INFLUENCE OF PREDEFORMATION ON PRECIPITATE COARSENING IN Al-Zn-Mg-Cu ALLOYS

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ABSTRACT The influence of predeformation on overageing and precipitate coarsening has been investigated in the Al-Zn-Mg-Cu alloys AA7030 and AA7108. Ageing curves for nondeformed and 10% deformed alloys were recorded by means of tensile testing while the precipitate structure was investigated by TEM. For TEM-investigations, micro- and mesoscale characterization was carried out with focus on precipitate size, size distribution and spatial distribution. An interesting observation is that during predeformation, the dislocations seem to organize themselves into a loose cell structure with broad cell boundaries. Since the nucleation and growth of precipitates is favored on dislocations, local differences in the precipitate size at a meso-scale could therefore be observed.
Keywords: *Al-Zn-Mg-Cu, predeformation, precipitation, coarsening, η-phase, zirconium*

1. Introduction

In the production of aluminium alloy bumpers for automobiles, stretch bending before age-hardening is an important processing step. During this forming operation, dislocations will be introduced into the material, influencing the nucleation, growth and coarsening of the hardening precipitates during the subsequent heat treatment. This effect of deformation has recently been studied by Poole and Shercliff [1], by Poole, Shercliff and Castillo [2] and by Deschamps, Brechet, Guyot and Livet [3]. In the works by Poole et al.[1,2], the kinetic and mechanical aspects were emphasized. They found that in the case of predeformation, the initial yield stress was higher, the peak stress occurred at shorter ageing times and the overageing kinetics became accelerated. Deschamps et al. [3] focused more on the details of microstructural evolution, demonstrating how vacancies which act as nucleation sites for the metastable η'-phase will migrate to the dislocations and inhibit homogeneous nucleation. This consequently leads to the formation of precipitate free zones (PFZ) around the dislocations. The dislocations can also act as heterogeneous nucleation sites for the equilibrium η-phase and increase the coarsening rate of these precipitates due to pipe diffusion along the dislocations. The present study examines these effects in alloys with a lower level of alloying additions, with and without the presence of zirconium.

2. Experimental

Two Al-Zn-Mg-Cu alloys have been investigated, AA7030 and AA7108, with their chemical composition given in Table 1. These are two medium strength alloys used for bumpers by several European car manufacturers. The main difference between them, is the addition of zirconium to the 7108-alloy. This leads to the formation of Al_3Zr-dispersoids which pin the grain boundaries during hot deformation, giving the material a fibrous grain structure. The 7030 alloy, which does not contain dispersoids, has a coarse equiaxed grain structure after hot deformation.

Table 1 Chemical composition of the investigated alloys

Alloy	wt% Zn	wt% Mg	wt% Cu	wt% Zr	wt% Al
7108	5.45	1.22	0.30	0.16	bal.
7030	5.45	1.20	0.27	-	bal

DC-cast and homogenized billets were extruded into sheet profiles, 5 mm thick and 150 mm wide. Tensile samples were machined from the sheets with the tensile axis parallel to the extrusion direction. Solid solution heat treatment at 480°C for 30 minutes was done in an air furnace followed by water quenching. Some of the samples were predeformed to 10% permanent elongation, which was done within 15 minutes after the quench. Undeformed and predeformed tensile samples were stored at room temperature for 24 hours prior to a two step heat treatment (100°C/5h + 150°C/various times). Ageing curves as function of time at 150°C for nondeformed and 10% deformed alloys were recorded by means of tensile testing in an Instron servohydraulic testing machine. The nominal strain rate was $6 \cdot 10^{-3}$ s^{-1}.

Microstructural investigations were made using a Hitachi H800 transmission electron microscope, with the samples taken from the longitudinal-transverse plane of the tensile samples. Thin-foil specimens were made by electropolishing at -15°C with an A7-solution, containing 10% perchloric acid, 20% glycerol and 70% methanol, followed by ion beam milling at 10° tilt for 20 minutes. For the TEM-investigations, micro- and mesoscale characterization was done with emphasis on precipitate size, size distribution and the spatial distribution in the material. Precipitate sizes were measured on enlarged prints of the TEM-micrographs. More than 300 precipitates were measured for each condition.

