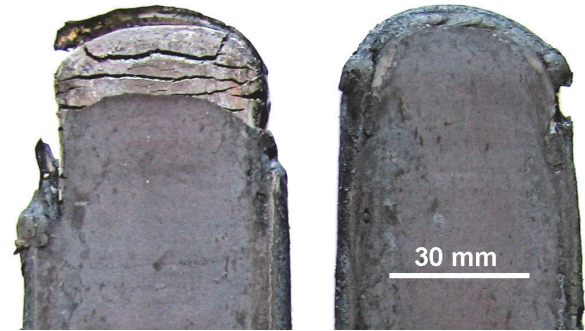


Fig. 4. The cracks on surface of the sample rolled without the protective capsule (final thickness is 17.3 mm)

Some protective capsules tore in the most complicated joints – see Fig. 5 for example. It was found that the tendency to capsule's tearing grows with decreasing both temperature and rolling speed. Such behaviour is related to the real temperature of rolled material. On the basis of the previous studies and results of papers [15, 16, 17] the following rolling temperatures were applied: 1160°C – 1180°C – 1200°C –

1220°C – 1240°C, while the rolls' speed was kept constant 100 rpm (the rolling speed is 0.35 m·s<sup>-1</sup> in this case). The strain rate corresponding to such conditions is 7.0 s<sup>-1</sup> for the 1st pass growing to 16.0 s<sup>-1</sup> for the 7th pass; for details of calculation see [18]. The optimal both formability and final microstructure was found at the products rolled at 1200°C and further experiments with the varied rolls' speed were performed at this temperature. The rolls' speed was changed including these values: 40 rpm – 80 rpm – 120 rpm – 150 rpm. The rolling speed changed from 0.14 m·s<sup>-1</sup> (for 40 rpm) to 0.53 m·s<sup>-1</sup> (for 150 rpm).

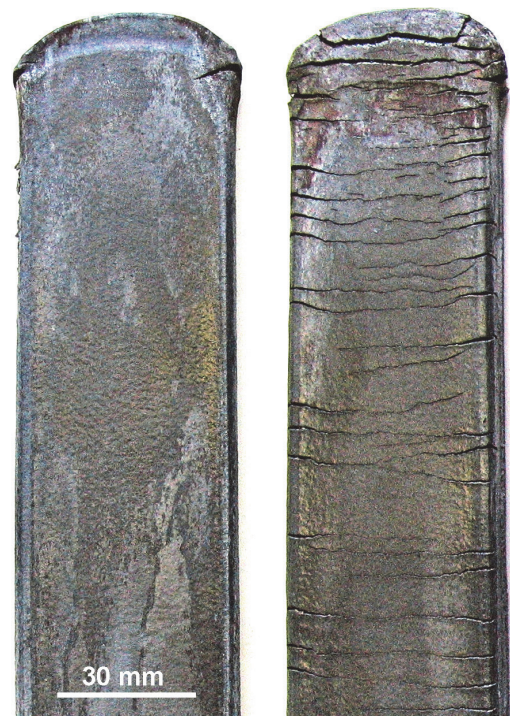


a) 40 rpm

b) 150 rpm

Fig. 5. The effect of the rolls' speed on the fracture of protective capsules; rolling temperature was 1200°C

The significant cracks were observed on all places, where the protective capsules were broken, while the protected areas of the products were mainly intact. Nevertheless, some products were degraded by the cross-cracks on whole surface – for example at the case of the lowest rolls' speed (Fig. 6).



