Conditions for the hot rolling of Fe_3Al-type iron aluminide

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Abstract

Conditions for optimization of the hot-rolling process of the iron aluminide Fe28.4Al4.1Cr-0.02Ce (at.%) were investigated. Parameters that must be controlled for rolling are temperature, strain and strain rate. All these quantities influence the deformation behaviour, which is described using a deformation resistance model and the observed structure and substructure originated during hot rolling.

K e y w o r d s: iron aluminides (based on Fe_3Al), optimized conditions for hot rolling, structure

1. Introduction

Iron aluminides possess excellent corrosion resistance against both oxidation and sulphidization even at high temperatures. Iron aluminides are applied as structural materials due to their low material costs (compared to corrosion resistant steels which contain Ni and/or Cr). It is very important to optimize technologies resulting in manufacturing of sheets that may be used as protective cladding on components exposed to corrosion environment.

Dynamic softening is the most important process, taking place during hot rolling. Experiments to determine the hot deformation resistance of iron aluminide were described by Hotař and Kratochvíl [1]. The experiment was designed to avoid damage to rolls during the preparation of sheets by hot rolling. These authors were able to determine equivalent mean stress using upsetting experiments at temperatures between 800 and 1300 ℃. Also Blackford et al. [2] studied the rolling of iron aluminide sheets at 800 and 1000 ℃ using 20% reductions. High temperature rolling was connected with dynamic recrystallization. Very interesting is cold rolling between 500 and 700 ℃ followed by the annealing procedures. This improved the strength but the ductility remained very small.

It is the purpose of the present paper to summarize the knowledge obtained during the laboratory hot rolling of an iron aluminide.

2. Experimental procedure

Mathematical models, which describe the deformation behaviour of the iron aluminide, were derived from laboratory test results obtained on the alloy having the composition: Fe-28.4Al-4.1Cr-0.2Mn-0.16C-0.02Ce (at.%). The main point was to describe the mean equivalent stress σ_{m} (MPa) and its dependence on logarithmic height strain ε_{h}, temperature T (℃) and mean strain rate γ (s^{-1}). Many authors have tried to develop such relationships involving the influence of temperature (exponential relationship), strain rate (power relationship) and influence of high strains or even dynamic softening to extend finally the range of applied strains in such models – see [3–6] for example. The present paper is based on laboratory rolling of flat samples within a range of thicknesses (4.0 mm, 4.6 mm, 5.4 mm and 6.5 mm, respectively), which were

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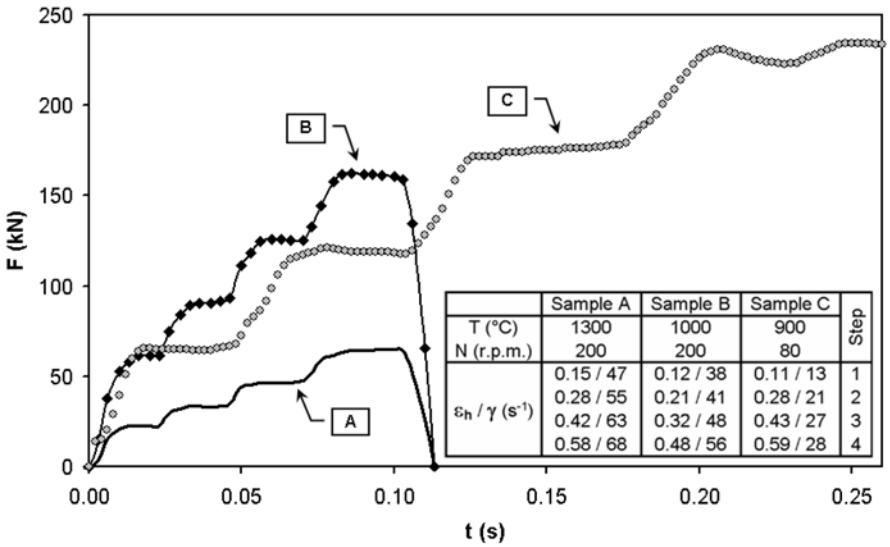

Fig. 1. Examples of the registered rolling force F depending on time t, deformation temperature T and nominal revolutions of rolls N.

used for determination of mean equivalent stress [7, 8]. These samples were machined from plates prepared by hot rolling between 1000 and 1200 ℃ from original ingot (thickness 38 mm) down to 8 mm. An advantage of using samples of 25 mm width and various initial thicknesses is far better data quality on rolling at exactly defined temperature as compared with rolling of one flat sample with constant thickness.

