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Influence of plastic deformation on occurrence of discontinuous precipitation

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We have studied the effect of plastic deformation by compression on the occurrence of discontinuous precipitation in Al-30% Zn alloy after ageing at two different temperatures (348 and 423 K) has been studied. Optical microscope, scanning electron microscope, X-ray diffraction and differential scanning calorimetry were used for characterization.

During ageing of undeformed alloy, the grain boundary of supersaturated solid solution represents the favorite site of precipitation by discontinuous mechanism. We found that the occurrence of discontinuous precipitation depends mainly on the degree of plastic deformation before ageing. The grain boundary act as reaction front and migrates in case of low degree of prior deformation. © *2005 Springer Science + Business Media, Inc.*

1. Introduction

Discontinuous precipitation (DP) (or cellular reaction) is a solid state decomposition reaction that converts supersaturated solid solution α_0 into a two phase $\alpha + \beta$ aggregate behind a migrating reaction front [1, 2]: $\alpha_0 \rightarrow \alpha + \beta$

The cellular reaction originates at high angle grain boundaries by the formation of stable β-plate enriched in one of solute elements [2]. The relationship between boundary structure, energy, mobility and diffusivity is complex, it is generally accepted that high angle incoherent boundaries are the most likely candidates for DP reactions fronts [2, 3].

The absence of DP on twin and other boundaries with a rational orientation relationship and low energy habit phase is attributed to either a perfect/near-perfect coincidence relationship [4] or poor mobility (due to low step density) of the low angle boundary concerned [5]. Precipitation in a supersaturated alloy is known to be altered considerably by a plastic deformation after solution-annealing and before ageing. This effect has been investigated particularly in several aluminum alloys [6–10]. It is known that prior plastic deformation may either enhance or diminish the discontinuous precipitation [2].

It is reported that prior deformation enhances the DP kinetics in Cu-In [11], Ni-based superalloys [12], Pb-Na [13] and Nb-Zr-Ti [14]. On the other hand, entirely the opposite effect has been observed in Cu-Be [15], Cu-Ni-Mn [16], and Al-Li [2]. A high matrix dislocation density as a consequence of prior straining may enhance continuous precipitation and thereby reduces the driving force for DP [17]. On the other hand, Pawlowski [18] has proposed that the mode of deformation is critical to determine whether DP kinetics are enhanced or decreased.

The aim of the present paper is the study of the effect of plastic deformation by compression on the occurrence of discontinuous precipitation. Two aging temperatures |settings have been investigated (348 and 423 K).

2. Experimental procedure

The alloy used for this study was aluminum alloy containing 30 wt% of zinc prepared by melting from high purity components. The cast alloy was homogenized during seven days at 703 K and quenched in iced water. Samples of $8 \times 8 \times 9$ mm^3 were cut and encapsulated in Pyrex tubes under vacuum, annealed for 3 hours at 703 K and quenched in iced water to obtain a supersaturated solid solution.

To study the influence of deformation upon DP during two aging temperatures (348 and 423 K), the quenched samples were cold-worked by compression from 8 to 70% thickness reduction. The treatment conditions were obtained from the existent literature on the discontinuous reaction and from the equilibrium phase diagram of the Al-Zn system [19–24].

Studies of the mechanisms of precipitation were followed mainly by optical microscope (OM). Scanning electron microscope (SEM), X-ray diffraction experiments using Cu$_{K\alpha}$ radiation, and differential scanning calorimetry (DSC) were used as complementary technics. The specimens were etched with solution of 10 ml HF (53%) + 30 ml Glycerin + 10 ml Nital, in order to observe them in OM and SEM.

3. Results and discussion

All specimens, i.e., deformed and undeformed were aged isothermally at two temperatures (348 and 423 K).