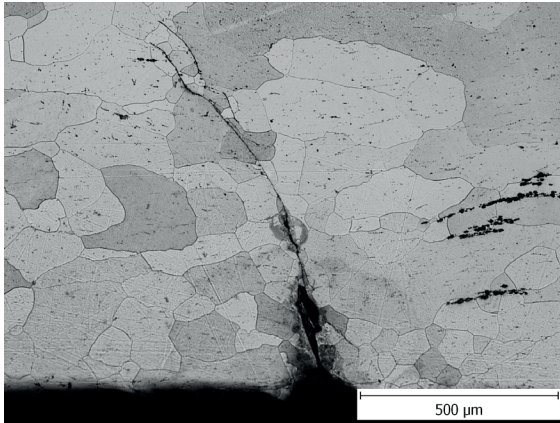
a) 80 rpm

b) 40 rpm

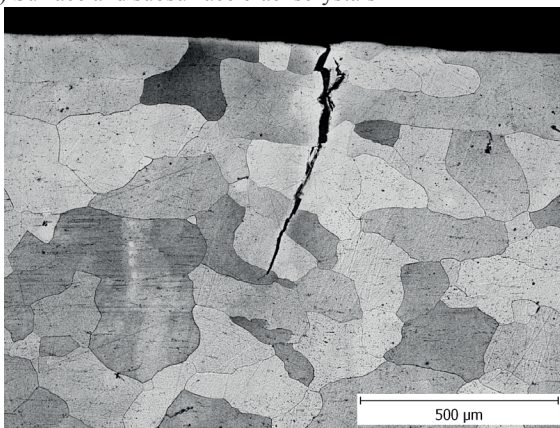
Fig. 6. The effect of rolls' speed on the fracture of product; rolling temperature was 1200°C



It was proved using metallography that the low real temperature of the material led to creation of cracks perpendicular to the rolling direction. Such conditions appear due to the low heating temperature prior to rolling (Fig. 7), or due to the low rolls' speed (Fig. 8).



a) Surface and subsurface cracks in crystals



b) Surface transverse crack

Fig. 7. The defects at the product rolled at 1160 °C with the rolls' speed 100 rpm

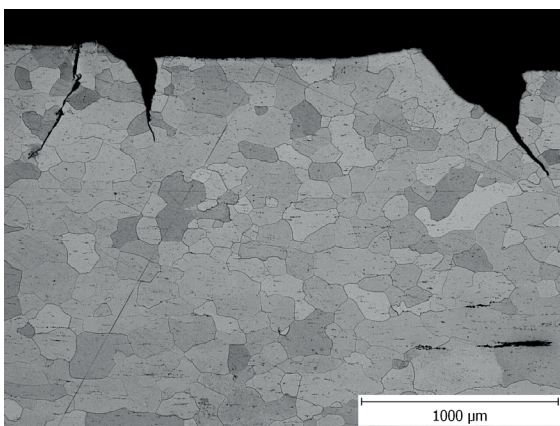


Fig. 8. Surface and subsurface cracks at the product rolled at 1200 °C with the rolls' speed 40 rpm

The surface quality of the products rolled with the highest rolls' speed was good, but internal defects (stretched cavities) appear at such conditions – see Fig. 9. These observations agree with results of the high rolls' speed plastometric experiments of Fe-40at.%Al alloy [15].

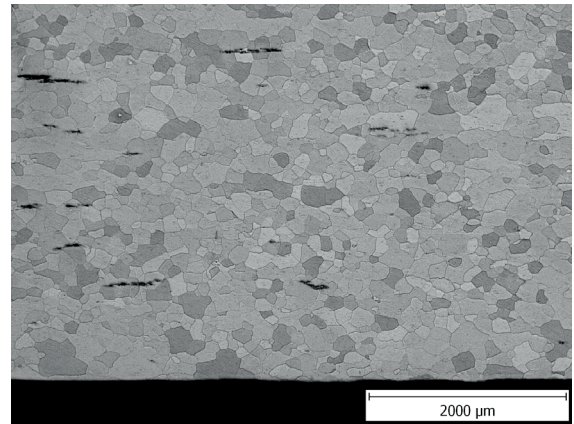


Fig. 9. The internal defects at the product rolled at 1200 °C with the rolls' speed 150 rpm

The best results give the application of both middle heating temperatures and rolls' speeds from investigated interval – see Fig. 10.