Each sample was heated in an electric resistance furnace to the forming temperature between 750 and 1300 ℃. Then it was immediately single-pass rolled in the Tandem [8] (diameter of plain work rolls 159 mm) laboratory mill with preset temperature, roll gap and nominal revolutions of rolls N (r.p.m.). Figure 1 represents examples of values of registered rolling force F (kN) as well as of the strain and strain rate corresponding to the individual steps of rolled samples. These values were calculated according to the equations

$$\varepsilon_{\mathrm{h}} = \ln \frac{H_0}{H_1}, \tag{1}$$

$$\gamma = \frac{2}{\sqrt{3}} \cdot \frac{v}{\sqrt{R \cdot (H_0 - H_1)}} \cdot \varepsilon_{\mathrm{h}}, \tag{2}$$

where H_0, H_1 (mm) are thicknesses of the sample before and after rolling, v (mm/s) is the peripheral velocity of rolls depending on the N-value [9].

The experiment included samples rolled at different strain rates in the range of 11 to 155 s^{-1} and height reductions in the range of 0.05 to 0.71. Mean equivalent stress σ_{m} (MPa) is calculated using the relation [10]

$$\sigma_{\mathrm{m}} = \frac{1000 \cdot F}{Q_{\mathrm{Fr}} \cdot \sqrt{R \cdot (H_0 - H_1)} \cdot B_{\mathrm{m}}}, \tag{3}$$

where Q_{Fr} is forming factor corresponding to the particular mill stand (for details see [7]), and B_{m} [mm] is mean width in the given place of the formed sample (the average of widths before and after rolling).

The structure of deformed samples was observed in the sections perpendicular to the rolling direction. This allowed studies of the softening processes during the rolling. Both optical (LOM) and transmission (TEM) microscopy were used: Nikon Epiphot 200 for LOM and JEOL 2000 FX for TEM. The preparation of samples for LOM was as follows: polishing using Struers OP-S solution and etching using Rollason (100 ml H_2O + 50 ml HCl 38% + 5 g Fe_3Cl), electrolytic polish in 20% solution of HNO_3 in metanol at −30 ℃ for TEM.

3. Results and discussion

At temperatures below 900 ℃ rolling forces increased so much that the applied laboratory equipment was not able to ensure safe rolling of all the length of samples with 4 height grades due to limited rolling force, torque and drive power. Thus the number of the obtained low-temperature σ_{m}-values is low. The transformation A2 (disordered ferrite) ⇔ B2 (ordered FeAl intermetallic) takes place in the vicinity of 900 ℃. It is supposed that this transformation is the reason for a sharp increase of deformation resistance. Values of mean equivalent stress at a temperature of 700 ℃ reached values higher than 1000 MPa, which corresponded to cold rather than hot forming. This is evident from the graph in Fig. 2, where all σ_{m}-values obtained from the measured rolling forces were plotted against deformation temperature without any reference to other deformation conditions (i.e.

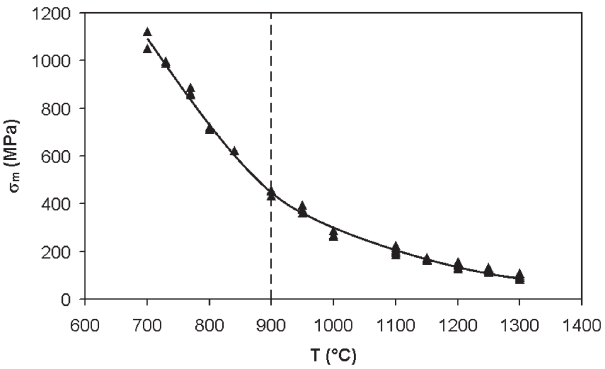

Fig. 2. Temperature dependence of the mean equivalent stress values calculated according to Eq. (3).

strain, strain rate), causing obvious scatter of the data.