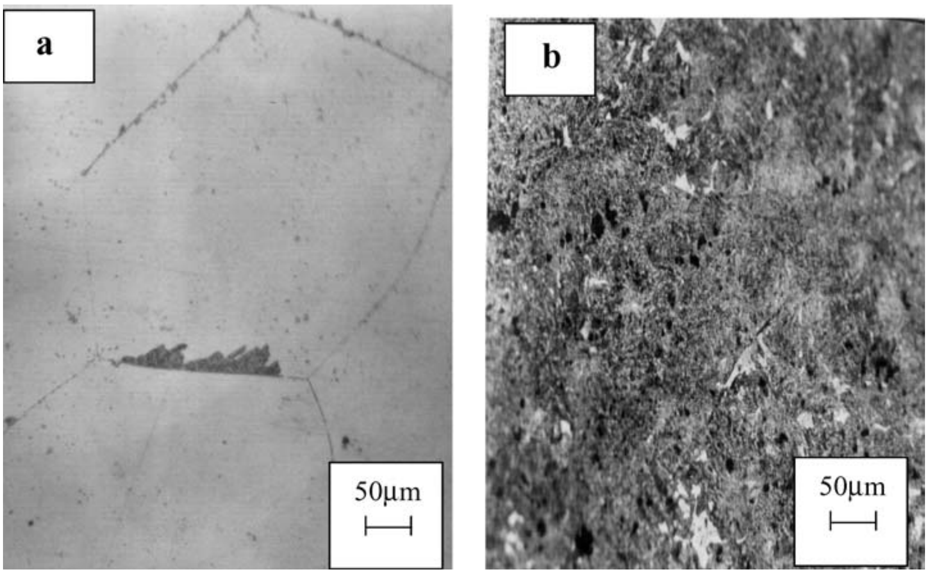

Figure 1 Microstructural evolution of Al-30 wt% Zn alloy during aging at 348 K for 5 h (a) and 72 h (b).

3.1. Ageing at 348 K

The results of ageing at 348 K are presented in Figs 1–4. The relationship between microstructure and the different amounts of compressive strain are shown. Examination of Fig. 1 of structural evolution of undeformed alloy, shows that the precipitate appears at grain boundaries by DP (Fig. 1a), and after prolonged aging time, discontinuous precipitates occupied the entire volume of alloy (Fig. 1b).

The structural evolution during ageing of the same areas of deformed alloys is shown by OM Figs 2 and 3, we note the formation of deformation bands. The grain boundaries remain at same location during ageing treatment. On the other hand, after prolonging the ageing time, for reduction greater than 40%, we observe a formation of finer precipitates on these deformation bands (Fig. 3b and c). It has been reported that the band itself consists of small irregular subgrains boundaries showing high misorientation [25]. It is known that increasing the degree of cold work leads to the formation of deformation bands which represent areas with locally high dislocation density. Consequently, during ageing these areas represent sites favoring continuous precipitation. The nucleation within deformation bands has been observed in several metals [26, 27]. For our case, after cold work of more than 40%, no DP is formed, i.e., no migrating boundary is developed. The same result has been obtained by Tsubakino [28] in Cu-Sn. Hamana and Choutri [29] have found that the DP rate is decreased by small amount of prior plastic deformation in Cu-Ag and Cu-Sb. One possible explanation for the absence of DP after critical deformation (40%) is increasing a degree of cold work leads to the formation of deformation bands. Therefore, the cellular precipitation cannot develop in these areas.

It is known that localized deformation may confine the plastic strain within a small volume and leads to the DP [17]. For instance, it has been shown that prior localized strain by hardness indentation or scratching on the external surface of single crystal or polycrystal may initiate DP from the deformed plastic zone in Cu-Ag [30–32] and Ni-Sn and Ni-In [33]. The opposite result is obtained in our study, i.e., that prior localized strain in Al-30 wt% Zn alloy applied by hardness indentation is not capable of initiating the DP (Fig. 4), but on the other hand, this DP reaction develops normally at grains boundaries.