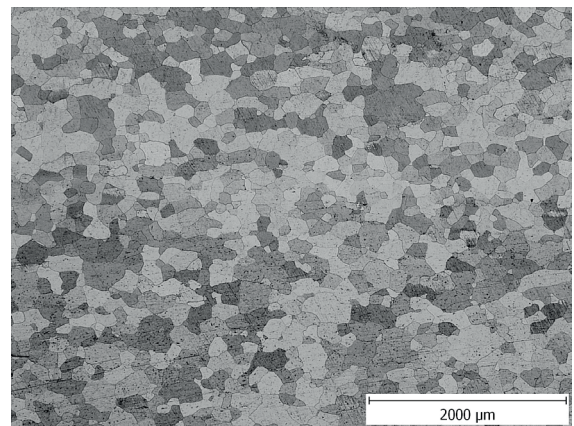
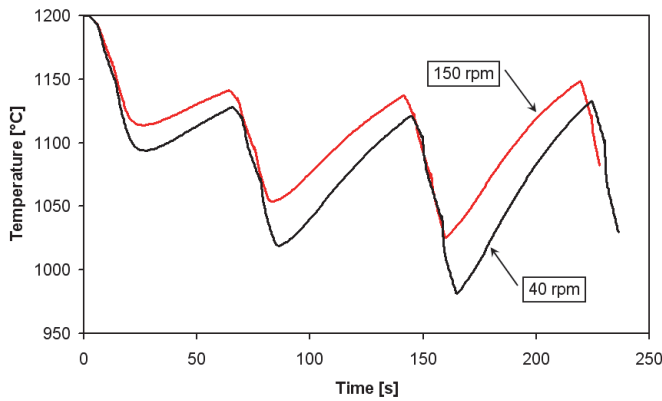


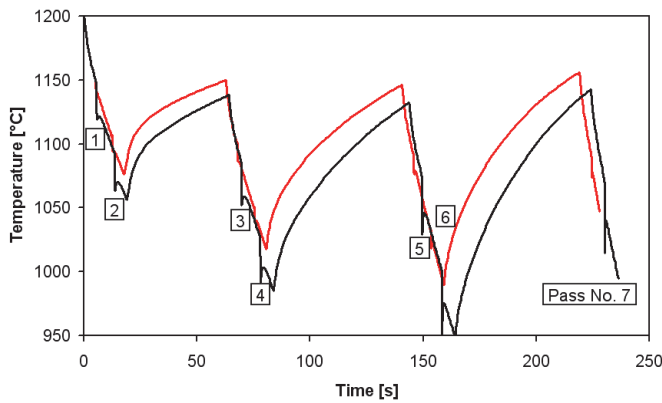
Fig. 10. The sample without any defects was rolled at 1200 °C with the rolls' speed 100 rpm

### 3.3 Temperature of the sample as the function of rolls' speed

The mathematical modelling using FEM was exploited to establish thermal history of the product, because the protective capsule prohibits direct measurement of the product's temperature. Program FORGE 3D was applied and the FEM simulation was performed for all 7 passes including heating and inter-heating in furnace at 1200°C for three selected rolls' speeds: 40 rpm, 100 rpm and 150 rpm. The data for the deformation history were taken from the detectors of rolling stand. The used rheological models were described by the equation designed by Hensel and Spittel [19]. The specific materials constants in this equation were obtained from the FORGE 3D database (i.e. steel X6Cr17 for the protective capsule) [20] or experimentally (for the iron aluminide Fe-40Al) [21, 22]. Selected results can be seen in Fig. 11.



a) The centre of the cross-cut of product



b) The edge of the surface of the product under the protective capsule

Fig. 11. The predicted time evolution of temperature at selected positions of the product during the rolling process

All pull-outs of relatively small sample from the heating furnace lead to quick decrease of the temperature, mainly in the surface layers. The FEM simulations confirm the presumption that the lower rolls' speed and thus even the longer contact time of the product with the cold working rolls lead to the decrease of temperature in order of tenth of degrees of Celsius. The intermediate heating in the furnace could not satisfy the raise of the temperature over the 1150°C. The decrease of the temperature in the middle of the product is from 108 up to 136°C for the rolls' speed 40 rpm, whereas just 84 up to 108°C for the rolls' speed 150 rpm, as can be seen from Fig. 11. Moreover the higher rolls' speeds produce even more deformation heating during the rolling.

