

Effect of cold/warm rolling following warm ECAP on superplastic properties of an Al-5.8%Mg-0.32%Sc alloy

E.V. Avtokratova[†], O.Sh. Sitdikov, M.V. Markushev

[†]avtokratova@imsp.ru

Institute for Metals Superplasticity Problems RAS, 39 Khalturin St., Ufa, 450001, Russia

Commercial extruded rod of 1570 aluminum alloy (Al-5.8%Mg-0.32%Sc) was subjected to equal channel angular pressing (ECAP) at a temperature of 325°C with an effective strain of ~8 and subsequent isothermal rolling at the same or ambient temperature up to strain of about 2.0 and 1.6, respectively. The mechanical behaviour in a wide temperature – strain rate range and consequent structure transformations were examined to evaluate an effect of the rolling temperature on the alloy superplastic response. It was found that the warm rolling significantly improved the homogeneity of the ultrafine-grained (UFG) structure formed by ECAP, resulting in near uniform UFG structure with 90-95% of grains having average size of about 1 μm. Such structure demonstrated high thermal stability and elongations to failure more than 2000% with the volume fraction of cavities not exceeding ~1.5% under tension at the strain rates of about 10⁻¹s⁻¹ in the temperature range of 450-475°C. In contrast the cold-rolled alloy provided much poorer superplastic behaviour with maximum elongations less than 350% due to low structural stability and intense cavitation. The nature of the alloy structure evolutions under static and dynamic annealing conditions, and superplastic parameters obtained in the as-ECAPed and warm / cold isothermally rolled alloys are discussed.

Keywords: aluminum alloy, severe plastic deformation, rolling, ultrafine-grained structure, superplasticity

Влияние теплого РКУП и последующей холодной/теплой прокатки на характеристики сверхпластичности сплава Al-5.8%Mg-0.32%Sc

Горячеэкструдированный пруток промышленного алюминиевого сплава 1570 (Al-5,8%Mg-0,32%Sc) был подвергнут равноканальному угловому прессованию (РКУП) при температуре 325°C до эффективной степени деформации ~ 8 и последующей изотермической прокатке, проводимой при той же или при комнатной температуре до степени деформации ~ 2,0 и ~ 1,6, соответственно. Механическое поведение сплава, а также соответствующие структурные изменения, изучали в широком интервале температур и скоростей деформации для того, чтобы оценить влияние температуры прокатки на характеристики сверхпластичности. Установлено, что теплая прокатка значительно повышает гомогенность ультрамелкозернистой (УМЗ) структуры, полученной РКУП, и ведет к формированию практически однородной УМЗ структуры с 90-95% зерен со средним размером около 1 мкм. Такая структура демонстрирует высокую термическую стабильность, обеспечивая удлинения до разрушения более 2000% с удельной долей пор не превышающей 1,5% при скорости деформации около 10⁻¹с⁻¹ в интервале температур 450-475°C. После холодной прокатки, напротив, характеристики сверхпластичности сплава снижаются из-за низкой стабильности структуры и интенсивного порообразования. Максимум удлинений в этом случае составил менее 350%. Эволюция структуры в условиях статического и динамического отжига, а также показатели сверхпластичности сплава, полученные после РКУП и последующей холодной/теплой прокатки обсуждаются в работе в деталях.

Ключевые слова: алюминиевый сплав, интенсивная пластическая деформация, прокатка, ультрамелкозернистая структура, сверхпластичность

1. Introduction

Non-heat-hardenable Al-Mg-Sc alloys with the high Mg content (> 4wt%) are very attractive structural materials for various kinds of applications owing to their enhanced strength, as well as excellent corrosion resistance and weld-

ability [1]. However these alloys are categorized as the hard-to-deform materials because of their high strain hardening and low plasticity at ambient temperature [1,2]. Therefore, an effective way for fabricating products from such alloys is the usage of structural superplasticity.

Grain refinement of commercial-base Al alloys to an ul-

trafine grained (UFG) scale level (grain size $<1\ \mu\text{m}$) is one of the major interests in a case, when superplastic forming is involved to manufacturing the net-shape parts. Equal-channel angular pressing (ECAP) is one of powerful metalworking techniques capable of producing such UFG structures by means of severe plastic deformation with minimum changes in shape and dimensions of working material and strong enhancement of its mechanical and physical properties [3–9]. In particular, the UFG Al-Mg-Sc alloys obtained by ECAP in [7] have shown unique superplastic behavior and elongations at both low temperatures and high strain rates.

