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Effect of cooling rate in overageing and other thermomechanical process parameters on grain refinement in an AA7475 aluminium alloy

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Fine-grained AA7475 aluminium alloy sheets were produced in this study by a thermomechanical treatment involving solution anneal, overageing, rolling and recrystallization steps. It has been found that the cooling rate after the intermediate overageing treatment should be fast to obtain the finest grain size. The fast cooling rate ensured the presence of relatively large particles of $MgZn_2$ and some supersaturation prior to cold rolling. Generally, the final grain structure was heterogeneous, with bands of fine grains lying parallel to the rolling direction. In material rapidly cooled after overageing, bands of fine grains were also observed in the transverse direction and these bands were associated with shear bands formed during rolling. The fine-grained AA7475 alloy sheets with an average grain size of about 9 μm showed large tensile elongations of about 800% when deformed at 516 °C and with an initial strain rate of 5×10^{-4} s^{-1}.

1. Introduction

The phenomenon of superplasticity requires a fine grain size; typically the mean grain diameter must be less than about 10 μm for superplastic behaviour to be observed. Fine grain structure in AA7075 can be obtained by thermomechanical treatments (TMT).

A number of intermediate TMT (ITMT) processes have been applied to 7xxx series aluminium alloys by investigators who sought improved ambient temperature properties through grain refinement [1, 2]. The original cast grain boundaries are eliminated by a recrystallization step prior to a conventional working process. Changes in grain size produced by ITMT processes have been attributed to differences in distribution of solute elements Zn, Mg and Cu [2] and to differing amounts and distributions of fine, Cr-bearing precipitates [1].

Based on the previously developed ITMT processes and improved understanding of the effects of dispersed particles on recrystallization, several studies [3–8] have been carried out, attempting to produce a fine grain size by thermomechanical processing, and to pin and stabilize the grain boundaries of this fine microstructure through some form of precipitates stable at high temperature. Work by Wert *et al.* [9] has indicated that grain refinement of conventional 7xxx series Al–Zn–Mg–Cu–Cr alloys is possible through thermomechanical treatments (TMT). The idea of using particles to create nucleating sites for recrystallization is the key to a four step thermomechanical processing sequence developed for grain refinement of AA7475. After initial homogenization, an appropriate overageing treatment is used to precipitate a high density of particles approximately 1 μm in diameter. This is followed by mechanical deformation at temperatures lower than conventional hot working temperatures, to minimize dynamic recovery, and to introduce a high degree of strain hardening. The deformation zones that form around the large particles during deformation serve as nucleation sites for recrystallized grains in a subsequent annealing treatment.

The mechanism of grain refinement by TMT has been further studied by Yoshida *et al.* [10]. They showed that the finest grain size was obtained in materials overaged at 400 °C for 8 h and then cold water quenched whereas the furnace cooled materials had significantly coarser grain sizes. These authors suggest that cell structures produced by multiple slip during rolling are stabilized by precipitation of supersaturated solute elements Zn, Mg and Cu (obtained by water quenching) at comparatively low temperature during the final annealing whereas, at high temperatures, those precipitates will dissolve and the aluminium–chromium dispersoids inhibit migration of grain boundaries, thus ensuring fine grain size.

The purposes of this investigation were to define more clearly the influence of thermomechanical processing variables, especially the cooling rate in overageing, on the development of fine grain structure in AA7475 for superplastic forming applications and to propose the mechanism of grain refinement in this alloy by TMT. The superplastic behaviour of the fine grained sheets was also evaluated.

2. Experimental procedure

The AA7475 ingot used for this study had a composition (weight per cent) of 5.4%Zn, 2.27%Mg, 1.5%Cu, 0.2%Cr, 0.06%Fe, 0.04%Si, 0.01%Mn and the balance Al. The AA7475 was further hot rolled in our laboratory at about 400 °C to plates 5 mm thick. These plates were then used as the starting material for grain refinement study.

The TMT route after conventional processing to the plate, about 5 mm thick, was the four step sequence, i.e., solution treatment, precipitation, rolling deformation and recrystallization. During solution treatment at 480 °C for 3 h followed by quenching into cold water (CWQ), the particles which precipitated during ingot homogenizing or hot working were dissolved to produce a standard initial condition. In the next step, ageing was carried out at temperatures from 160–420 °C for a time period of 15 min–34 h, followed by either CWQ, air cooling or furnace cooling at a rate of 16 $°C\,h^{-1}$ to control the size and volume ratio of second phase particles and the content of supersaturated solute elements. In the third step, rolling was carried out at temperatures ranging from room temperature to 250 °C, and with thickness reductions of between 50 and 90 per cent. In the final step, the cold or warm rolled sheets were recrystallized at 480 °C for 30 min in a salt bath and then quenched into water.

In addition, two types of modified TMT processes had been used to further clarify the effect of $MgZn_2$ precipitates on the refined grain size of the AA7475 material. In the first type, a short solution heat treatment was carried out after the overageing treatment (400 °C × 11 h), but prior to the rolling step in the standard TMT processing sequence. In the second type of TMT process, a two stage ageing treatment was employed, which involved a high temperature ageing treatment at 400 °C for 11 h followed by CWQ and a relatively low temperature ageing for 8 h at temperatures including 200 and 300 °C followed by CWQ.

A series of constant crosshead speed tensile tests was performed on an Instron testing machine to evaluate the superplastic behaviour of the fine-grained 1 mm thick sheets which had an average grain size of about 9 μm. Specimens with a gauge length of 7 mm and small shoulder radius (~ 2 mm) were used to keep the extended specimen in the constant temperature zone of the furnace.

Specimens for optical and electron microscopy examinations were prepared with standard techniques. Grain size measurements were performed by the linear intercept method on micrographs of specimens electrolytically etched/anodized using a solution of 33.3%HNO_3 and 67.7% methanol [11]. Foils used for transmission electron microscopy (TEM) were removed from the bulk specimens parallel to the rolling plane.