In the course of mathematical processing of the results (using the multiple non-linear regression analysis with UNISTAT statistical software), it was shown that mean equivalent stress could not be described in the whole temperature range by a single relationship. For the temperature region 900 to 1300 °C the following equation was derived for calculation of the mean equivalent stress $\sigma_{\mathrm{m-c}}$ (MPa) values:

$$\sigma_{\mathrm{m-c}} = 11984 \cdot \varepsilon_{\mathrm{h}}^{0.10} \cdot \exp(-0.41 \cdot \varepsilon_{\mathrm{h}}) \cdot \gamma^{0.09} \cdot \cdot \exp(-0.00377 \cdot T). \quad (4)$$

Such an equation should reflect first of all the technological conditions for modern rolling mills, i.e. it has to meet requirements over a wide range of strains and temperatures, with special emphasis on high strain rates. It includes terms representing deformation hardening, $\varepsilon_{\mathrm{h}}^{0.10}$, as well as dynamic softening, $\exp(-0.41 \cdot \varepsilon_{\mathrm{h}})$.

The influence of dynamic softening and effect of strain rate was simplified [11] in comparison with some more complicated equations describing equivalent stress during hot forming. This is possible due to the fact that the equation has been derived using mean values of deformation resistance and thus is less sensitive to changes of strain as an independent variable.

In the case of low-temperature deformation the situation became more complicated due to a limited range of deformation (max. 0.28) and strain rate (max. 94 s^{-1}). A sufficient number of input data was not available to develop the partial regression relationships. For the low-temperature range the constants from the high-temperature equation were applied and thus only the temperature dependence of mean equivalent stress was investigated. Accordingly, for temperatures 700 to 900 °C the following equation was ob-

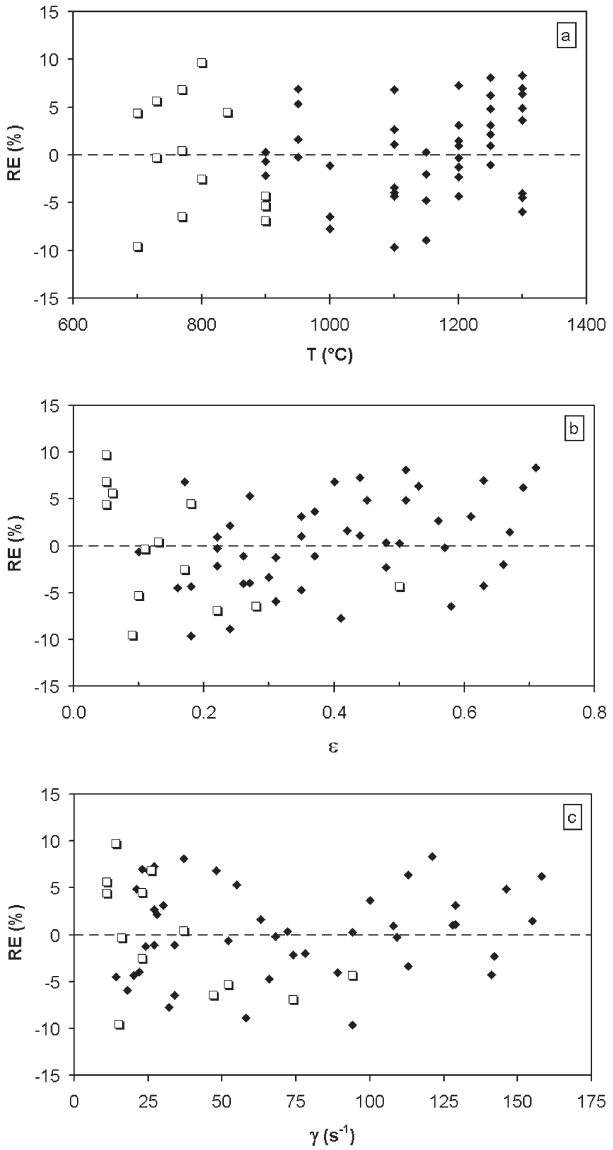

Fig. 3. Scatter of relative errors RE calculated according to Eqs. (4–6) in relation to temperature T, strain ε and strain rate γ.