The ECAP is commonly used to manufacture UFG rod or plate — shape billets. However, such products cannot be applied directly in superplastic forming without additional shaping operations, such as rolling. In this way, investigations of the influence of rolling on superplastic behavior of ECAP processed materials are important. As for Al-Mg-Sc alloys the data on superplastic behavior of UFG ECAPed and rolled products are quite poor, especially for high alloyed materials.

The present work is dedicated to examine the effects of two types of post-ECAP rolling — warm isothermal and cold (room temperature) ones (WR and CR, respectively), on the structure and superplastic properties of one of the alloys of Al-Mg-Sc system with Mg contents more than 4%.

2. Material and procedure

The commercial 1570 alloy having standard chemical composition (Al-5.8 Mg-0.32 Sc-0.4 Mn-0.2 Si-0.1 Fe (in wt.%) was direct chill casted and solution treated at 520°C for 24 h. Further direct extrusion was performed at 390°C to a strain of about 0.7, followed by one-hour annealing at 400°C . Plates for ECAP were machined parallel to the extrusion axis and subjected to warm ECAP at 325°C to a total effective strain of ~ 8 by route Bcz (rotation by 90° around the normal axis to the plate plane between passes) using a die with rectangular cross-section and channel inner angle of 90° . The ECAP temperature of 325°C ($\sim 0.6T_m$) was chosen since it was well documented in the previous works [10,11] as a most suitable for Al-Mg-Sc(-Zr) alloys with Mg content $\sim 5\text{-}6\%$ to form UFG structures with large fractions of high angle boundaries.

Samples for rolling were machined from the as-ECAPed billets. The warm rolling was carried out under isothermal

conditions (at a constant temperature of a sample and rolls) at 325°C with total reduction of 87% ($\epsilon\sim 2$), and the cold rolling was performed at ambient temperature with a total reduction of 80% ($\epsilon\sim 1.6$). In both cases, the rolling direction (RD) coincided with the last pressing direction. Microstructure was examined by optical and transmission electron microscopy (OM and TEM) (see more details in [11]). Tensile tests were carried out on Instron 1185 in the temperature range of $350\text{--}520^\circ\text{C}$ under constant crosshead speed using specimens with a gauge part $3\times 6\ \text{mm}^2$ machined along the RD.

3. Results and discussions

Microstructures after ECAP and rolling are represented in Fig. 1. After ECAP the alloy had a relatively uniform UFG structure (dark-color regions in Fig. 1a) with the volume fraction of ultrafine grains of $\sim 80\text{--}85\%$ and an average grain size of about $1\ \mu\text{m}$. Further warm rolling mainly resulted in increasing the homogeneity of structure (Fig. 1b): the fraction of ultrafine grains increased up to 90–95%, suggesting that additional grain refinement took place through transformation of remnant original grains (white regions in Figs. 1a and b). At the same time, the other parameters of the structure, e.g. the size and shape of ultrafine (sub)grains evolved during ECAP, remained unchanged. This may suggest that the main mechanism of plastic deformation in the UFG matrix under warm rolling was grain boundary sliding, which was attributed to the comparatively high rolling temperature [12].

Cold rolling, in contrast, led to pancake shape of both ultrafine and coarse remnant grains (omitted here) and formation inside them high-density dislocations arranged in a well-developed cellular substructure with the average cell size of $0.2\text{--}0.5\ \mu\text{m}$ (Fig. 1c).

Superplastic behavior. It is seen in Figs. 2 and 3 that superplastic characteristics of the alloy after ECAP and warm rolling are significantly higher than that after ECAP and cold rolling. For instance, tension of the warm rolled specimens with an initial strain rate, $\dot{\epsilon}$, of $1.4\times 10^{-3}\ \text{s}^{-1}$ showed the noticeable strain localization at 350 and 500°C only (Fig. 2a). At other temperatures a relatively uniform straining within the gauge part was observed and the maximum elongation of 2330% was found at 450°C (Fig. 3c). Besides, the warm rolled alloy demonstrated a well-defined maximum of