The minimal immediate temperatures of the aluminide sample just in the deformation times are the key values from the formability point of view. The decrease of the temperature is 16°C for the rolls' speed 150 rpm, whereas 49°C for the rolls' speed 40 rpm at the last pass, when the product is the thinnest and its cooling is the most intensive. It means that decrease of the temperature is more than 3 times higher at rolls' speed 40 rpm. The temperature decreased at the edge to 1021°C for the rolls' speed 150 rpm, to 1011°C for the rolls' speed 100 rpm or even to non-acceptable 948°C for the rolls' speed 40 rpm during the 6th pass. Here we can find the explanation, why the products cracked even under the protective capsule at the lowest rolling speed, which seems to be in contrast with the general growth of the plasticity with decrease of stain rate,

as was found in plastometric testing with the heated working tools [15].

The temperature maps (see Fig. 12) show how effective is cooling of the product with cold working rolls and how the protective capsule prevents the unwanted surface temperature decrease of the rolled aluminide. The border of protective capsule is shown by thin black line.

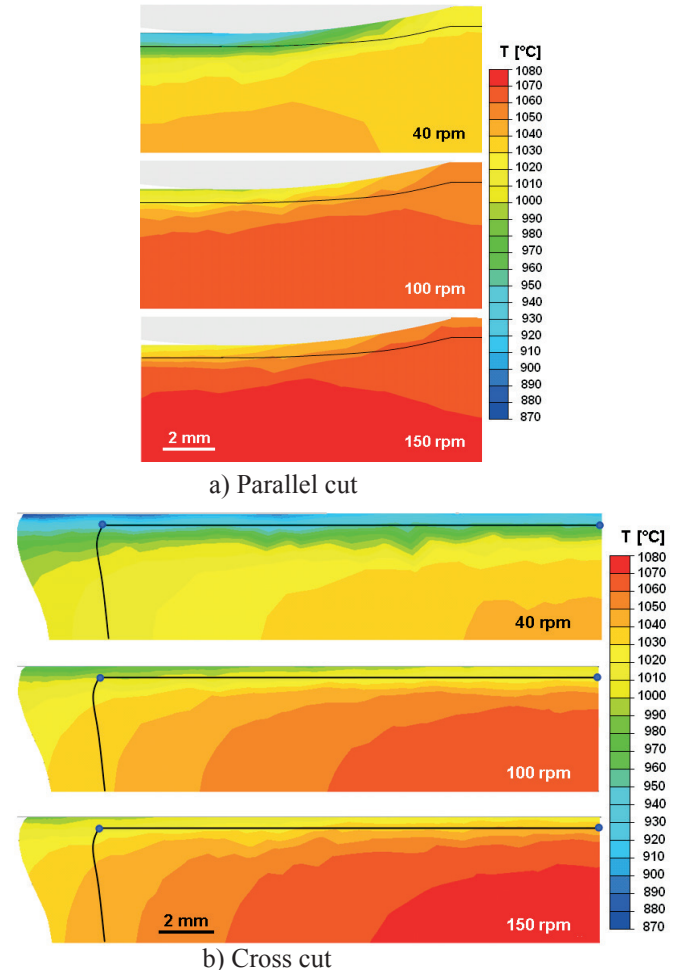


Fig. 12. The temperature maps just after the 6th pass calculated for various rolls' speeds

### 3.4 Microstructure of rolled products

The microstructure of the rolled products was investigated on the cross-cuts, which were prepared in the planes parallel (TD) or perpendicular (RD) to rolling direction. The effect of the rolling temperature can be seen in Fig. 13, and effect of rolls' speed in Fig. 14. Each of these figures gives complex data obtained by EBSD method. The columns give from the left the rolling temperature or rolls' speed, the structure orientation coded by inversion pole figure, grain size colour coded by area of the particular grain (explanation of both can be seen in Fig. 15) and pole figure calculated from these orientation maps.



