Influence of Overaging on the Superplastic Behavior of an Al-Zn-Mg-Cu-Zr-Sc Alloy

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This study examines the influence of overaging on the superplastic behavior of an Al-8.55Zn-2.65Mg-2.48Cu-0.1Zr-0.06Sc alloy (in mass%). Results show that the M-phase particles formed during overaging play the most crucial role during the high-temperature tensile deformation of the Al-Zn-Mg-Cu-Zr-Sc alloy. Even though specimens without overaging maintain their small grain structure due to the existence of fine coherent $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$ particles, overaged specimens exhibit better superplasticity. The presence of liquid-like viscous flow may be the main factor contributing to the superior superplasticity of overaged specimens. [doi:10.2320/matertrans.MRA2008197]

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1. Introduction

Superplastic forming is an attractive manufacturing process, and is widely used in numerous alloy systems. Superplastic forming of aluminum alloys allows the fabrication of complex sheet-metal components for automobile and aerospace applications. Superplastic forming requires small and stable grains which are usually less than 10 μm in diameter.¹⁾ In high-strength 7XXX Al alloys, various thermomechanical processes (TMP) have been developed to produce a fine grain structure. The TMP first proposed by Wert *et al.* for 7475 and 7075 alloys is the most famous of these processes, and is considered to be an effective grain refinement method for Al-Zn-Mg(-Cu) alloys.²⁾ A typical TMP involves solution treatment, overaging at 400°C, warm rolling, and annealing. During the overaging stage, coarse secondary particles, which are a type of Al-Zn-Mg(-Cu) compound known as M-phase, can form in the Al-Zn-Mg(-Cu) alloys. These particles act as nucleation sites for recrystallization, thereby refining the grain structure and enhancing the superplasticity of the Al-Zn-Mg(-Cu) alloys.

In traditional fine-grained Al-Mg alloys, superplasticity is limited due to significant grain growth during superplastic deformation.^{3,4)} Thus, it is important to find an effective grain stabilizing agent for superplastic Al-Mg alloys. Adding Sc as an alloying element is considered to be the most effective way to improve the properties of Al-Mg alloys. Further, the effects of Sc can be greatly amplified by Zr. Therefore, the high-temperature tensile behavior of Al-Mg alloys can be enhanced by adding Sc and Zr.^{5,6)} The extremely fine and coherent $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$ particles are more thermally stable than Al_3Sc or Al_3Zr particles at elevated temperatures,^{5,7)} which provides greater inhibition of grain growth at high temperatures.⁸⁾

Although many studies have examined superplastic Al-Zn-Mg(-Cu) alloys⁹⁻¹²⁾ and the effects of Sc and Zr on the high-temperature tensile behavior of Al-Mg alloys,^{5-8,13)}

few studies discuss the superplastic behavior of high-strength Al-Zn-Mg-Cu-Zr-Sc alloys. To address this gap, the current study examines the influences of overaging and $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$ particles on the high-temperature deformation behavior of Al-Zn-Mg-Cu-Zr-Sc alloys.

2. Experimental Procedures

Chung-Shan Institute of Science and Technology (Taiwan) supplied the material used in this study. This sample material had a chemical composition of 8.55Zn, 2.65Mg, 2.48Cu, 0.1Zr, 0.06Sc, 0.04Fe, 0.03Mn, 0.02Ti, 0.04Si, <0.01 Cr (mass%), with Al filling the balance. The alloy was received in an extruded form with a thickness of 12 mm. The TMP concept proposed by Wert *et al.*²⁾ was applied to this alloy. The alloy was first hot-rolled at 450°C to a thickness of 9 mm. Solution treatment was conducted in an air furnace at 470°C for 4 h followed by water quenching. Some specimens of the material were then overaged at 400°C for 8 h. Both types of specimens, with and without overaging, were finally warm-rolled at 200°C to a final thickness of 2 mm, representing a reduction of 77%. In this study, specimens with overaging are called TMP-A and specimens without overaging are called TMP-B. To more clearly and easily investigate the performance of $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$ and coarse secondary particles during the high-temperature tensile deformation of the Al-Zn-Mg-Cu-Zr-Sc alloy, this study omits the final high-temperature annealing in Wert *et al.*'s research.²⁾ This is because final high-temperature annealing would dissolve most of the coarse secondary particles in TMP-A in matrix.