tained:

$$\sigma_{\mathrm{m-c}} = 45778 \cdot \varepsilon_{\mathrm{h}}^{0.10} \cdot \exp(-0.41 \cdot \varepsilon_{\mathrm{h}}) \cdot \gamma^{0.09} \cdot \cdot \exp(-0.00521 \cdot T). \quad (5)$$

The accuracy of derived equations was evaluated by a relative error RE (%) according to the equation

$$\mathrm{RE} = \frac{\sigma_{\mathrm{m}} - \sigma_{\mathrm{m-c}}}{\sigma_{\mathrm{m}}} \cdot 100, \quad (6)$$

where σ_{m} values are experimentally obtained from the measured rolling forces (see Eq. (3)) and $\sigma_{\mathrm{m-c}}$ values are calculated for the analogous deformation

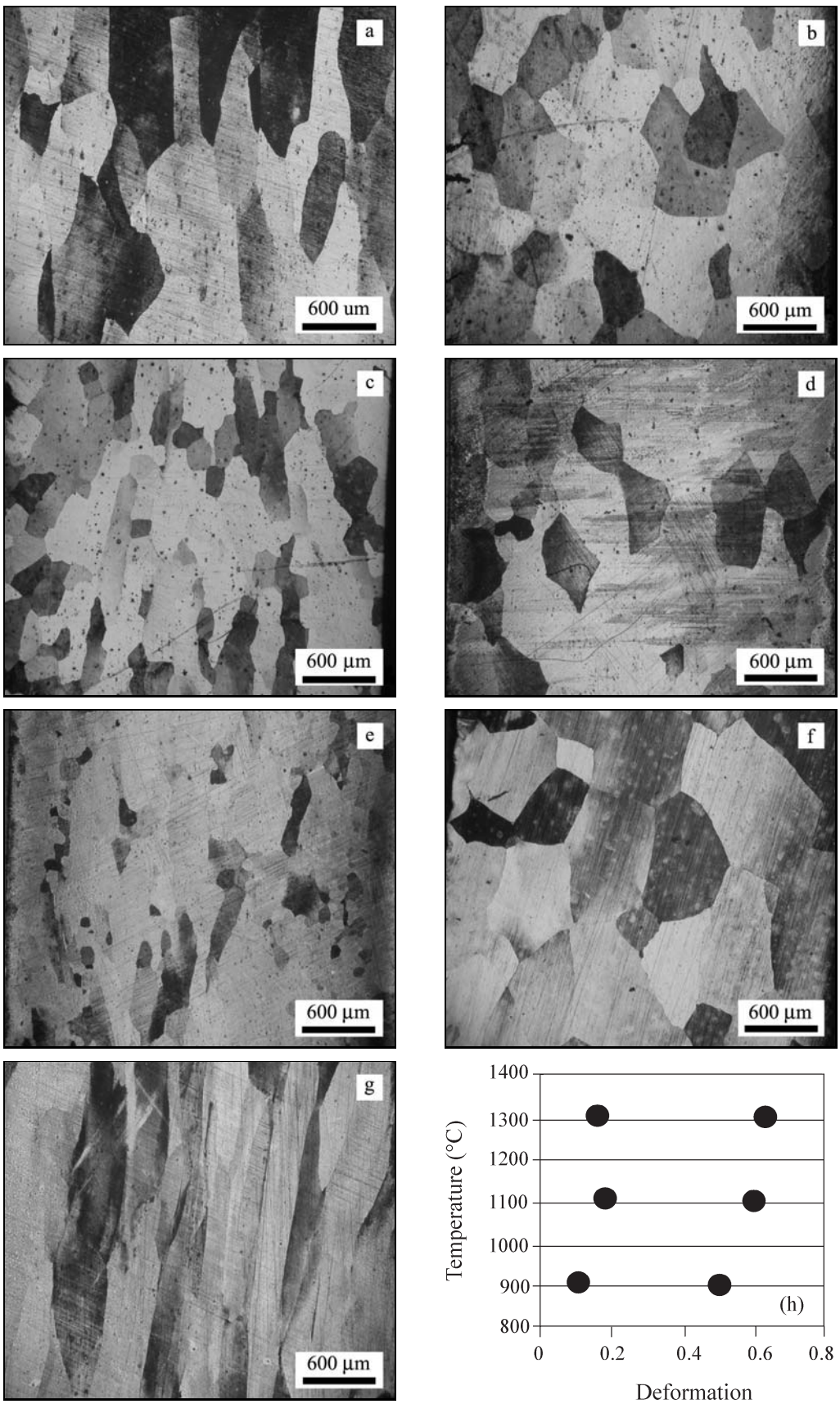