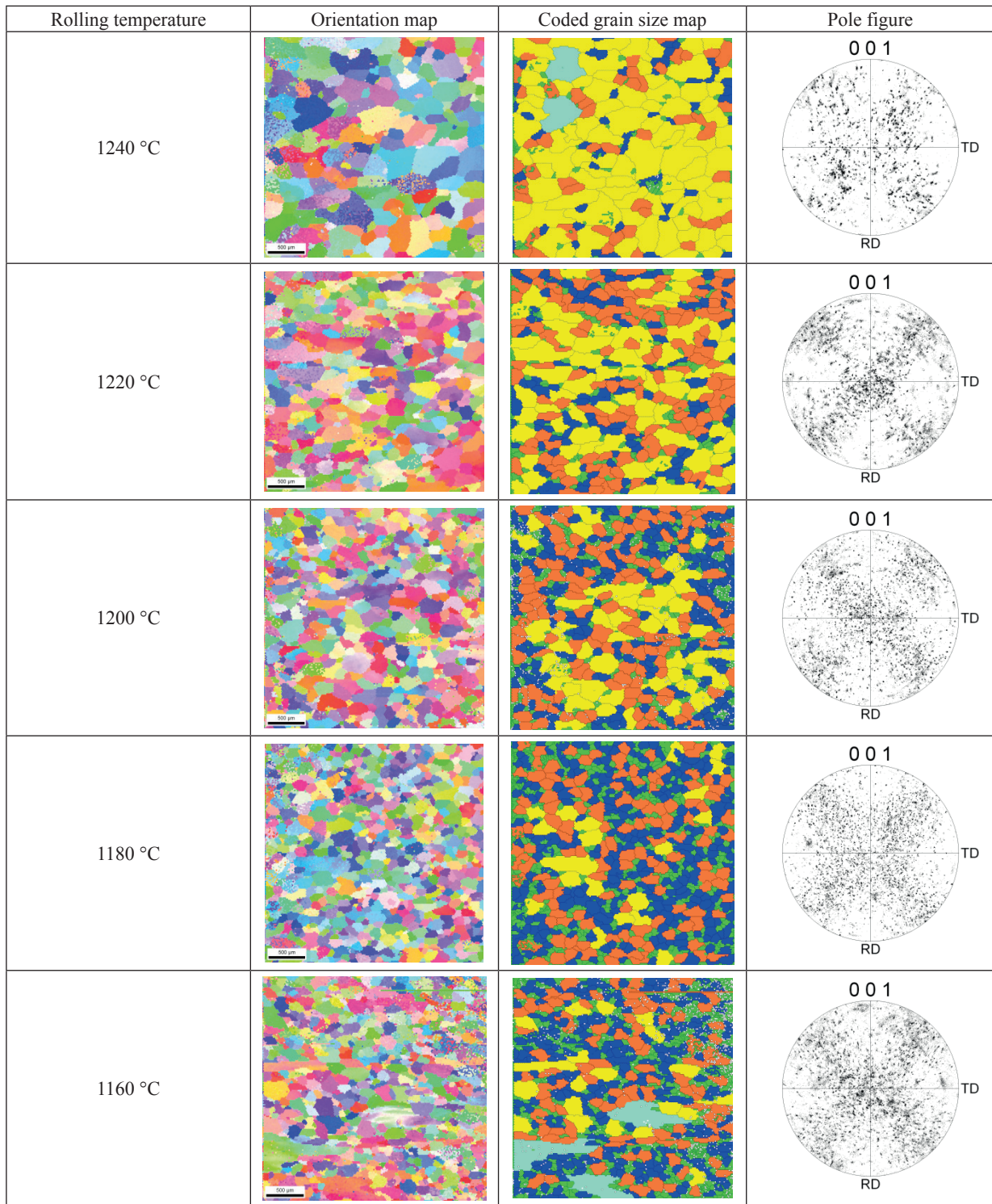


Fig. 13. The effect of temperature on the rolled product microstructure (the rolls' speed was 100 rpm for all samples)

TABLE 1

The grain size [ $\mu\text{m}$ ] for material rolled under various conditions.

Temperature / Rolls' speed	1240°C	1220°C	1200°C	1180°C	1160°C
40 rpm			180 ± 70		
80 rpm			190 ± 60		
100 rpm	290 ± 70	200 ± 70	170 ± 60	150 ± 50	170 ± 60
120 rpm			170 ± 60		
150 rpm			150 ± 50		