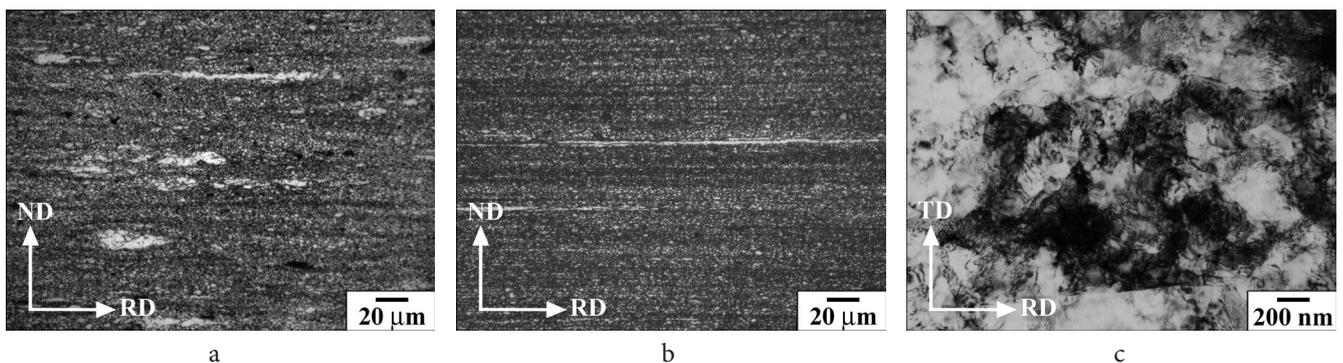


Fig. 1. OM (a, b) and TEM (c) structures of an Al-5.8Mg-0.32Sc alloy after ECAP (a), ECAP and warm rolling (b), ECAP and cold rolling (c). ND and TD - normal and transverse direction of the pressed and rolled samples, respectively.

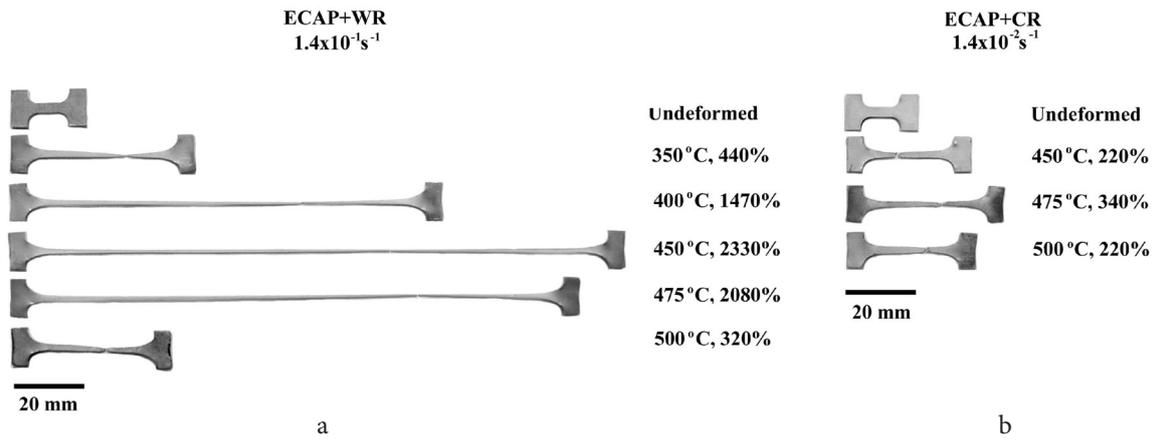


Fig. 2. An appearance of samples deformed up to failure at different temperatures: (a) ECAP+WR at $\dot{\epsilon} \sim 1.4 \times 10^{-1} \text{ s}^{-1}$ and (b) ECAP+CR at $\dot{\epsilon} \sim 1.4 \times 10^{-2} \text{ s}^{-1}$.

strain rate sensitivity coefficient, m , at 350°C and strain rate $\sim 10^{-2} \text{ s}^{-1}$ (Fig. 3a). At higher temperatures maximum m moved to higher strain rates and reached much higher values that were out of strain rate range tested in the present study. It should be noted that similar superplastic behavior was earlier observed in this alloy just after warm ECAP at the same temperature (325°C) [9], even though the authors had to apply a much higher total effective strain (~ 16) to form the UFG structure with the similar size and fraction of ultrafine grains. This allows concluding that such superplastic behavior may be typical for the ultrafine-grained Al-Mg-Sc alloy with high Mg content (4–6%) and high fraction of ultrafine grains, obtained at closed conditions, irrespectively on scheme of thermomechanical processing.