3. Results

Many external variables in the four step thermomechanical treatment can, to some extent, affect the recrystallized grain sizes and their distribution. The variables examined in this study are as follows: 1. ageing temperature; 2. ageing time; 3. cooling rate after overageing treatment; 4. rolling reduction and 5. rolling temperature.

3.1. Microstructural study of the TMT process by TEM

In order to understand the roles played by second phase particles in the grain refinement of AA7475, the microstructural changes of the alloy in the previously described four-step TMT were examined by both optical microscopy and transmission electron microscopy. A TEM micrograph of the 400 °C × 8 h overaged structure is shown in Fig.1a. Composition analysis of the large particles shown in Fig. 1a by scanning transmission electron microscopy (Phillips EM 400) revealed that there were two types of particles in this size category:

1. The majority of the particles analysed contained Al, Zn and Mg as depicted in Fig. 2a and were identified as $MgZn_2$. Generally, the overageing precipitates had a light grey appearance and they were easily identified from the typical etching effect produced by the thinning solution.
2. Some of the particles analysed contained Al, Si or Fe as indicated in Fig. 2c. These particles are often called constituent particles. They were not dissolved during solution treatment and existed in the form of stringers after rolling. These constituent particles played some role in recrystallization, which will be discussed later.

After the overageing treatment, there existed a bimodal particle dispersion. Large particles in the size range 0.5–1 μm were equilibrium precipitates formed during overageing. Dispersoid particles in the size range 50–100 nm were also present.

Fig. 1b shows the microstructure after the complete recrystallization treatment of 30 min at 480 °C. In addition to recrystallization of the matrix, the large precipitates have dissolved, leaving the dispersoid particles in the recrystallized, fine grain matrix. The role of the fine dark grey dispersoid particles in restricting grain growth at annealing temperature is clearly seen from Fig. 1b where several dispersoid particles produce substantial distortions in a grain boundary. These dispersoid particles were analysed and found to be Cr-containing dispersoids (Fig. 2b) which formed by solid state precipitation subsequent to casting.

3.2. Effect of ageing temperature and time and cooling rate after overageing on recrystallized grain size

Overageing is the key step in the four-step thermomechanical treatment. There are three process variables involved in this step, i.e. overageing temperature, time and cooling rate after overageing. For this study,

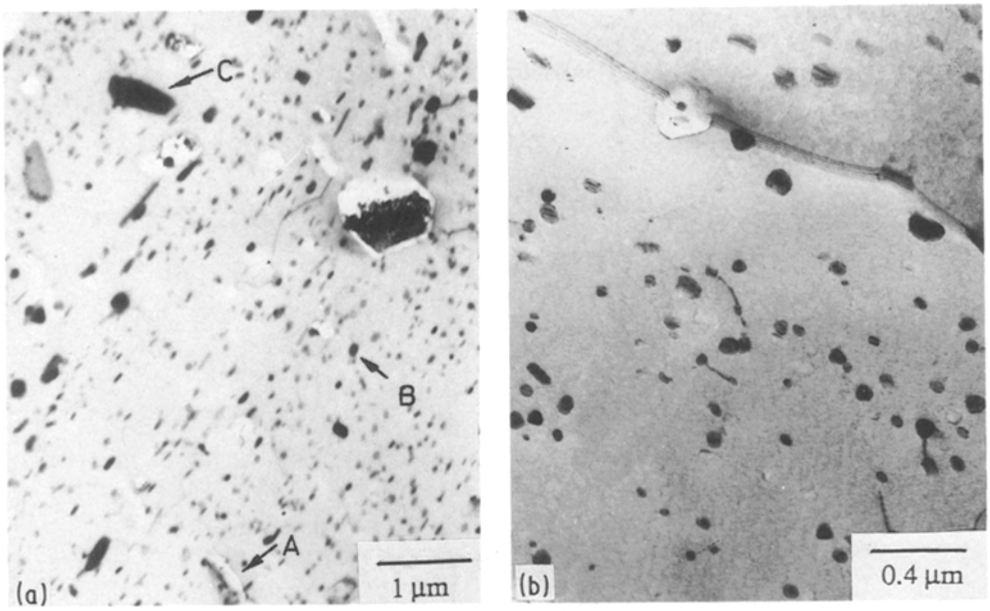

Figure 1 TEM microstructures of AA7475: (a) solution treated 480 °C × 3 h + CWQ, overaged at 400 °C × 8 h; (b) treatment (a) and rolled at 200 °C by 80% in thickness reduction, completely recrystallized at 480 °C for 30 min.

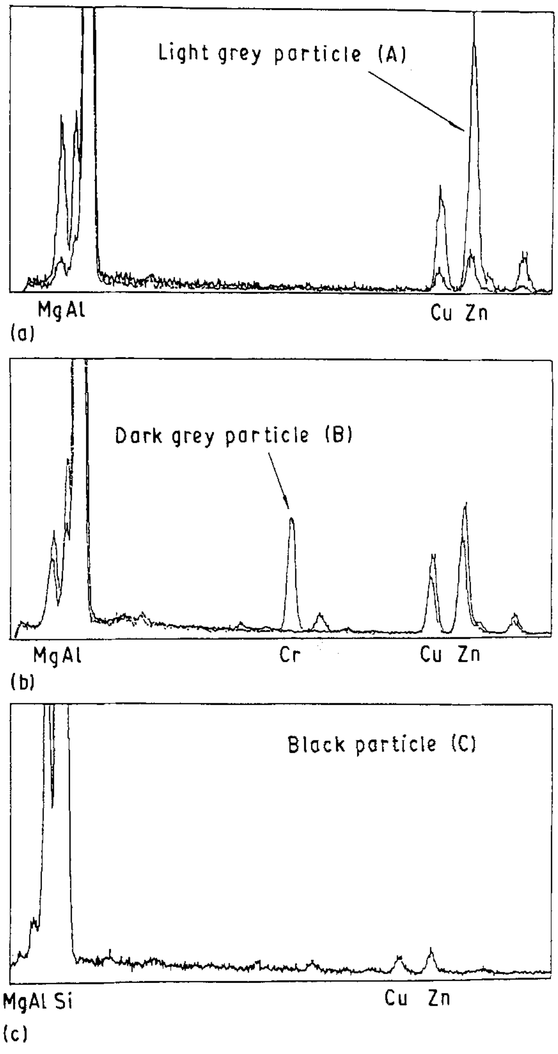

Figure 2 Microanalysis of second phase particles in AA7475 by STEM microscopy: (a) light grey particles ($MgZn_2$ precipitates); (b) fine dark grey dispersoids; (c) large dark particles formed by Fe and Si impurities. The background in (a) and (b) is the matrix composition.

thickness reduction of 80% was held constant in the cold rolling step.