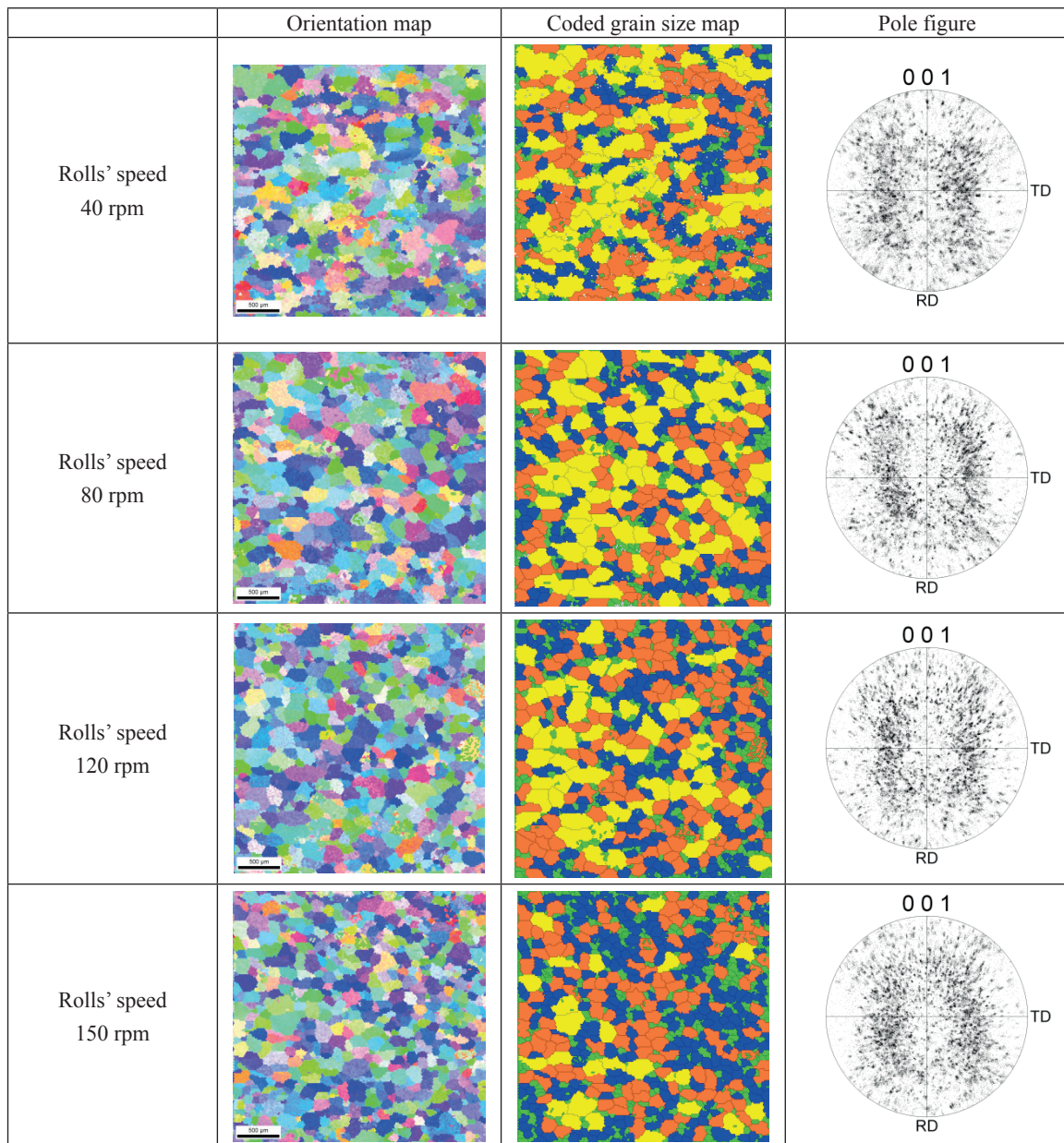


Fig. 14. The effect of rolls' speed for material heated to 1200°C (the material rolled with the rolls' speed 120 rpm at temperature 1200°C can be seen in Fig. 13)

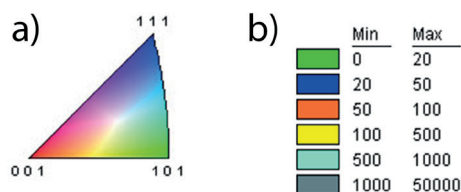


Fig. 15. The colour coded figures and maps

a) The colour coded inversion pole figure giving the colour for orientation maps in the columns of Figs. 13 and 14;

b) The colour coded grain size map (the number of point in particular grain is equivalent to grain area and the area of one point is due to mapping step 20 μm and hexagonal grid 200 μm<sup>2</sup>).