3. Results

Figure 1a and 1b show the yield stress ($Rp_{0.2}$) as function of ageing time at 150°C for the two alloys in predeformed and non-deformed conditions. Each data point represents the average of two measurements. One observes that the time to reach the peak aged condition is the same for both alloys. The alloy containing zirconium (7108) has a higher yield stress for almost the entire ageing treatment, as compared to the alloy without zirconium. For the longest ageing times (70 hours), however, both of the undeformed alloys showed similar yield stress.

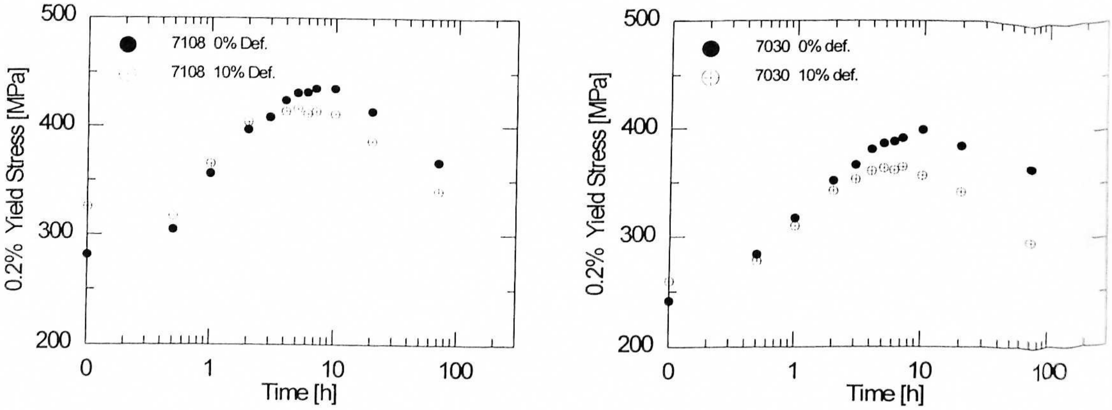


Fig. 1: Yield stress as function of ageing time at 150°C (after room temperature storage for 24 hours and ageing at 100°C/5 hours) for a) the 7108-alloy and b) the 7030-alloy.

The two alloys also demonstrate a qualitatively similar behaviour when one compares the nondeformed and the predeformed conditions: The predeformed materials have a slightly higher yield stress in the initial stages. During ageing, this changes as the peak strength is lower when dislocations have been introduced. One also observes that the peak strength is obtained for shorter ageing times in the predeformed condition as compared to the non-deformed. An interesting observation can be made for the overaged condition: In the 7030 alloy (i.e. the one without zirconium), one can see that the difference between the predeformed and the nondeformed condition increases with increasing ageing time, while this is not the case for the 7108 alloy. For this latter case, the difference remains nearly constant.

The TEM-micrographs shown in Figure 2 for the 7108-alloy, demonstrate the influence of predeformation on the precipitation behaviour. In the undeformed alloy, the precipitates appear to have a more or less rounded shape. When dislocations are introduced into the material, the precipitates are larger, they vary more in size and they also tend to differ more in shape than in the undeformed condition.

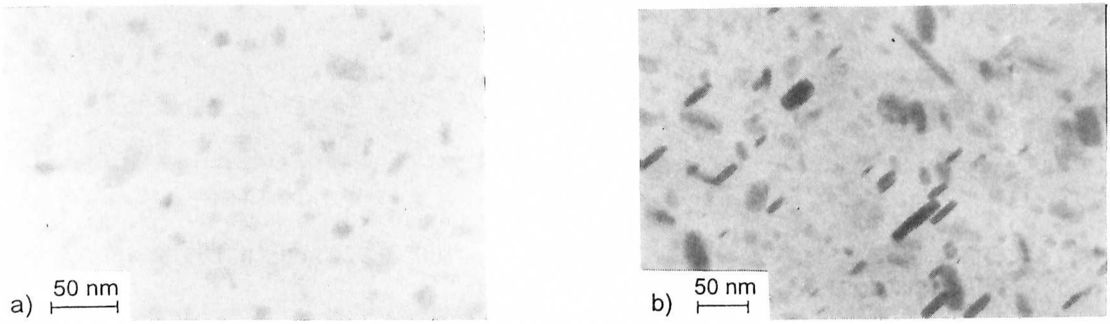


Fig. 2: TEM-micrographs from a) the nondeformed and b) predeformed conditions for the 7108-alloy aged for 70 hours at 150°C.