The first relationship examined was between ageing temperature and final grain size. The ageing time of 8 h was held constant and the ageing temperature ranged from 160 to 420 °C. Aged specimens were cold water quenched. The effect of ageing temperature on grain size is shown in Figs 3 and 4. As can be seen from those inserts in Fig. 3, ageing for 8 h at 160 °C causes precipitation to occur in the supersaturated solid solution. Higher overageing temperatures cause coarsening of the precipitate dispersion in the usual manner: the average particle size increases but the overall particle density decreases. It is also clear that grain boundary precipitates are of much larger size than the precipitates in the grain interior.

It is obvious from Figs 3 and 4 that the overaged precipitates play an important role in the grain refinement process. The final grain size decreases very sharply as the overageing temperature increases to above 300 °C. A minimum in grain size is obtained in specimens held at 400 °C, which produced the coarsest precipitates. It is also noteworthy that unaged material give a finer grain size than the low temperature (e.g. 160, 280 °C) aged material. The rate of dislocation accumulation as a function of cold rolling is expected to be higher in the unaged material than the material aged at comparatively lower temperatures. The reason for this conjecture is more effective binding interaction between supersaturated solute elements and dislocations than similar interaction between dislocations and precipitates formed during low temperature ageing. This produces higher stored strain energy, the driving force for recrystallization, in the unaged material after the rolling operation. It is also seen from Fig. 4 that, after reaching a minimum of about 9 μm, the recrystallized grain size increases with further increase in overageing temperature. This sug-

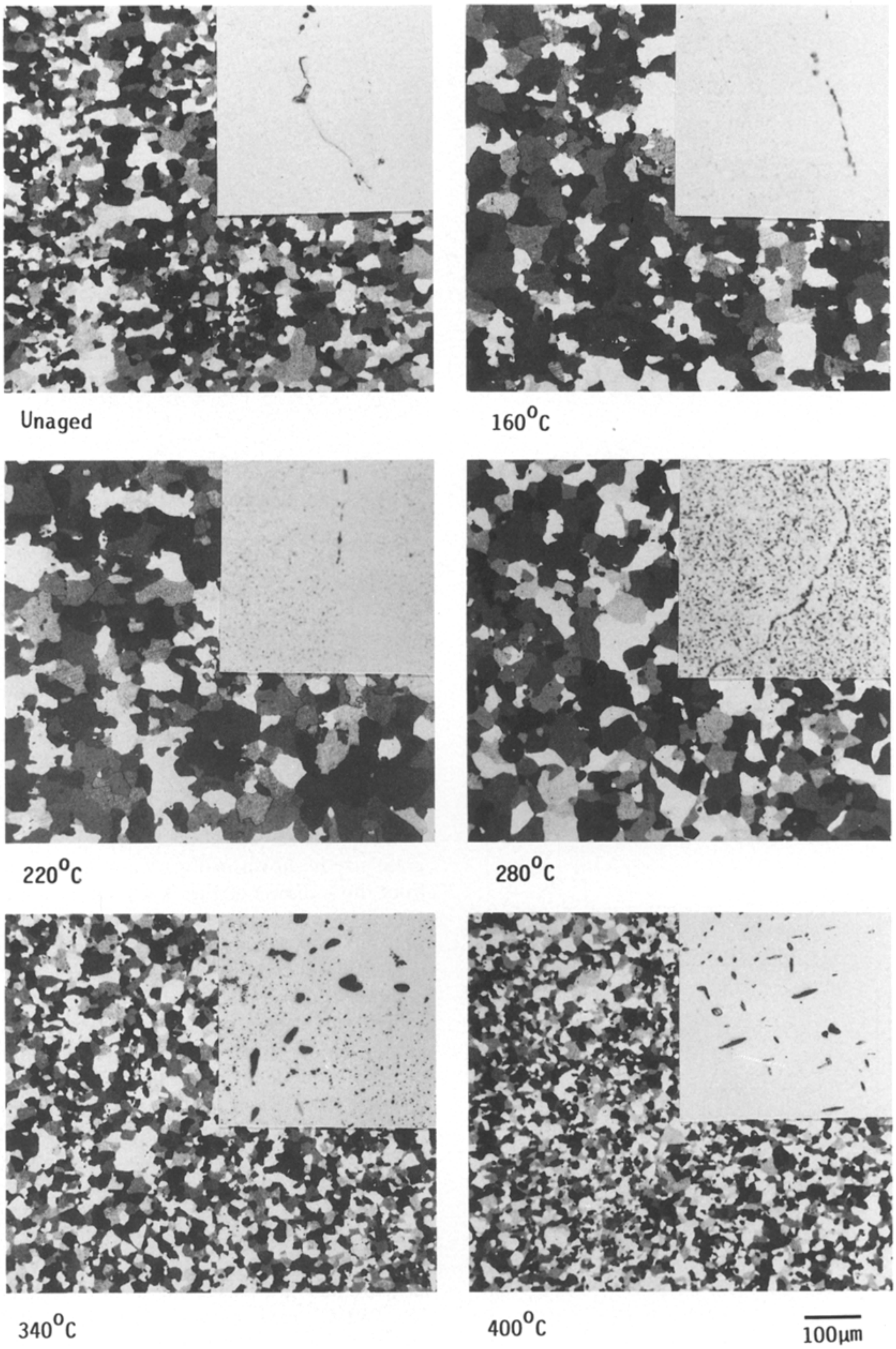

Figure 3 Recrystallized grain structures for AA7475 overaged for 8 h at temperatures of 160, 220, 280, 340 and 400 °C. The related precipitation structures after overageing for 8 h at the indicated temperatures are also shown (900 ×).

gests that less nucleation sites are created by overageing at temperatures above 400 °C as a result of the decrease in precipitate density accompanied by an increase in particle size and decrease in solid state solubility of alloying elements.