It can be seen from the orientation maps that the grain size decreases with the decreasing rolling temperature. Nevertheless, the calculated grain size has high standard error due to wide distribution of grain sizes – see Table 1. The colour coded grain size maps give the immediate view on the microstructure evolution as the grain size growth is seen easily (Fig. 13). The evolution of orientation maps with changing rolls' speed is even the less visible, but the colour coded grain size shows the trend of bigger grains for lower applied rolls' speed.

The pole figures at Fig. 13 – dependence of microstructure on rolling temperature – were prepared from cross sections of direction RD with respect to rolling notation, whereas the pole figures at Fig. 14 – dependence of microstructure on rolls' speed – were prepared from parallel sections of direction TD. Nevertheless, both types of pole figure describe the same texture, which remains strong after all applied conditions.



Applying the repeated static recrystallization during heating and intermediate heating, the homogeneous microstructure in whole volume of product was reached except the lowest heating temperature 1160°C. The equiaxed grains are about an order finer than in cast material – see Table 1.

Temperature of rolling has the major effect on microstructure, while the rolls' speed at separate temperature has the minor effect. The finest grains were obtained at rolling temperature 1180°C and rolls' speed 100 rpm –  $150 \pm 50 \mu\text{m}$ . The grain size was higher both for lower rolling temperature due to portion of unrecrystallized grains and even at higher rolling temperatures due to the longer time and thermal power for recrystallization. The variation of rolls' speed refines the microstructure just a slightly – from grain size  $180 \pm 70 \mu\text{m}$  for the rolls' speed 40 rpm down to  $150 \pm 50 \mu\text{m}$  for the rolls' speed 150 rpm.

#### 4. Summary

- Use of the protective steel capsules made it possible to manage the laboratory hot flat rolling of the extremely brittle as-cast aluminide Fe-40at.%Al-Zr-B with the total height reduction of almost 70 %. Rolling of the given material without the capsule lead to immediate formation of transversal cracks on the surfaces cooled by the work rolls, and that is why this procedure is not feasible at all.
- In the case of maintaining the sample's temperature at the sufficient level (e.g. by means of the intermediate heating) the investigated rolled material adequately softened thanks to the repeated static recrystallization and it bore the partial height reductions approaching 20 % without failure of cohesiveness, which is very promising from the practical point of view.
- The hot rolling parameters were optimized to obtain the best combination of deformation temperature and rolling speed. The resistance against cracking and refinement of the highly heterogeneous cast microstructure (with coarse grains even over 2 mm) were the main criteria. The deformation temperature was changed from 1160°C up to 1240°C and rolling speed from 0.14 m·s<sup>-1</sup> up to 0.51 m·s<sup>-1</sup>, which is equivalent to strain rate from 2.8 s<sup>-1</sup> up to 24.0 s<sup>-1</sup>.
- Both experiments and mathematical simulations based on FEM demonstrated that it is not possible to exploit enhanced plasticity of the investigated alloy at low strain rates in the hot rolling process. The heat flux from the sample to the working rolls is so intensive at rolling speed 0.14 m·s<sup>-1</sup> that even the protective capsule does not prevent massive appearance of the surface cracks perpendicular to rolling direction. It seems evident that the temperature of used material should not decrease below 1000°C during the rolling process.
- The homogeneity and size of product's grain was influenced significantly by temperature of deformation, whereas the effect of rolling speed was relatively negligible. The temperature 1160°C was too low for the full recrystallization, but the highest used temperature 1240°C caused too coarse final grains. The optimal forming parameters were found (combining the criteria of formability and final microstructure parameters) as rolling temperature 1200°C and the rolling speed 0.35 m·s<sup>-1</sup>. Such conditions satisfy the homogeneous microstructure of the product with the grain size  $170 \pm 60 \mu\text{m}$ .
- The effective technology of the iron aluminide Fe-40at.%Al-Zr-B preparation by simple processes of laboratory melting, casting and hot rolling was established. The advantage of this method is exploitation of traditional, cheap technological processes over the other sophisticated, but the more expensive technologies.

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