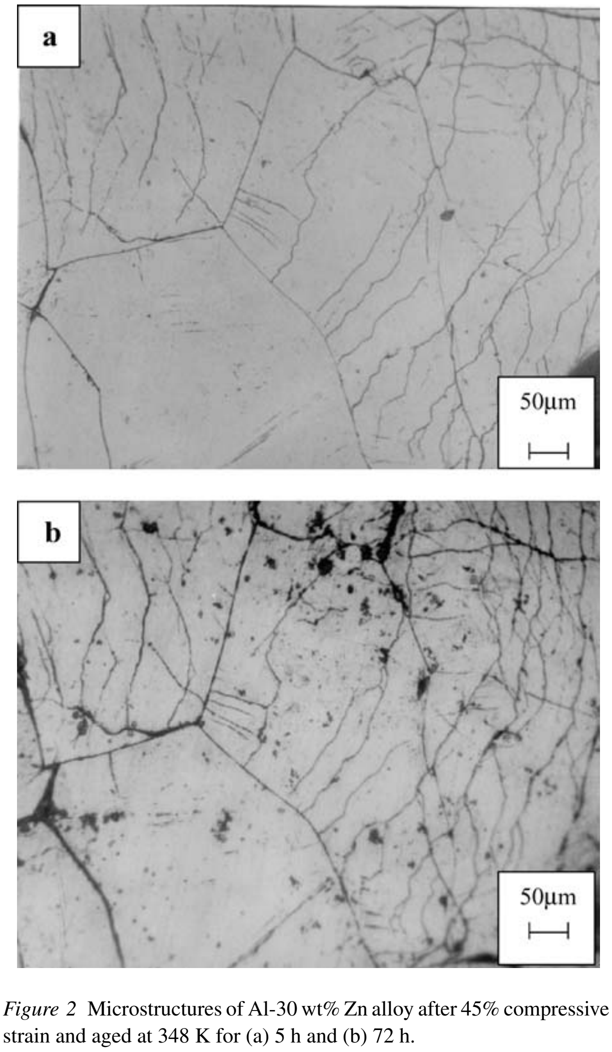

Figure 2 Microstructures of Al-30 wt% Zn alloy after 45% compressive strain and aged at 348 K for (a) 5 h and (b) 72 h.