The final recrystallized grain sizes of the alloy as a function of overageing time at 400 °C for three different cooling rates (CWQ, air and furnace cooling) are plotted and shown in Fig. 5. A minimum in grain size is observed in specimens held at 400 °C for approximately 11 h followed by CWQ. The increase in grain size at longer overageing time than 11 h suggests that some dissolution of relatively smaller precipitates has occurred. Some examples of the microstructures resulting from the study of cooling rate effects on final grain size are shown in Fig. 6. It can be seen from

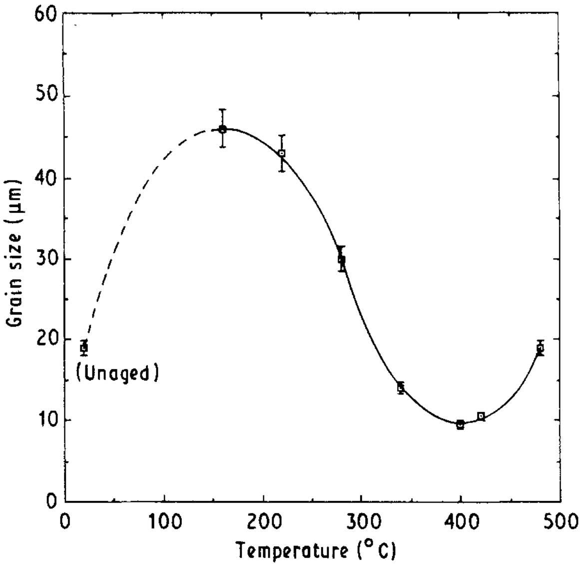

Figure 4 Effect of overageing temperature on the final grain size. The overageing time of 8 h was held constant, (CWQ plus cold rolling at 80%).

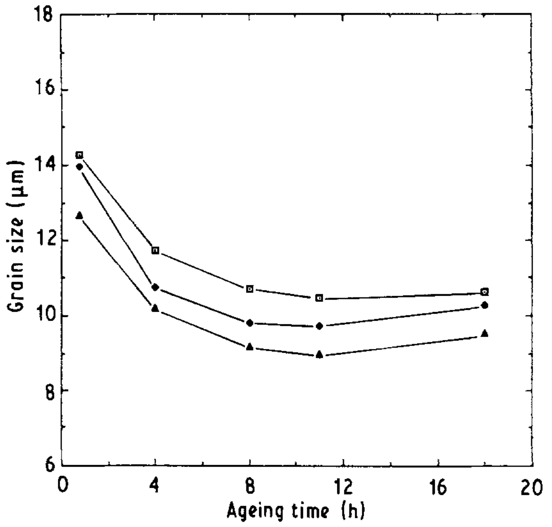

Figure 5 The final recrystallized grain size of AA7475 as a function of overageing time at 400 °C for three different cooling rates: (▲) CWQ; (◆) air and (⊡) furnace cooling.

Fig. 6 that the thermomechanically processed sheets generally contain a heterogeneous grain structure. This duplex grain structure is influenced by cooling rate after the overageing treatment. In the case of comparatively slow cooling rate conditions, such as air or furnace cooling, the final grain structure consists of alternate coarse and fine grain bands lying parallel to the rolling direction whereas rapid cooling, such as water quenching, produces networks of fine grain bands along both longitudinal (L) and long transverse (LT) directions.

In order to establish the causes for the formation of bands of fine grains in the long transverse direction, it is helpful to note that shear bands have been observed under optical microscopy only in cold working of water-quenched material as shown in Fig. 7. Traces of shear bands observed on the L-LT plane occurred perpendicular to the rolling plane. In fact, the shear band lines, approximately 1 mm apart, were clearly visible without a microscope on the sheet surface after rolling and they caused shear crack at rolling reduction higher than 80%. In preparing metallographic

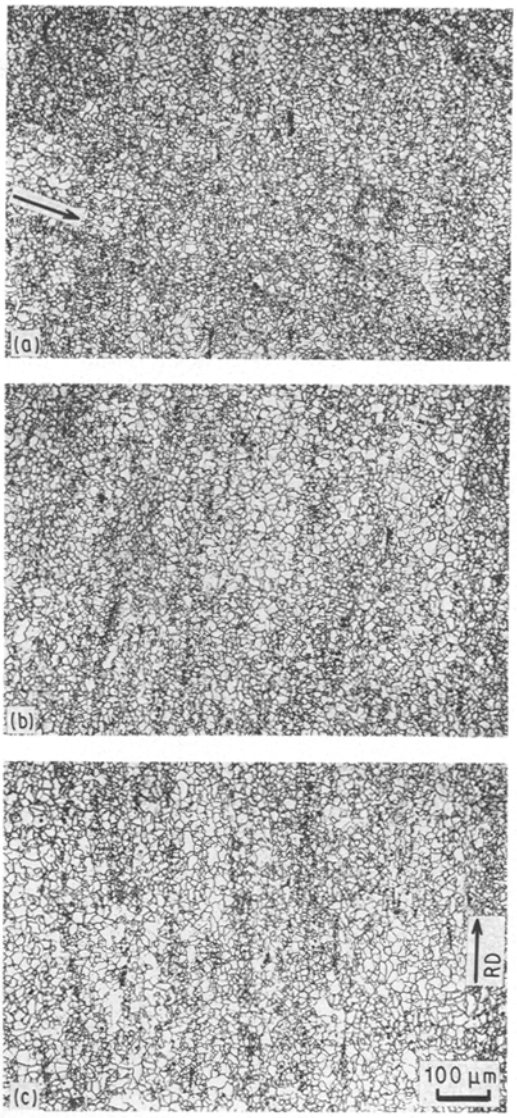

Figure 6 Examples of grain structures of AA7475 overaged at 400 °C for 11 h followed by three different cooling rates: (a) CWQ, (b) air and (c) furnace cooling. The arrow in (a) indicates the direction of bands of fine grains.