Fig. 4. Structures after different strains and temperatures typical for the present rolling experiment: (a) initial state, (b) rolled at 1300 °C, $\varepsilon_h = 0.16$, (c) rolled at 1300 °C, $\varepsilon_h = 0.63$, (d) rolled at 1100 °C, $\varepsilon_h = 0.27$, (e) rolled at 1100 °C, $\varepsilon_h = 0.59$, (f) rolled at 900 °C, $\varepsilon_h = 0.10$, (g) rolled at 900 °C, $\varepsilon_h = 0.50$.

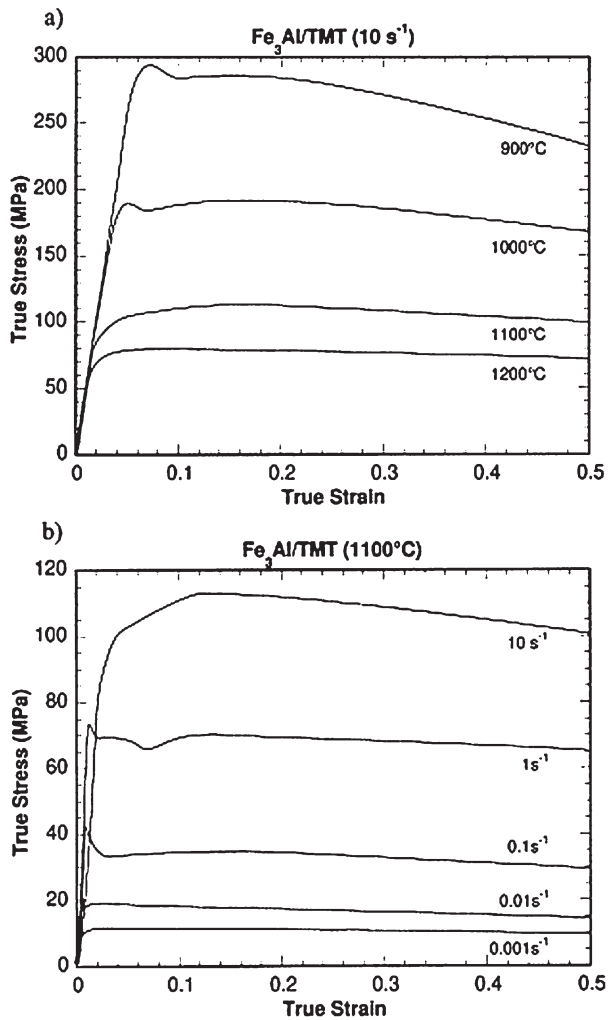

Fig. 5. The effect (a) of the deformation temperature (at strain rate 10 s^{-1}) and (b) of strain rate (at 1100 °C) on the true stress-true strain curves, according to [13].