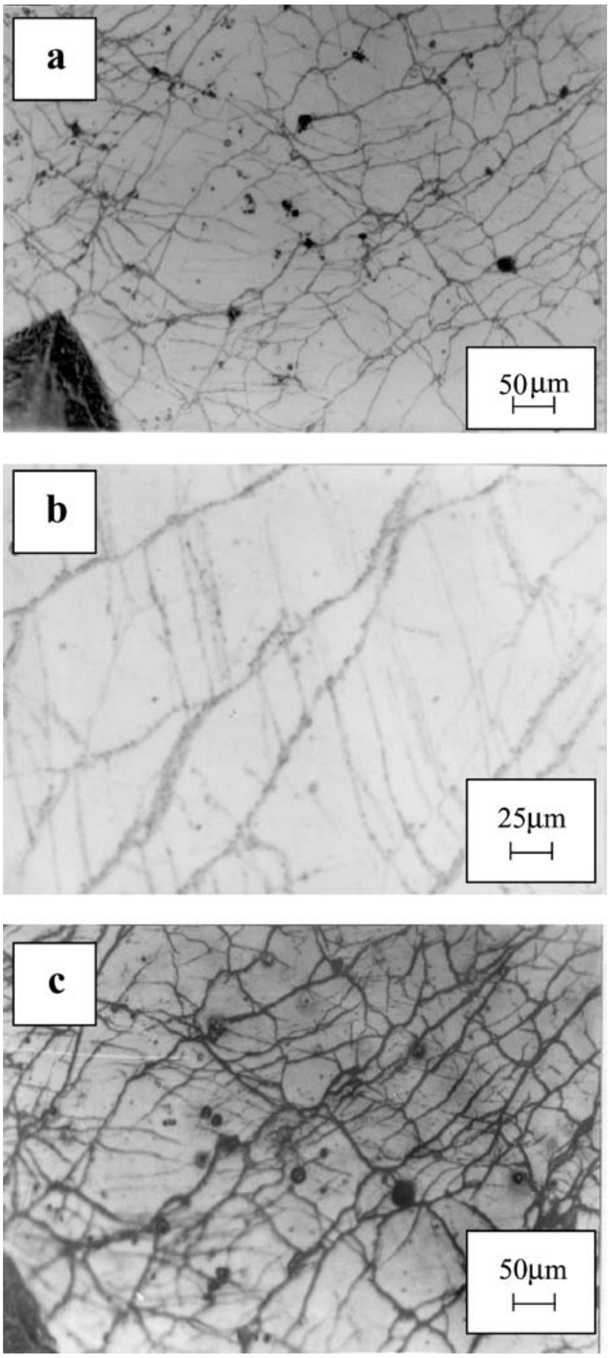

Figure 3 Microstructures of Al-30 wt% Zn alloy after 45% compressive strain and aged at 348 K for (a and b) 16 h and (c) 72 h.

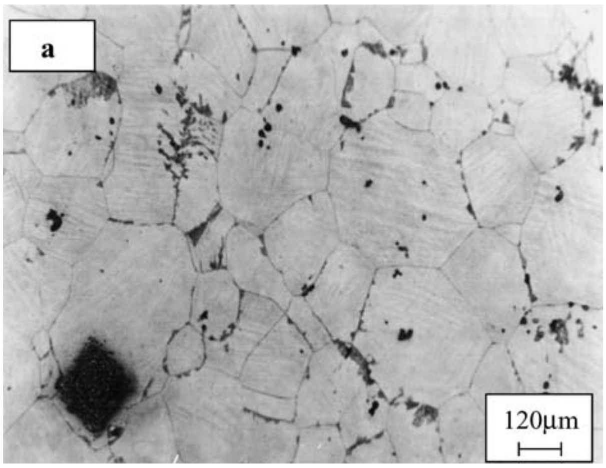

Figure 4 Microstructure of Al-30 wt% Zn alloy after aging at 348 K for 25 h. (hardness indentation located in the lower left corner of figure)

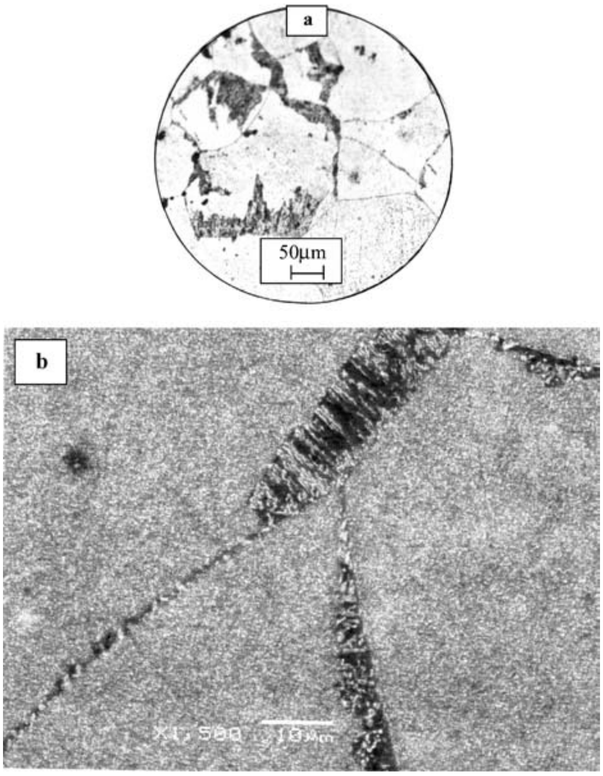

Figure 5 Microstructure by (a) OM and (b) SEM of Al-30 wt% Zn alloy after 20% compressive strain and aged at 423 K for 55 h.