Tensile specimens with a gauge length of 10 mm and cross section of $6 \times 2 \text{ mm}^2$ were machined from TMP-A and TMP-B sheets, with the loading axis parallel to the rolling direction. Tensile tests were conducted at temperatures between 300°C and 450°C at a strain rate range of 1×10^{-2} – $2 \times 10^{-4} \text{ s}^{-1}$. The highest working temperature 450°C was selected based on the DSC experiment that eutectic melting occurred above 485°C to avoid liquid formation. All high-

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Fig. 1 SEM microstructure of the Al-Zn-Mg-Cu-Zr-Sc alloy after: (a) solution treatment and (b) overaging; (c) and (d) shows EDS analysis result of the white particles in (a) and (b), respectively.

temperature tensile tests were conducted on a Hung-Ta HT8150 materials test system with a five-zone furnace operating at a constant crosshead displacement rate. The value of the strain rate sensitivity, m , was determined according to the following equation:

$$m = \frac{\partial \log \sigma}{\partial \log \dot{\epsilon}}$$

This study used optical microscopy (OM), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) to examine the microstructure of the Al-Zn-Mg-Cu-Zr-Sc alloy after high-temperature tensile tests. The samples chosen for TEM observations were prepared by mechanical grinding to a thickness of about 10 μm , and further thinned to a thickness of electron transparency using double-jet electro-polishing in a 25% nitric acid and 75% methanol solution operated at around -30°C and 30 V. Samples were examined on a JOEL-100CXII microscope operating at 100 kV.

3. Results

3.1 Microstructure of thermomechanically processed specimens

Figures 1(a) and (b) show the influence of overaging on the microstructure of the Al-Zn-Mg-Cu-Zr-Sc alloy. Figure 1(a) is the BSE image of the Al-Zn-Mg-Cu-Zr-Sc alloy after solution heat treatment. Some small white inclusions appear in the matrix, and most of them are $\text{Al}_7\text{Cu}_2\text{Fe}$ compounds (Fig. 1(c)). These $\text{Al}_7\text{Cu}_2\text{Fe}$ compounds are

insoluble by solution treatment, and usually appear in high-strength Al-Zn-Mg-Cu-Zr-Sc alloys.⁸⁾ Figure 1(b) is the BSE image of TMP-A after overaging treatment at 400°C for 8 h. Coarse secondary particles formed during overaging are present in the alloy, and can be identified as Al-Zn-Mg-Cu compounds (Fig. 1(d)). These coarse Al-Zn-Mg-Cu compounds, also known as M-phase, can introduce higher strain energy during warm-rolling and act as nucleation sites to refine grain structure during recrystallization.²⁾

Figures 2(a)–(d) show the TEM images of TMP-A and TMP-B in the as-rolled state. Both Fig. 2(a) and (b) show the typical highly deformed microstructure of Al alloys. Several coarse and small hollows appear in TMP-A (Fig. 2(a)). The interfaces between the M-phase particles and the matrix introduce a chemical potential difference that accelerate the dissolution of the interfaces. Therefore, the coarse hollows in TMP-A can be regarded as the original sites of coarse M-phase particles, and small hollows are the sites of smaller M-phase particles or $\text{Al}_7\text{Cu}_2\text{Fe}$ particles. Figure 2(b) shows that few hollows appear in TMP-B. Figures 2(c) and (d) are the corresponding TEM images of Figs. 2(a) and (b), respectively, at a higher magnification. Both small round M-phase particles and fine spherical $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$ particles appear in TMP-A and TMP-B in the as-rolled state. These small round M-phase particles mostly form during warm-rolling at 200°C , while the fine spherical $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$ particles mostly form during material making processes such as casting, homogenization, and extrusion. Both of these particles interact with dislocations during warm-rolling.

Fig. 2 TEM micrographs of (a) TMP-A and (b) TMP-B; (c) and (d) are the higher magnification of (a) and (b), respectively.