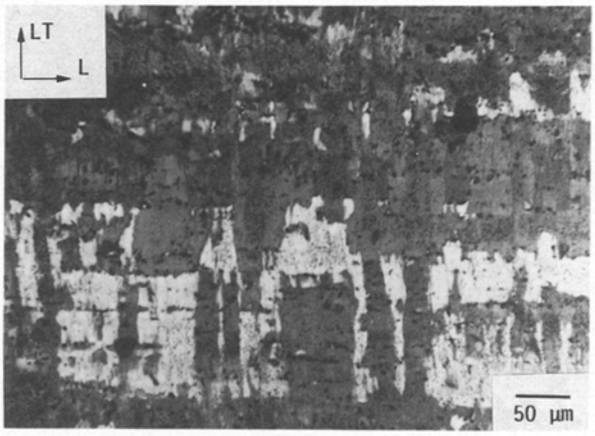

Figure 7 Microstructure of cold rolled AA7475 which was previously overaged at 400 °C for 11 h and cold water quenched.

specimens these macroscopic shear band lines were removed during mechanical polishing but, after etching, new lines appeared again at the same location as the macroscopic shear bands. The new lines were identified by optical microscopy to be associated with bands of fine grains. The direction of these fine grain bands are indicated by an arrow in Fig. 6a. The fact that the bands are at 75 °, instead of 90 °, to the rolling direction is a result of the difficulties in maintaining the feeding direction of sheets in the same direction as the original rolling direction in the cold rolling step. On the microscopic scale, the shear band width is 20–40 μm (Fig. 7), which correlates well with the spacing between bands of fine grain size in the transverse direction in the water quenched material, Fig. 6.

The heterogeneous deformation resulted in duplex grain structures in the processed sheets, and consequently, affected the average refined grain sizes. After overageing treatment at the same temperature for the same ageing period, the cold water quenched material was found to have the finest grain size (average) and the furnace cooled the coarsest, as can be seen from Fig. 5.

3.3. The effect of rolling reduction and temperature on grain size

The final grain size achieved after salt bath annealing was found to decrease as the rolling reduction at room temperature was increased from 50 to 80%, and it then tended to stabilize as the reduction was increased to 90%. The minimum grain size achieved in these experiments was 8 μm as shown in Fig. 8a, and larger rolling reductions would not be expected to reduce this figure significantly. This agrees with the observation by Wert *et al.* [9] who suggest that very high rolling reductions do not continue to generate intense deformation zones around smaller and smaller particles, i.e., new particles are not activated as nucleation sites.

For the material that was rolled 80%, it was found that increasing the rolling temperature to 200 °C caused only a 15% increase in grain size (Fig. 8b). This suggests that the ability of the particles to nucleate new grains is not significantly reduced by the recovery mechanisms operating at elevated temperatures. This is important, for example, in developing commercial production schedules.

3.4. Grain size after modified TMT processes

To verify the role of coarse $MgZn_2$ precipitates in grain refinement a brief solution treatment (480 °C × 5 min) was introduced between the second and third steps (i.e., between the overage treatment and rolling deformation) of the standard four-step TMT process. After the extra solution treatment at 480 °C for 5 min, large precipitates formed during the overageing step were dissolved and the absence of large second phase particles resulted in a coarse grain size of ~ 30 μm.

By carrying out a relative low temperature ageing at 200 or 300 °C after ageing at 400 °C for 11 h and water quenching, the supersaturated Zn, Mg and Cu alloying elements precipitated out as $MgZn_2$ particles. In comparison with the normal single stage overageing treatment at 400 °C for 11 h followed by CWQ, the duplex ageing treatment produced much coarser grain size (~ 15 μm) in the recrystallized sheets.

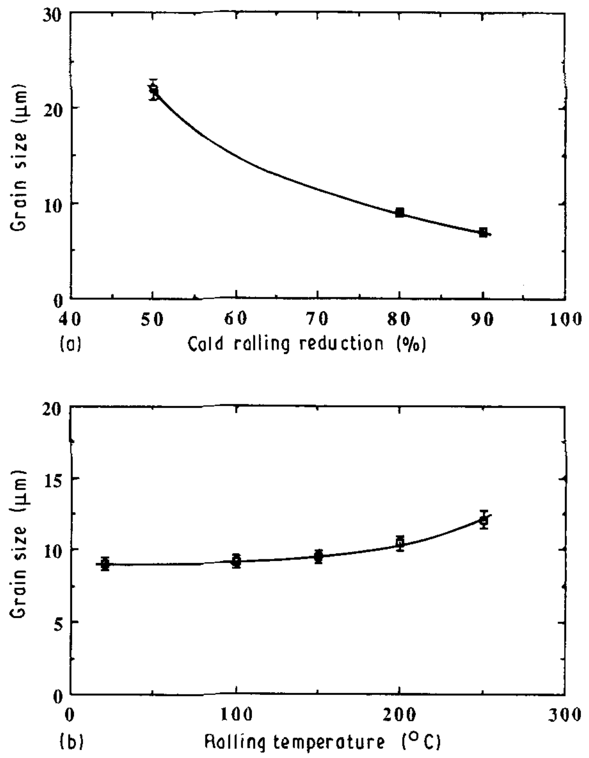

Figure 8 Recrystallized grain size as a function of (a) cold rolling reduction and (b) rolling temperature for a thickness reduction of 80% (400 °C × 11 h + CWQ).