3.2. Ageing at 423 K

The same analyses are used in the case of ageing at 423 K of deformed alloys. During this higher temperature, we have observed the formation of cellular precipitates in all deformed alloys below 70% (Fig. 5a). For this state of treatment, a morphology of lamellar structure of precipitates is observed by SEM (Fig. 5b).

Consequently, we can say that the ageing temperature increases the critical deformation for suppression of DP. The increased ageing temperature of deformed alloys leads to annihilation of dislocations and favors the development of DP.

It is important to notice the absence of recrystallization mechanism in our deformed alloys at these temperatures, because some authors have observed this phenomenon at precipitation temperature. For example, Hamana and Boumerzoug [34] have observed grain growth occurs in Cu-Sb alloy before DP.

X-ray diffraction showed that at two ageing temperatures (348 or 423 K) the undeformed alloys decomposed completely by cellular precipitation into a lamellar mixture of face centered cubic, aluminum rich α solid solution and an hexagonal close packed, zinc rich β solid solution. X-ray diffraction patterns from the aged deformed alloy is shown in Fig. 6, in the 2θ range between 30 and 150 deg, where, we observe the peaks of α and β phases. The following table contains different lattice parameters of α_0, α and β phases obtained from different patterns:

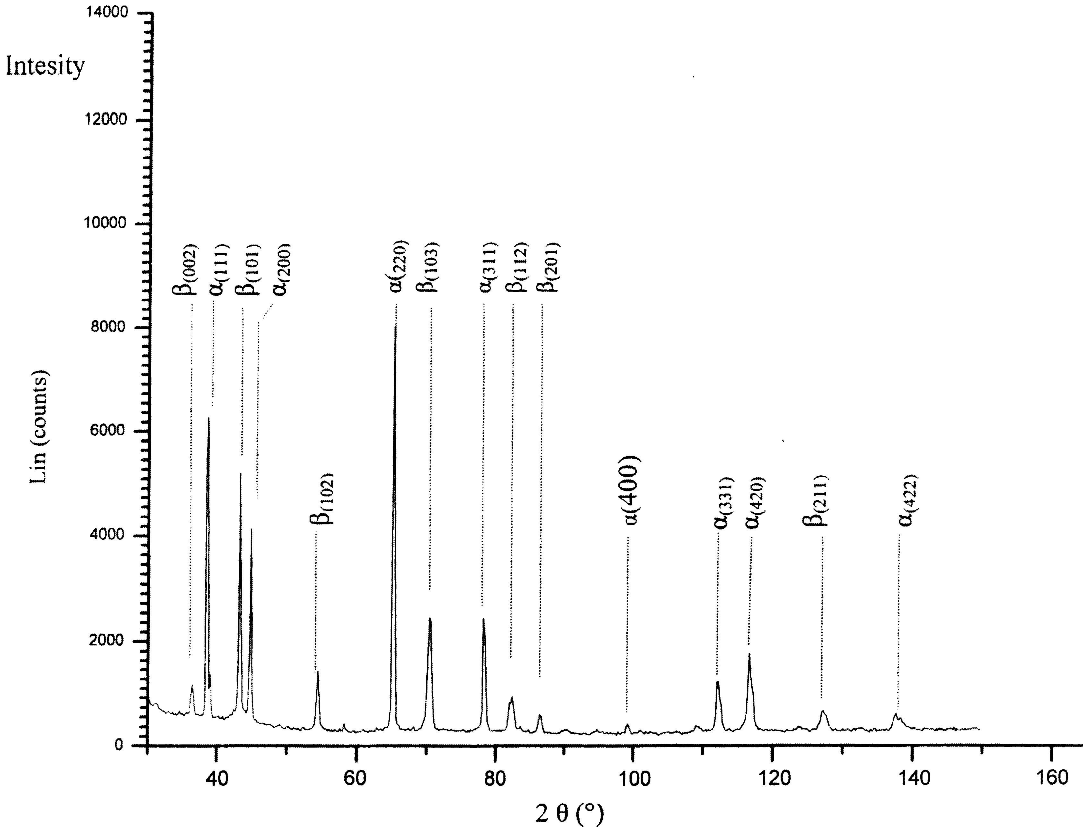

Figure 6 X-ray diffraction patterns of Al-30 wt% Zn alloy quenched, and aged at 423 K for 72 h.

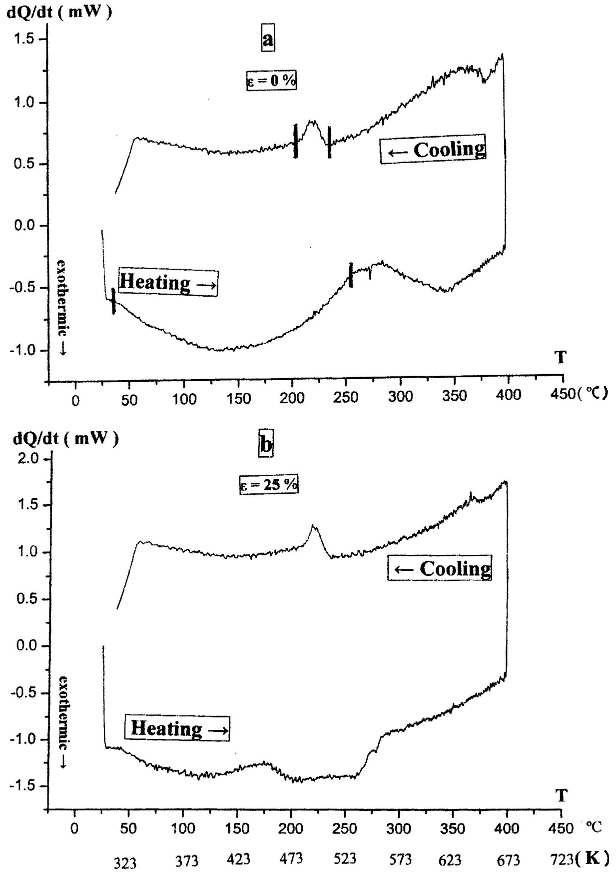

Figure 7 DSC curves of (a) undeformed and (b) deformed alloy, after quenching in iced water, and followed by heating and cooling with 5°C/min.

Quenched alloy	Aged alloy at 423 K
$\mathbf{a}_{\alpha 0} = 4.057$ Å	$\mathbf{a}_{\alpha} = 4.039$ Å, $\mathbf{a}_{\beta} = 2.665$ Å $\mathbf{c}_{\beta} = 4.94$ Å

Likewise, the effect of plastic deformation is confirmed by DSC. DSC heat flow curves obtained in undeformed (Fig. 7a) and deformed quenched alloys (Fig. 7b) after continuous heating until 673 K followed by cooling show the difference between these alloys. Upon heating there is difference between deformed and undeformed alloys, because the undeformed alloy (Fig. 7a) exhibits two exothermic peaks in the range temperature 308–483 K and 512–673 K, these peaks correspond to discontinuous precipitation which is confirmed by OM. But the deformed alloy (Fig. 7b) exhibits low exothermic peaks, corresponding to low dissipation of energy. During cooling, the same shape of curve is obtained in deformed or undeformed alloys, characterized by one peak between 483 and 513 K. This peak corresponds to the mechanism of non lamellar precipitation at grain boundaries.