Fig. 3 Appearances of the optimal elongation at various temperatures of (a) TMP-A and (b) TMP-B.

3.2 High-temperature tensile behavior

Figures 3(a) and (b) show the optimal elongation at various temperatures for TMP-A and TMP-B, respectively. These figures show that TMP-A exhibits better ductility than TMP-B at temperatures ranging from 350–450°C. The high-temperature tensile behaviors of both TMP-A and TMP-B are not very outstanding. The maximum elongation in TMP-A was just 421% at 400°C and $5 \times 10^{-4} s^{-1}$. However, TMP-A achieved an acceptable elongation of 402% when deformed at 450°C and $1 \times 10^{-2} s^{-1}$. The strain rate $1 \times 10^{-2} s^{-1}$ is high, and an elongation of 402% is acceptable in common superplastic applications.¹⁴⁾ The barely acceptable ductility performance of TMP-A may be due to the elimination of the final recrystallization step used in Wert *et al.*'s study.²⁾ The alloy could not achieve the equiaxed grain structure essential to the sliding of the grain boundary. Figures 4(a)–

(d) show the true stress-strain curves of TMP-A and TMP-B specimens deformed at strain rates of $1 \times 10^{-2} s^{-1}$ and $5 \times 10^{-4} s^{-1}$ at various temperatures. At a higher strain rate of $1 \times 10^{-2} s^{-1}$ (Fig. 4(a) and (b)), TMP-A has a higher flow stress than TMP-B at temperatures ranging from 300–400°C, while at 450°C, TMP-A had a lower flow stress than TMP-B. In addition, at a lower strain rate of $5 \times 10^{-4} s^{-1}$ (Fig. 4(c) and (d)), the flow stress of TMP-A remained higher than TMP-B at 300°C, while the flow stress of TMP-A was lower than TMP-B above 350°C.

Figures 5(a) and (b) show the TEM images of the grain structures of TMP-A and TMP-B after high-temperature deformation at 450°C and $1 \times 10^{-2} s^{-1}$, and 400°C and $5 \times 10^{-3} s^{-1}$, respectively. Both specimens experienced approximately the same testing period, including 15 mins isothermal holding and nearly 7 mins deformation time, and

Fig. 4 True stress-strain curves of TMP-A and TMP-B tested at strain rates of $1 \times 10^{-2} \text{ s}^{-1}$ and $5 \times 10^{-4} \text{ s}^{-1}$ at various temperatures.

Fig. 5 TEM microstructure of (a) TMP-A after deformation at 450°C and $1 \times 10^{-2} \text{ s}^{-1}$ and (b) TMP-B after deformation at 400°C and $5 \times 10^{-3} \text{ s}^{-1}$.

showed a very small grain size of $3 \mu\text{m}$. Many small hollows appear in the grain boundaries in the TMP-A sample, while only few hollows appear in the TMP-B grain boundaries. The incompletely dissolved coarse M-phase particles remaining after high-temperature deformation generated these small hollows.

Fine and coherent spherical $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$ particles also appear in both TMP-A and TMP-B specimens after high-temperature deformation (Fig. 6). These particles are extremely small: 23 nm in TMP-A after deformation at 450°C and $1 \times 10^{-2} \text{ s}^{-1}$; 18 nm in TMP-B after deformation at 400°C and $5 \times 10^{-3} \text{ s}^{-1}$. The TMP-A and TMP-B specimens exhibit no great differences in the size and the density of $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$ particles. These particles had strong interaction with the dislocations (Fig. 6(b)) and the grain boundaries during deformation.¹³⁾ In TMP-A (Fig. 6(c)), both the coarse M-phase particles and the $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$ particles strongly interacted with the grain boundaries. On the other hand, the coarse secondary particles were absent from the

grain boundary interaction in TMP-B (Fig. 6(d)). The $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$ particles effectively maintained the fine grain structure in TMP-B (Fig. 5(b)).