In the cold rolled state, in contrast, all tensile samples possessed quite earlier strain localization (Fig. 2b). Under straining at 450°C, the m value was less than 0.3 (Fig. 3b). Increase of deformation temperature resulted in m growth, reaching the value of 0.46 at 500°C and the strain rate $\sim 10^{-2} \text{ s}^{-1}$. However the maximum elongations to failure were obtained at 475°C and an initial strain rate of $1.4 \times 10^{-2} \text{ s}^{-1}$ and did not exceed 340% (Fig. 3c). It should be noted that a similar degradation of the superplastic properties was also observed after cold rolling of the ultrafine-grained Al-5.4Mg-0.5Mn-0.1Zr alloy

produced by ECAP [13]. At the same time it was reported that Al-3Mg-0.2Sc and Al-3.2Mg-0.13Sc alloys after ECAP and subsequent cold rolling demonstrated enhanced superplastic proprieties [14,15]. Thus no clear explanation has yet been offered how cold rolling of UFG Al-Mg-Sc(Zr) alloys with high Mg content (i ECAP and warm rolling remains essentially stable during static annealing. Only very limited normal grain growth from 1 to 1.5 μm was observed with increasing temperature up to 475°C. Such a high thermal stability of the UFG structure is obviously related to the high density of Al_3Sc dispersoids that are present in the aluminum matrix (Fig. 4b). A strong pinning effect of such nanosized precipitates restricts a drastic grain growth at elevated temperatures and provides high superplastic properties [7]. As it is seen in Fig. 4c, tension leads to a more significant dynamic grain growth, nevertheless the microstructure of the alloy remains fine-grained: after elongation to 2330%, the grain size was about 5.3 and 2.7 μm in tensile and transverse directions, respectively. At that the volume fraction of pores in the sample gauge near the fracture zone was relatively low ($\leq 2\%$) (Fig. 5a). Obviously, the low pore density was owing to a relatively slow and uniform grain growth upon dynamic annealing.

At that time it was found that the premature failure of

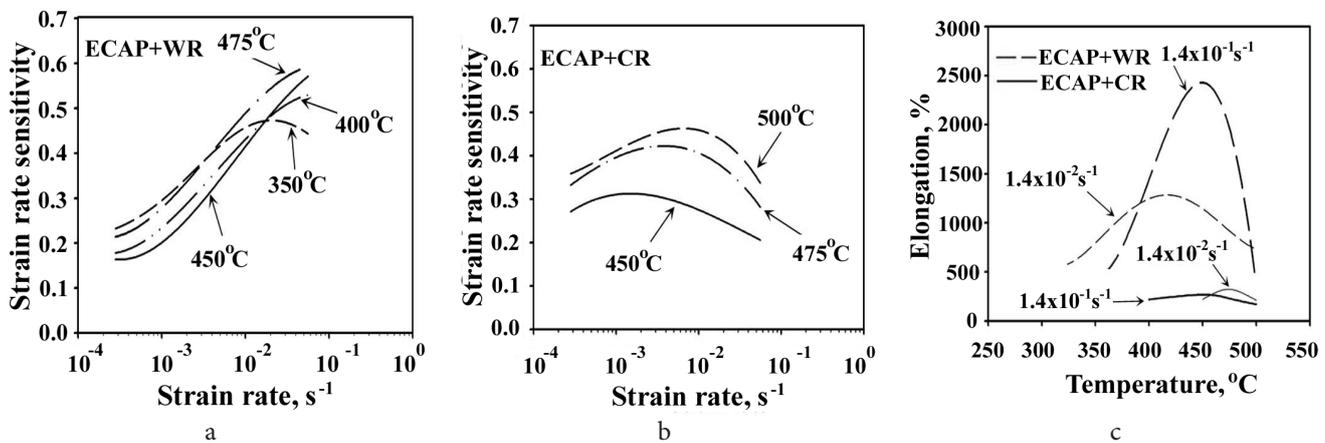


Fig. 3. The variation of strain rate sensitivity coefficient with strain rate (a, b) and elongation to failure with temperature (c) for the Al-5.8Mg-0.32Sc alloy processed through ECAP and subsequent warm rolling (WR) and cold rolling (CR).