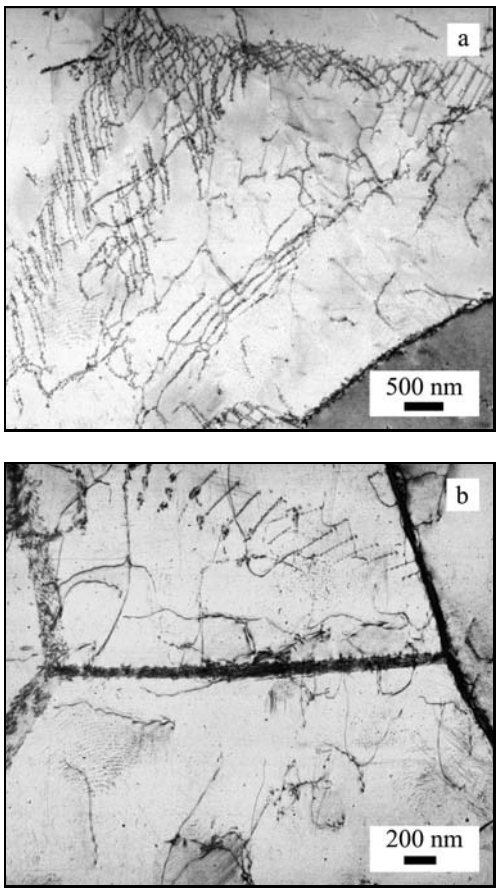

Fig. 6. The configuration of dislocations after rolling at 1100 °C. The subboundaries formed by coupled superdislocations with little dislocations inside subgrains are visible.

conditions using Eqs. (4) and (5). Both models describe pertinent relationships with very good accuracy as demonstrated by the graphs in Fig. 3. Deviations of relative errors in these graphs (□ for the low-temperature and ■ for the high-temperature equation) give very good results with limited scattering without evidence of any additional independent variable. Calculated *RE*-values do not exceed 10 % of the actual values of mean equivalent stress, which may be considered to be a very good result over wide range of deformation conditions applied.

The effect of rolling temperature and of the strain on the grain structure is obvious in Fig. 4. The samples before rolling are heated long enough so that the material may recrystallize before rolling depending on the temperature of the experiment. For deformations smaller than 0.2 – see Fig. 4 – no substantial change of the shape of the grains is observed. For higher deformations (approx. 0.6) there is an obvious effect on the grain structure. At 900 °C the grain elongates; at 1100 °C the nucleation of small recrystallized grains is visible; and finally at 1300 °C already well-developed recrystallized grains appear. This corresponds very well to the type of the deformation curves observed on examining a similar material [11]. The dynamic resistance observed in our experiments is very well comparable with stress data by Voyzelle and Boyed [12] (see Fig. 5) for similar material (niobium as an additive to the base Fe28Al4Cr alloy). Even better agreement is obtained with Prasad et al. [13], who examined powder metallurgy material at temperatures at 900 and 1150 °C at stresses of approximately 400 MPa and 150 MPa respectively for strain rates of 10 up to 100 s^{-1}. The typical influence of temperature and strain rate on the shape of the stress-strain curve was obvious. Both types of stress-strain curves indicate that dynamic softening process takes place – either dynamic recrystallization or dynamic recovery.

This point of view is supported by the observation of dislocation substructure using transmission electron microscopy (TEM). Subgrains with a small density of dislocations inside them and subboundaries formed by

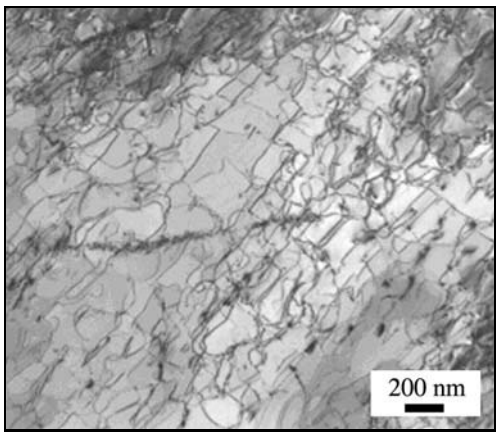

Fig. 7. Tangled configurations of dislocations after rolling at 900 ℃.

superdislocations of ⟨111⟩ type Burgers vector typical for the B2 ordered structure – Fig. 6 – are typical for rolling at 1100 and 1300 ℃. Such substructures are characteristic of dynamic recrystallization. The dislocation substructure in samples rolled at 750 and 900 ℃ is formed by unstable, unrelaxed tangled configurations (Fig. 7) typical of a limited amount of dynamic recovery.