3.3 Fracture behavior

Figures 7(a) and (b) show the SEM fractographs of TMP-A and TMP-B specimens after high-temperature tensile deformation at 400°C and $5 \times 10^{-4} \text{ s}^{-1}$. Both the TMP-A and TMP-B fracture modes were intergranular that the topography of the grains was clearly seen. Whiskers on the TMP-A fracture surface were also observed (Fig. 7(a)), whereas no whiskers were found in TMP-B (Fig. 7(b)). The TMP-A whisker formation at a strain rate of $5 \times 10^{-4} \text{ s}^{-1}$ occurred in a temperature range of $350\text{--}450^\circ\text{C}$, which is close to the overaging temperature of 400°C . Notice that TMP-A exhibits lower flow stress than TMP-B at a strain rate of $5 \times 10^{-4} \text{ s}^{-1}$ (Figs. 4(a) and (b)) at temperatures ranging from $350\text{--}450^\circ\text{C}$. These whiskers frequently appear in high-strength 7XXX aluminum alloys after high-temper-

Fig. 6 TEM micrographs of: (a) the $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$ particles and (b) the interaction between the $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$ particles and dislocations in TMP-B after deformation at 400°C and $5 \times 10^{-3} \text{ s}^{-1}$; the grain boundaries of (c) TMP-A after pulling to failure at 450°C and $1 \times 10^{-2} \text{ s}^{-1}$ and (d) TMP-B after deformation at 400°C and $5 \times 10^{-3} \text{ s}^{-1}$.

ature tensile deformation, and are thought to be evidence for a liquid-like viscous flow originating at the grain boundaries.¹²⁾ In addition, the grain size of TMP-B ($\sim 6 \mu\text{m}$) remained quite small after deformation, which is even smaller than TMP-A ($\sim 9 \mu\text{m}$). Though superplastic is feasible in small grains, typically less than $10 \mu\text{m}$, the high-temperature tensile behavior of TMP-B was quite poor. Figures 7(c) and (d) show the fractographs of TMP-A and TMP-B after deformation at 450°C and $1 \times 10^{-2} \text{ s}^{-1}$. In this case, TMP-A specimens had a flow stress lower than TMP-B. The topography of TMP-A grains was “wet”, which is also an evidence of the partial melting of M-phase particles. On the other hand, the topography of TMP-B was “dry”, which indicates typical ductile fracture characteristics.

Figures 8(a) and (b) show the OM images of the gauge sections near the fracture tip of TMP-A and TMP-B after deformation at 400°C and $5 \times 10^{-4} \text{ s}^{-1}$. These figures show that TMP-A endured more cavitations than TMP-B. Comparing to Fig. 7(a) and (b), even with the absence of coarse M-phase particles, the grains in TMP-B remain small, and even smaller than grains in TMP-A after deformation.

4. Discussion

4.1 High-temperature tensile behavior of overaged specimens

Coarse M-phase particles forming during the overaging process can serve as recrystallization nucleation sites, thereby refining the grain structure of Al-Zn-Mg(-Cu) alloys.⁶⁾ In addition, these particles appear to inhibit grain growth and

maintain a fine grain structure during high-temperature tensile deformation (Figs. 5(a) and 6(c)). Moreover, Fig. 7(a) and (c) show that adequate partial melting of the coarse M-phase particles can enhance solvent diffusivity and form a liquid-like diffusion flow.¹²⁾ This liquid-like diffusion flow lubricates the grain boundary during high-temperature deformation, and in turn, relaxes the stress concentration at the junction of the grain boundaries. Therefore, when tested at a lower strain rate of $5 \times 10^{-4} \text{ s}^{-1}$ (Fig. 4(c) and (d)), partial melting of the M-phases particles at $350\text{--}450^\circ\text{C}$ had sufficient time to let the solvent atoms diffuse. This in turn releases the stress concentration at the grain boundaries, and reduces the flow stress in TMP-A to be even lower than TMP-B. At a higher strain rate of $1 \times 10^{-2} \text{ s}^{-1}$, the liquid-like diffusion flow can only effectively offset the stress accumulation at grain boundaries when deformed at a high temperature of 450°C where diffusion is very fast, as a result, the flow stress of TMP-A was lower than TMP-B at temperature of 450°C (Figs. 4(a) and (b)). The reduction of flow stress in Fig. 4 may be evidence for strain energy releasing caused by the partial melting of coarse M-phase particles. Jiang *et al.* produced similar results, in that the strain hardening of overaged specimens was lower than that of non-overaged specimens.¹⁵⁾