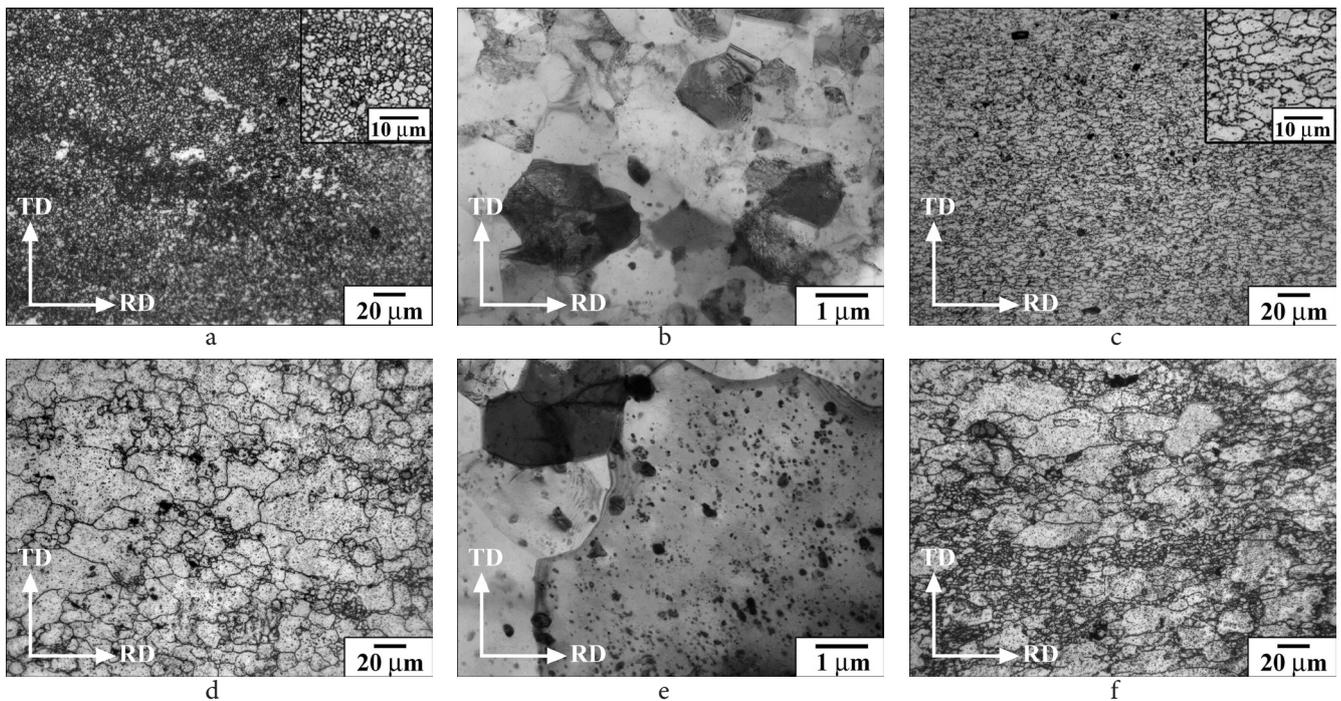


Fig. 4. Typical OM (a, c, d, f) and TEM (b, e) microstructures developed in the Al-5.8Mg-0.32Sc alloy after ECAP+WR (a-c) and ECAP+CR (d-f) upon tensile testing at $T=450^{\circ}\text{C}$: in the grip section by static annealing at exposure times, $t=0.38\text{h}$ (a,b) and 0.33h (d,e); in the gauge section by dynamic annealing at strain rates, $\xi\sim 1.4\times 10^{-1}\text{s}^{-1}$, $\delta=2330\%$ (c) and $\xi\sim 1.4\times 10^{-2}\text{s}^{-1}$, $\delta=220\%$ (f).

the specimens obtained by combination of ECAP and cold rolling was caused by the heterogeneity of the structure, appeared during early stages of both static and dynamic annealing (Fig 4d–f), resulting in instability of plastic flow. Quite fast abnormal grain growth up to $60\ \mu\text{m}$ was already found in the grip section of the specimen deformed at 450°C (Fig. 4d). The coarse grains were free from dislocations and contained the high density of incoherent compact Al_3Sc particles with size much larger than after warm rolling (see Figs. 4b and e). Simultaneously, fine grains were also observed in the gauge section after tension at this temperature, but their fraction did not exceed $\sim 20\%$ (Fig. 4f). Thus, it could be concluded that the alloy structure became heterogeneous after cold rolling following ECAP due to loss of coherency of Al_3Sc dispersoids resulting in a rapid deterioration of their thermal stability. Tension of the material with such non-uniform structure was accompanied by intense cavitation (Fig. 5b). So, the fraction of pores was $\sim 5.2\%$ at $T=475^{\circ}\text{C}$, $\xi\sim 1.4\times 10^{-2}\text{s}^{-1}$. Coalescence of pores occurred mainly in the

direction perpendicular to the tensile direction, leading to early sample failure.