4. Conclusion

Our investigation represents a contribution to the study of the effect of prior plastic deformation on the occurrence of discontinuous precipitation reaction. We have proved that plastic deformation by compression applied to quenched Al-30 wt%. Zn alloy affects this reaction during aging treatments. The occurrence of DP depends on the degree of prior plastic deformation. This reaction is observed only for low degree of deformation. For higher deformation, the alloy is occupied only by deformation bands which represent favorite sites of finer continuous precipitates.

References

1. W. GUST, in "Phase transformation", The Institution of Metallurgists, London, (1979). Vol. 1, p. II/27.
2. D. B. WILLIAMS and E. P. BUTLER, *Intern. Met. Rev.* **3** (1981) 153.
3. M. FREBEL and J. SCHENK, *Z. Metallkd.* **70** (1979) 230.
4. P. N. T. UNWIN and R. B. NICHOLSON, *Acta Metall.* **17** (1969) 1379.
5. E. HORNBOGEN, *Metall. Ts.* **3** (1972) 2717.
6. M. VON HEIMENDAHL, *Acta Met.* **15** (1967) 417.
7. *Idem.*, *ibid.* **21** (1967) 606.
8. E. HORNBOGEN, *Aluminium* **43** (1967) 163.
9. U. KÖSTER and E. HORNBOGEN, *Z. Metallk.* **59** (1968) 792.
10. C. LAIRD and H. I. AARONSON, *Trans. AIME* **242** (1967) 591.
11. R. A. FOURNELLE and J. B. CLARK, *Metall. Trans.* **3** (1972) 2757.
12. J. M. OBLAK and W. A. OWCZARSKI, *Trans. AIME* **242** (1968) 1563.
13. J. PETERMANN, *Z. Metallk.* **62** (1971) 324.
14. M. KITADA and T. J. DOI, *Jpn. Inst. Met.* **34** (1970) 369.
15. H. BORCHERS and H. SCHULZ, *Acta Mtall.* **24** (1976) 639.
16. S. SHAPIRO, D. E. TAYLER and R. LANAM, *Metall. Trans.* **5** (1974) 2457.
17. I. MANNA, S. K. PABI and W. GUST, *Int. Mat. Rev.* **46**(2) (2001) 53.
18. A. PAWLOWSKI, *Scripta Met.* **13** (1979) 791.
19. V. MELHORTHA and K. B. RUNDMAN, *Metall. Trans.* **3A** (1972) 1551.
20. E. P. BUTLER, V. RAMASWAMY and P. R. SWANN, *Acta Metll.* **21** (1973) 517.
21. K. N. MELTON and J. W. EDINGTON, *ibid.* **22** (1974) 1457.
22. A. PAWLOWSKI, *Scripta Met.* **13** (1979) 791.
23. M. VIGAYALAKSHNI, V. SEETHARAM and V. S. RAGHUMATHAN, *Acta Metal.* **30** (1982) 1147.
24. P. ZIEBA and W. GUST, *Z. Metallkd.* **91**(7) (2000) 532.
25. J. DUTKIEWICZ, *Met. Tech.* Oct. (1978) 333.
26. R. D. DOHERTY, *Met. Sci.* **8** (1974) 132.
27. R. CAHN, in "Recrystallization of Metallic Materials", edited by F. Haessner, (Riederer Verlag, 1971) p. 43.
28. H. TSUBAKINO, *Metallography* **17** (1984) 371.
29. D. HAMANA and H. CHOUTRI, *Scripta Metall.* **25** (1991) 859.
30. I. MANNA, S. K. PABI and W. GUST, *J. Mater. Sci.* **26** (1991) 4888.
31. I. MANNA and S. K. PABI, *J. Mater. Sci. Lett.* **9** (1990) 1226.
32. *Idem., ibid.* **13** (1994) 62.
33. T. H. CHUANG, R. A. FOURNELLE, W. GUST and B. P. PREDEL, *Scr. Metall.* **20** (1986) 25.
34. D. HAMANA and Z. BOUMERZOUG, *Z. Metallkde.* **85**(7) (1994) 479.

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