Figures 8(a) and (b) show that the liquid-like diffusion flow may help withstand higher void coalescences during high-temperature tensile deformation.¹²⁾

4.2 High-temperature tensile behavior of non-overaged specimens

The combination of Sc and Zr additives causes the

Fig. 7 SEM fractographs of (a) TMP-A and (b) TMP-B after deformed at 400°C and $5 \times 10^{-3} \text{ s}^{-1}$; (c) TMP-A and (d) TMP-B after deformed at 450°C and $1 \times 10^{-2} \text{ s}^{-1}$.

Fig. 8 Gauge sections of (a) TMP-A and (b) TMP-B after deformation at 400°C and $5 \times 10^{-4} \text{ s}^{-1}$.

formation of $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$ particles with very attractive properties. These particles nucleate rapidly at high number densities, are homogeneously distributed, and coarsen quite slowly.^{16,17} These $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$ particles stabilize the grain structure of the alloy and prevent recrystallization after hot working by pinning grain and subgrain boundaries. In our previous study,¹³ we found that $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$ particles showed significant interaction with the dislocations and grain boundaries in a cold-rolled Al-6Mg-0.4Sc-0.13Zr alloy at temperatures of 400°C and 450°C. The Al-6Mg-0.4Sc-0.13Zr alloy resulted in poor ductility at 400°C and 450°C; it was not until the alloy was deformed at temperature above 500°C that the alloy exhibited excellent ductility of 1100%. The $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$ particles maintain the grain structure quite effectively at very high temperatures.¹³ However, without enough thermal activation, these particles act as heavily pinning nails, strongly impeding dislocation restoration and degrading grain boundary mobility during high-temperature deformation.¹³ The thermal activation energy in this study was not enough to help the dislocations and grain boundaries

overcome the super-high zener pinning pressure caused by the extremely fine $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$ particles at 400°C and 450°C. Without the partial melting of the coarse M-phase particles, the strain energy cannot be released and the diffusion is slow, resulting in poor grain boundary mobility. This may be the reason why TMP-B ductility is less than TMP-A even though the strong pinning $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$ particles at the grain boundaries effectively maintained the fine grain structure in TMP-B after high-temperature tensile deformation.

4.3 Strain rate sensitivity

Figures 9(a) and (b) plot the flow stress logarithm as a function of the strain rate logarithms of TMP-A and TMP-B, respectively. TMP-A exhibits higher strain rate sensitivities (m) than TMP-B at temperatures ranging from 350–450°C, e.g., at 350°C, $m_A = 0.54$ while $m_B = 0.31$; at 400°C, $m_A = 0.78$ while $m_B = 0.32$; at 450°C, $m_A = 0.70$ while $m_B = 0.33$. The high m values of TMP-A at temperatures from 350–450°C were in excess of 0.5, indicating that grain

Fig. 9 Logarithm of flow stress is plotted as a function of the logarithm of strain rate: (a) TMP-A; (b) TMP-B.

boundary sliding plays an important role. This implies that the high m value of TMP-A is also the consequence of partial melting of M-phase particles even though the small grains in TMP-B were maintained solely by $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$ particles during high-temperature tensile deformation.

With respect to superplastic deformation, $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$ particles can not only refine the grain structure of Al alloys⁸⁾ but can also prevent excessive grain growth at high-temperatures.^{5,13)} Though the small grain structure of TMP-B was effectively retained solely by extremely fine $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$ particles, and the grain structure of TMP-B could be smaller than TMP-A after high-temperature deformation, the elongation of TMP-B was poorer than TMP-A.