3.5. High temperature tensile tests of AA7475 aluminium alloy

AA7475 alloy sheets with an average grain size of 9 μm processed by optimum TMT (i.e., overageing 400 °C × 11 h, followed by CWQ) were used for tensile testing. Excellent superplastic behaviour of the fine grained AA7475 are clearly demonstrated in Fig. 9a and b where elongation is plotted as a function of testing temperature and crosshead speed, respectively. Elongations higher than 800% were measured for the AA7475 alloy deformed at 516 °C and at a constant crosshead speed of 0.5 mm min^{-1} which gave an initial strain rate of $7 \times 10^{-4}\ s^{-1}$.

4. Discussion

4.1. The role of second phase particles in grain refinement of AA7475 aluminium alloys

The grain refinement of AA7475 aluminium alloy involves complex metallurgy because the control of final grain size is often dictated by complex interactions between the deformed state, particle size and distribution, solute content of the matrix and other variables such as time and temperature.

The volume fraction, size and distribution of dispersoid particles stay quite stable throughout the four-

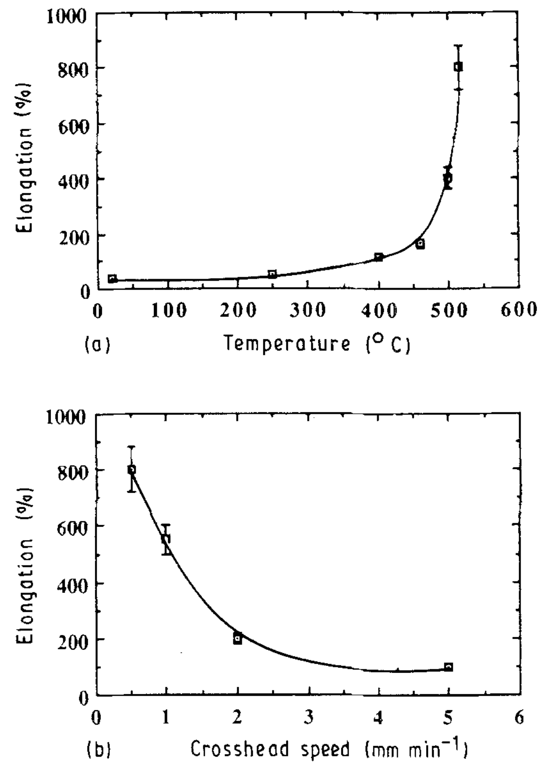

Figure 9 Effect of: (a) temperature at a speed of 0.5 mm min^{-1} and (b) crosshead speed at a temperature of 516 °C on superplastic elongation of grain size 9 μm.

step TMT. The primary importance of these dispersoids resides in the final solution anneal: inhibiting nucleation of recrystallized grains and stabilizing the recrystallized grain size, as shown in Fig. 1b. The stable grain size at which grain growth is arrested is a function of the volume fraction and the average size of the Cr-containing dispersoids. The fact that the final grain size saturates at a level of 9 μm when the rolling reduction is over 80% and with other TMT parameters being optimal suggests that the finest achievable grain size of 9 μm is close to the stable grain size determined by the dispersoids.

Based on the observation that the $MgZn_2$ phase precipitates heterogeneously on the Cr-bearing dispersoids [12], it may be concluded that the distribution of the dispersoids can influence the final grain size not only through its influence on restricting grain growth but also through its influence on the distribution of the large $MgZn_2$ particles formed during overageing.

The overageing precipitates play a totally different role in grain refinement from the dispersoids. The dispersoids control the coarsening of refined grain structure; in contrast, the overaged precipitates are used to create nucleation sites for recrystallization and refine the grain size.

For a constant overageing time of 8 h, increasing the overageing temperature (up to 400 °C) produces an increasingly coarser $MgZn_2$ precipitate dispersion in the following manner: the average particle size increases but the over-all particle density decreases (Fig. 3). It is thus suggested that the change in solubility of Zn and Mg in the matrix is very limited up to 400 °C or slightly higher temperatures. However, the change in the size and density of $MgZn_2$ precipitates with overageing temperature can strongly influence the recrystallized grain size. Considerable grain refinement was achieved for overageing at temperatures above approximately 340 °C, suggesting that particles larger than a critical size can create nucleation sites for recrystallizing grains. This observation agrees with the results of Wert *et al.* [9] who found that the minimum size of particles capable of nucleating new grains was 0.75 μm.

It is sometimes argued that further precipitation of the Cr-containing dispersoids during ageing treatment contributes to the overall grain refinement through its influence on grain growth control. Experimental results obtained in this study have shown that this is not true. As reported before, when a brief solution treatment was introduced between overageing (400 °C × 11 h + CWQ) and cold rolling the grain size increased to 30 μm compared with the usual refined grain size of 9 μm. It is believed that during the short solution treatment, the size and distribution of the dispersoids stay quite stable while the $MgZn_2$ precipitates are dissolved into solution. A lack of large $MgZn_2$ precipitates prior to rolling deformation results in coarse grain size in the recrystallized sheet.

With regards to the relative importance of overageing precipitates and impurity-containing constituent particles in forming nucleation sites for new grains, it has been reported that after overageing for 8 h at 400 °C the density of precipitates having diameters more than 0.75 μm is more than forty times the density of constituent particles with diameters larger than 0.75 μm [9]. Although constituent particles and precipitates are equally effective as nucleation sites, the number of grains nucleating at precipitates is more than forty times the number nucleating at constituent particles; this suggests that constituent particles do not make a substantial contribution to the fine grain size in the optimally aged material. Thus, the AA7475 aluminium alloy should be capable of achieving approximately the same recrystallized grain size as AA7075 which has higher content of Fe and Si than AA7475.