Very recently Konrad et al. [14] had studied the hot rolling process using a hot deformation stimulator (WUMSI) in combination with data analysis. They concluded that it is possible to use high temperature deformation processes at high strain rates for rolling of Fe_3Al based alloys. The effect of ordering to the state B2 was also identified. These authors also concluded, that recrystallization took place from dynamically generated nuclei resulting in post-dynamic recrystallization. As here, these authors also identified a well-developed cell structure when rolling in the B2 order regime.

4. Conclusions

1. Based on the measurement of forces during laboratory hot rolling and the mathematical analysis of data, a high-temperature as well as a low-temperature equation describing the mean equivalent stress for the iron aluminide (Fe28.4Al4.1Cr0.02Ce at.%) was developed as a function of temperature, strain and strain rate. These equations are simple but precise enough over a wide range of strains to reflect both hardening and dynamic softening processes.

2. It was confirmed that rolling at high temperatures in the disordered A2 region is favourable due to pronounced dynamic recovery and recrystallization and thus low deformation resistance. The deformation resistance during hot rolling corresponds very well with stress values determined during hot tensile tests at the same temperature and strain rates.

3. Dislocations observed in the rolled samples correspond very well to the behaviour of the material during hot rolling at temperatures between 700 ℃ and 1300 ℃.

Acknowledgements

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References

[1] HOTAŘ, A.—KRATOCHVÍL, P.: Kovove Mater., *4*, 2002, p. 45.
[2] BLACKFORD, J. R.—BUCKLEY, R. A.—JONES, H.—SELLARS, C. M.—BRIGUET, C.—MORRIS, D. G.: Mater. Sci. Technol., *14*, 1998, p. 1132.
[3] MEDINA, F. C.—HERNANDEZ, C. A.: Mémoires et Études Scient. Revue de Métalurgie, *89*, 1992, p. 217.
[4] DAVENPORT, S. B.—SILK, N. J.—SPARKS, C. N.—SELLARS, C. M.: Mater. Sci. Technology, *16*, 2000, p. 539.
[5] SCHINDLER, I.—HADASIK, E.: J. Mater. Process Technology, *106*, 2000, p. 132.
[6] TAKUDA, H.—MORISHITA, T.—KINOSHITA, T. —SHIRAKAWA, N.: In: 13th International Scientific Conference on Achievements in Mechanical and Materials Engineering. Ed.: Dobrzanski, L. A. Gliwice, Silesian University of Technology 2005, p. 671.
[7] RUSZ, S.—SCHINDLER, I.—MAREK, M.—BOŘUTA, J.—KUBINA, T.: Acta Metallurgica Slovaca, *11*, 2005, p. 265.
[8] http://www.fmmi.vsb.cz/model/
[9] KREJNDLIN, N. N.: Calculation of pass reductions in the non-ferrous metals rolling. Moscow, Metallurgizdat 1963 (in Russian).
[10] YANAGIMOTO, J.—MORIMOTO, T.—KURAHASHI, R.—CHIKUSHI, I.: Steel Research, *73*, 2002, p. 56.
[11] SCHINDLER, I.—MAREK, M.—DÄNEMARK, J.: Hutnické listy, *57*, 2002, p. 34 (in Czech).
[12] VOYZELLE, B.—BOYED, J. D.: Mater. Sci Eng., *A258*, 199l, p. 243.
[13] PRASAD, Y. V. R. K.—SASTRY, D. H.—DEEVI, S. C.: Intermetallics, *8*, 2000, p. 1067.
[14] KONRAD, J.—ZAEFFERER. S.—SCHNEIDER, A. —RAABE, D.—FROMMAYER, G.: Intermetallics, *13*, 2005, p. 1304.