Although TMP-A overaging conducted at 400°C may be suitable for the precipitation and growth of $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$ particles,¹⁸⁾ the limited amount of Sc and Zr in the alloy restrict the coarsening kinetics of the $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$ particles. In addition, the insoluble property of these particles does not grow via Ostwald ripening mechanism.¹⁹⁾ Therefore, no significant coarsening of the $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$ particles occurs in TMP-A. Assuming that the small M-phases formed during warm-rolling rapidly dissolve in the matrix and the influences of the $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$ particles during deformation in both TMP-A and TMP-B are similar, the main difference in high-

temperature deformation between TMP-A and TMP-B is the existence of the coarse M-phase particles. The M-phase particles formed during overaging still play the most crucial role in enhancing the high-temperature ductility of Al-Zn-Mg-Cu-Zr-Sc alloys.

5. Conclusion

The coarse M-phase particles formed during overaging play the most crucial role in the high-temperature tensile deformation of the Al-Zn-Mg-Cu-Zr-Sc alloy. These particles partially melt during high-temperature deformation, which relaxes the stress concentration, assists grain boundary sliding, and endures more cavity coalescences. Coarse M-phase particles also lead to great flow stress reduction and higher strain rate sensitivities. Since no coarse M-phase particle melt during high-temperature tensile deformation in non-overaged specimens, these specimens exhibited less ductility than overaged specimens. This result occurred despite the fact that non-overaged specimens can also maintain their small grain size at high temperature due to the existence of $\text{Al}_3(\text{Sc}_x\text{Zr}_{1-x})$ particles effectively retarding the grain growth.

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REFERENCES

- 1) O. D. Sherby and J. Wadsworth: *Prog. Mater. Sci.* **33** (1989) 166.
- 2) J. A. Wert, N. E. Paton, C. H. Hamilton and M. W. Mahoney: *Metall. Trans.* **12A** (1982) 1981–1267.
- 3) A. Friedman and A. K. Ghosh: *Metall. Mater. Trans. A* **27** (1996) 3827–3839.
- 4) D. H. Bae and A. K. Ghosh: *Acta Mater.* **48** (2000) 1207–1224.
- 5) S. Lee, A. A. Utsunomiya, H. Akamatsu, K. Neishi, M. Furukawa, Z. Horita and T. G. Langdon: *Acta Mater.* **50** (2002) 553–564.
- 6) H. B. Geng, S. B. Kang and B. K. Min: *Mater. Sci. Eng. A* **373** (2004) 229–238.
- 7) S. Fujikawa: *J. Jap. Inst. Light Met.* **49** (1999) 128–144.
- 8) Z. Yin, Q. Pan, Y. Zhang and F. Jiang: *Mater. Sci. Eng. A* **280** (2000) 151–155.
- 9) R. Kaibyshev, T. Sakai, F. Musin, I. Nikulin and H. Miura: *Scr. Mater.* **45** (2001) 1373–1380.
- 10) Z. Y. Ma and R. S. Mishra: *Acta Mater.* **51** (2003) 3551–3569.
- 11) D. H. Shin, C. S. Lee and W. J. Kim: *Acta Mater.* **45** (1997) 5195–5202.
- 12) W. D. Cao, X. P. Lu and H. Conrad: *Acta Mater.* **44** (1996) 697–706.
- 13) Y. Y. Li, W. H. Wang, Y. F. Hsu and S. Trong: *Mater. Sci. Eng. A* **497** (2008) 10–17.
- 14) K. T. Park, D. Y. Hwang, Y. K. Lee, Y. K. Kim and D. H. Shin: *Mater. Sci. Eng. A* **341** (2003) 273–281.
- 15) X. G. Jiang, Q. Wu, J. Z. Cui and L. X. Ma: *J. Mater. Sci.* **28** (1993) 6035–6039.
- 16) V. G. Davydov, V. I. Elagin, V. V. Zakharov and D. Rostoval: *Met. Sci. Heat Treat.* **38** (1996) 347–352.
- 17) V. V. Zakharov: *Met. Sci. Heat Treat.* **39** (1997) 61–66.
- 18) J. Røyset and N. Ryum: *Inter. Mater. Rev.* **50** (2005) 19–44.
- 19) F. J. Humphreys and M. Hatherley: *Recrystallization and Related Annealing Phenomena*, (Oxford, Pergamon, 1995).