4. Conclusions

1. ECAP of an Al-5.8Mg-0.32%Sc alloy at 325°C to $\varepsilon\sim 8$ resulted in formation of ultrafine-grained structure with the mean grain size of $\sim 1\ \mu\text{m}$ and the volume fraction of $\sim 80\text{--}85\%$.

2. Warm rolling at 325°C to $e\sim 2$ enhanced the uniformity of the microstructure produced by ECAP and provided formation of the ultrafine-grained microstructure with an average grain size of about $1\ \mu\text{m}$ $\sim 90\text{--}95\%$. The microstructure obtained exhibited very high thermal stability and resulted in superplastic ductilities more than 2000% in the temperature range of $450\text{--}475^{\circ}\text{C}$ at the strain rates of about 10^{-1}s^{-1} .

3. Cold rolling to $e\sim 1.6$ following ECAP resulted in a heavily deformed cellular structure with the cell size of $\sim 0.2\text{--}0.5\ \mu\text{m}$. Such structure exhibited low thermal stability and provided relatively weak superplastic properties. The maximum elongation to failure was less than 350% at all temperatures and strain rates investigated.

References

1. Y. A. Filatov, V.I. Yelagin, V.V. Zakharov, Mater. Sci. Eng. **A280**, 97 (2000).
2. T.G. Nieh, L.M. Hsiung, J. Wadsworth, R. Kaibyshev, Acta Mater. **46**, 2789 (1998).
3. Z. Horita, M. Furukawa, M. Nemoto, A.J. Barnes, T.G. Langdon, Acta Mater. **48**, 3633 (2000).

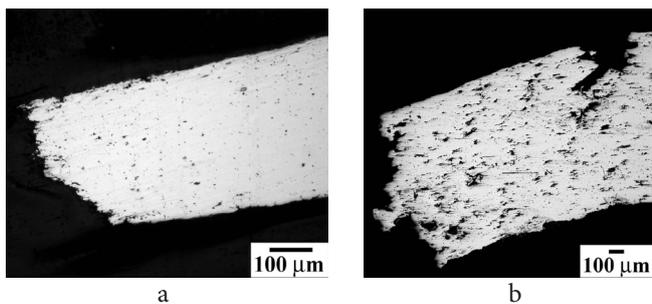


Fig. 5. Cavities near the the fracture zones of samples tested: ECAP+WR, $T= 450^{\circ}\text{C}$, $\xi\sim 1.4\times 10^{-1}\text{s}^{-1}$, $\delta=2330\%$ (a) and ECAP+CR, $T= 475^{\circ}\text{C}$, $\xi\sim 1.4\times 10^{-2}\text{s}^{-1}$, $\delta=340\%$ (b).

4. S. Lee, A. Utsunomiya, H. Akamatsu, K. Neishi, M. Furukawa, Z. Horita, T.G. Langdon, *Acta Mater.* **50**, 553 (2002).
5. S. Ferrasse, V. Segal, F. Alford, J. Kardokus, S. Strothers, *Mater. Sci. Eng. A.* **493**, 130 (2008).
6. M. V. Markushev, *Letters on materials.* **1**, 36 (2011).
7. E. Avtokratova, O. Sitdikov, M. Markushev, R. Mulyukov, *Mater. Sci. Eng. A.* **538**, 386 (2012).
8. E. Avtokratova, O. Sitdikov, O. Mukhametdinova, M. Markushev, *Mater. Sci. Forum.* **710**, 223 (2012).
9. F. Musin, R. Kaibyshev, Y. Motohashi, G. Itoh, *Met. Mat. Trans.* **35A**, 2383 (2004).
10. O.Sh. Sitdikov, E.V. Avtokratova, R.I. Babicheva, *Phys. Met. Metall.* **110**, 153 (2010).
11. O. Sitdikov, T. Sakai, E. Avtokratova, R. Kaibyshev, K. Tsuzaki, Y. Watanabe, *Acta Mater.* **56**, 821 (2008).
12. F.J. Humphreys, M. Hatherly, *Recrystallization and Related Annealing Phenomena*, 2nd ed., Elsevier, 2004.
13. S. Malopheyev, A. Kipelova, I. Nikulin, R. Kaibyshev, *Mater. Sci. Forum.* **667—669**, 815 (2011).
14. H. Akamatsu, T. Fujinami, Z. Horita, T.G. Langdon, *Scripta Mater.* **44**, 759 (2001).
15. K. Park, H. Lee, C. Lee, W. Nam, D. Shin, *Scripta Mater.* **51**, 479 (2004).