The fine grain size bands in the longitudinal direction of the final sheets (Figs 3 and 6) cannot be simply attributed to the long stringers of coarse constituent particles in the rolling direction. It is proposed that coarse grain boundary precipitates, which also exist in the form of long stringers after the rolling operation, are much more effective in producing bands of fine grains mentioned above than the coarse constituent particles.

4.2. Effect of cooling rate in overageing on the recrystallized grain size

The results concerning the effect of the cooling rate after overageing treatment on recrystallized grain size and grain size distribution have been presented in Figs 5–7. For an overageing temperature of 400 °C and ageing time of 8 h, a grain size of 9 μm has been obtained in cold-water quenched material compared with a minimum grain size of 11 μm achieved in the furnace cooled material. Moreover, CWQ also influences the recrystallized grain structure. Duplex grain structures have been observed in all possible combinations of ageing temperature, ageing time and cooling rate while the cooling rate influences the grain size distribution patterns in the recrystallized sheets.

In comparatively slow cooling conditions such as air and furnace cooling, the final grain structure consists of alternate coarse and fine grain bands in the rolling direction. It is also noticed that these fine grain bands seem to be closely related to large size particles (Fig. 6). These stringers of particles are again associated with the grain boundaries of the starting material, the structure of which is very coarse and elongated during rolling deformation. As previously discussed in Section 4.1, the fine grain bands in the rolling direction are most probably a result of the coarse grain boundary precipitates which form during the overageing step and spread out in the form of stringers in the rolling direction during rolling deformation.

In rapidly cooled conditions, however, fine grain bands have been observed in the final grain structure, not only in the rolling direction but also in the transverse direction. It is known that the Portevin–Le Chatelier effect occurs in the presence of supersaturated solute elements and produces shear bands during deformation. Based on the optical microstructural observations it is proposed that the bands of fine grains in the transverse direction are a result of the heterogeneous deformation, i.e., shear bands in the transverse direction shown in Fig. 7, during rolling of the water quenched material. More direct evidence of this needs to be provided by means of an *in situ* transmission electron microstructural study of both the deformed structure and the initial stage of final recrystallization. From the viewpoint of commercial process control, cross rolling has been employed to reduce the extent of the non-uniformity of the final grain structure.

Additional grain refinement obtained by CWQ seems to be a result of the fine grain size bands lying along the transverse direction. Yoshida *et al.* [10] have proposed that supersaturated solute elements obtained by CWQ after ageing treatments form precipitates on the dislocations and dislocation networks during cold rolling or heating up in final annealing, thus inhibiting recovery of the deformed structure and leading to the development of the finest grain size. However, they did not provide any direct evidence to support their proposition.

4.3. Summary of the grain refinement mechanism in AA7475

Schematic diagram of grain refinement described above is shown in Fig. 10. Interpretation of the diagram is as follows. The solution treatment of a hot-rolled plate has little influence on grain size in recrystallized sheets [9]. This step is simply designed to produce a uniform starting condition by dissolving the soluble precipitates, placing in solution the Zn, Mg and Cu alloying elements. The finest grain size in this study is obtained by overageing at 400 °C × 11 h + CWQ prior to cold rolling (the right column in Fig. 10). Under this condition, the large overageing precipitates and supersaturated solute elements coexist in the matrix. Multiple slip occurs during deformation in the presence of the large second phase particles which have no coherency with the matrix. Multiple slip promotes the formation of sessile dislocations and dislocation networks, while the supersaturated solute elements interact with the dislocations and their networks, hindering their mobility and their ability to rearrange themselves into low energy configurations. The existence of supersaturated solute elements also promotes the formation of shear bands during rolling deformation. A high driving force for recrystallization together with nucleation sites for new grains are achieved during cold rolling. The final anneal step can be divided into two stages in terms of temperatures: the low and the high temperature stages. At low temperatures of annealing, the supersaturated solute elements obtained by CWQ after ageing form precipitates on the dislocations and dislocation networks, thus inhibiting recovery of the deformed structure. At very high temperatures, while the precipitates dissolve, the migration of boundaries of grains or subgrains is inhibited by Cr-containing dispersoids. This is the main reason why the finest grain is formed in the recrystallized sheets. It is also worth noticing that the cold water-quenched material is harder than the slowly cooled one. Therefore, for a certain amount of cold reduction, intense deformation zones can be generated around smaller particles for water-quenched material than those for the slowly cooled material. In other words, more particles are activated as nucleation sites for water-quenched material. In addition, heterogeneous deformation in the form of shear bands is produced in cold rolling of cold water-quenched material. On these shear bands intense deformation zones are expected to be generated around smaller particles compared with regions between the shear bands. Bands of fine grains are therefore produced in the final annealed sheets.

When only supersaturated solute elements exist and large particles are absent, as in the 480 °C × 3 h + CWQ condition (left column in Fig. 10), shear bands occur during cold rolling and dislocation density becomes heterogeneous in the deformed structure. The grain size in this condition is controlled by the recovery of regions with high dislocation density where supersaturated solute elements precipitate, and therefore may be correlated with the spacing of shear bands.

When supersaturated elements do not exist, as in the 400 °C × 8 h + FC condition (centre column in Fig. 10), multiple slip caused by large particles produces cell structures during cold working. Then subgrains or fine grains are formed at comparatively low temperatures and grain growth occurs gradually during annealing. Thus maximum reduction in grain size cannot be achieved in this case.

If the overaged specimen is successively aged at lower temperatures after ageing at 400 °C for 8 h, supersaturated elements, obtained by water quenching from 400 °C, precipitate preferentially on the pre-existing large $MgZn_2$ particles, causing coarsening

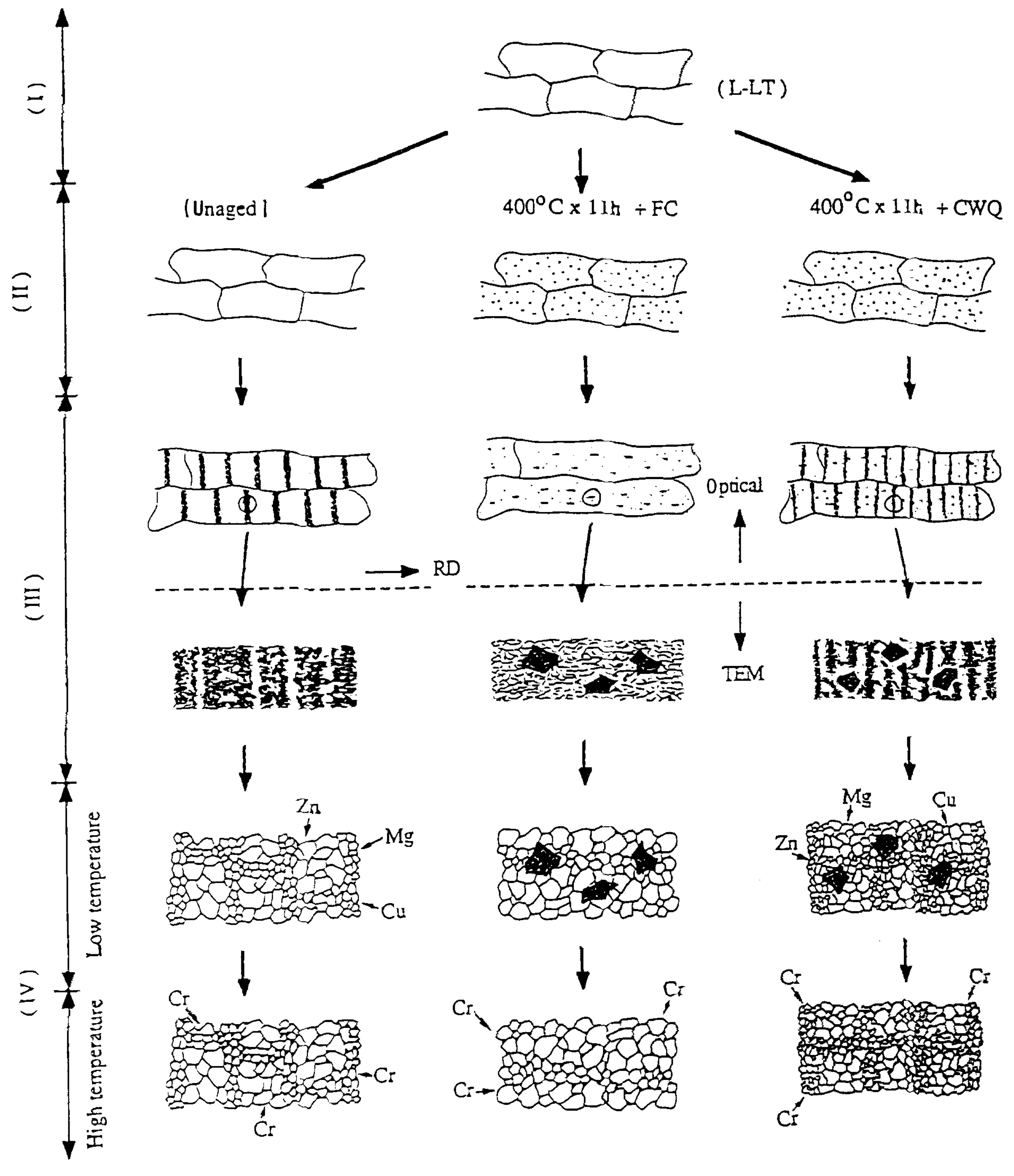

Figure 10 Schematic diagram of grain refinement in the Al–Zn–Mg–Cu–Cr alloy. Zn, Mg and Cu refer to either these elements in supersaturated solution or precipitates formed by these elements. Cr refers to Cr-containing dispersoids.

of the $MgZn_2$ precipitates. Simultaneously, the amount of the supersaturated solute elements is greatly reduced. The resulting microstructure is more or less similar to that of the specimen overaged at 400 °C for 8 h and furnace cooled. For the same reason as discussed for the latter case, an intermediate grain size is formed.

Regarding the effect of heating rate in the final annealing, it is suggested that, in the case of slow heating, the migration and annihilation of dislocations becomes easier because of coagulation of precipitates which are pinning the grain boundaries at comparatively low temperatures; in other words, some thermal recovery of the matrix defect structure occurs below the recrystallization temperature during slow heating. This reduces the number of active recrystallization nuclei and thus increases the recrystallized grain size. Moreover, isolated and highly favoured nucleation sites become activated at relatively low temperatures (about 300–360 °C) [13]. These nuclei then have time to grow and consume neighbouring, less favoured nucleation sites before annealing temperatures are finally reached at which a larger number of nucleation sites become activated. This results in a coarse grain size.

5. Conclusions

Thermomechanical treatments for an AA7475 aluminium alloy were investigated in order to develop fine grain microstructures. The process involves four steps: solution treatment, precipitation or overageing, rolling and recrystallization. The following conclusions can be drawn from this investigation:

1. The finest grain is obtained by ageing at 400 °C × 11 h followed by quenching into cold water in the overageing step. To obtain the finest final grain size, the coexistence of large second phase particles which have no coherency with the matrix and supersaturated solute elements is required prior to cold rolling; in other words, the cooling rate after overageing treatment should be as rapid as possible if the minimum grain size is to be achieved.
2. Cooling rate after overageing treatment also influences the grain size distribution patterns in the recrystallized sheets of AA7475. In comparatively slow cooling conditions such as air and furnace cooling, the final grain structure consists of alternate coarse and fine grain size bands in the rolling direction. It is proposed that the coarse grain boundary precipitates formed during the overageing treatment are responsible for the longitudinal bands of small grains. In rapidly cooled conditions, however, fine grain bands were observed in the final grain structure, not only in the rolling direction but also in the transverse direction. Fine grain bands in the transverse direction are a result of the heterogeneous deformation in the form of shear bands occurring during rolling of the water-quenched material.

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