The influence of predeformation can also be observed if one studies the diffraction images from the two conditions. In Figure 3, the <111> zone-axis for the undeformed and the predeformed material overaged for 70 hours are shown. By using the technique described in [3] for interpretation of diffraction images, one observes that the precipitate diffraction spots are different for the two conditions, showing reflection from η_1-precipitates in the undeformed alloy and from η_2 and η_4-precipitates in the deformed one.

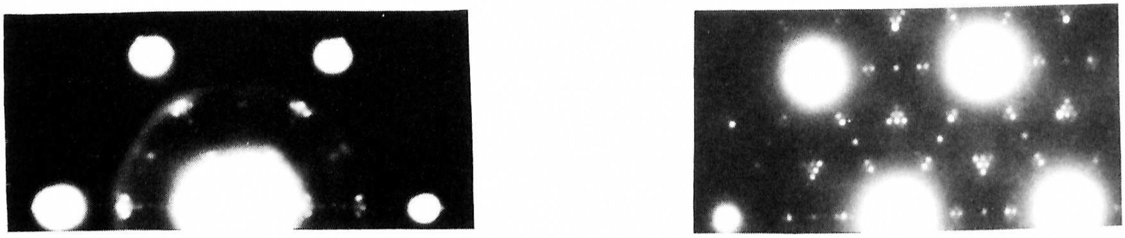
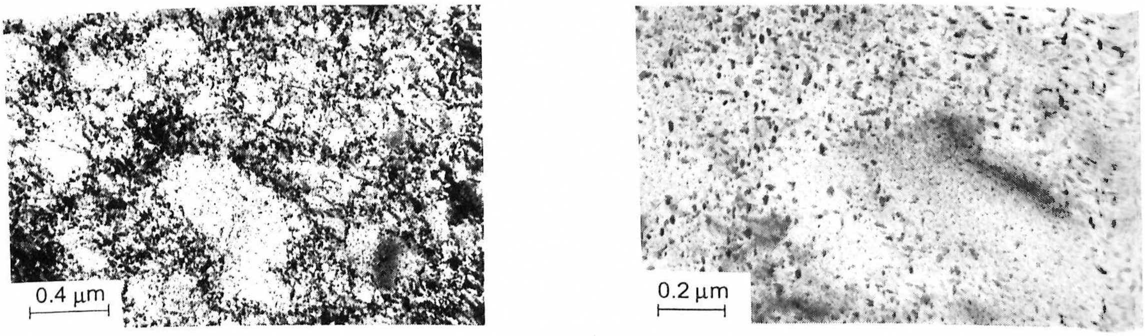


Fig. 3: Diffraction images from the <111> zone-axis of a) the nondeformed and b) predeformed conditions for the 7108-alloy aged at 100°C/5hours + 70 hours at 150°C.

An interesting observation which was made during the present investigation, was the spatial distribution of dislocations and precipitates in the predeformed alloys. By studying the material at a low magnification in the TEM, it was revealed that the dislocations organized themselves into a loose cell structure, consisting of very broad cell walls and with virtually no dislocations in the cell interiors, see Figure 4a. This feature was apparent in both alloys after predeformation. In Figure 4b, it is shown how this influences the precipitation behaviour, i.e. the precipitates in the cell interiors are much smaller than the ones which have nucleated and grown on the dislocations in the cell walls. However, it should be noted that the cell structure could not be observed everywhere in the material. In some locations, one could see an almost even distribution of dislocations. But the cell structure shown in Fig. 4 appeared to be a dominating feature.



a) b)

Fig. 4: Cell structure in the 7030-alloy, aged for 5 hours at 100°C + 20 hours at 150°C. a) Dislocation structure b) precipitate structure.

Measurements of precipitate sizes and size distributions were carried out on enlarged TEM-micrographs. Figure 5a and 5b show the size distributions for the precipitates in the alloys aged for 70 hours at 150°C, demonstrating that a log-normal distribution of the precipitates can be found. The only apparent difference between the various conditions, is that the tail of the size distributions becomes much longer when the material is predeformed before ageing. Quantitative data from the precipitate size measurements are given in Table 2. One observes that both the average size and the maximum size in the predeformed is larger than in the nondeformed alloys. Further, the results demonstrate that the precipitates in the 7108-alloy have a larger size than in the 7030-alloy.

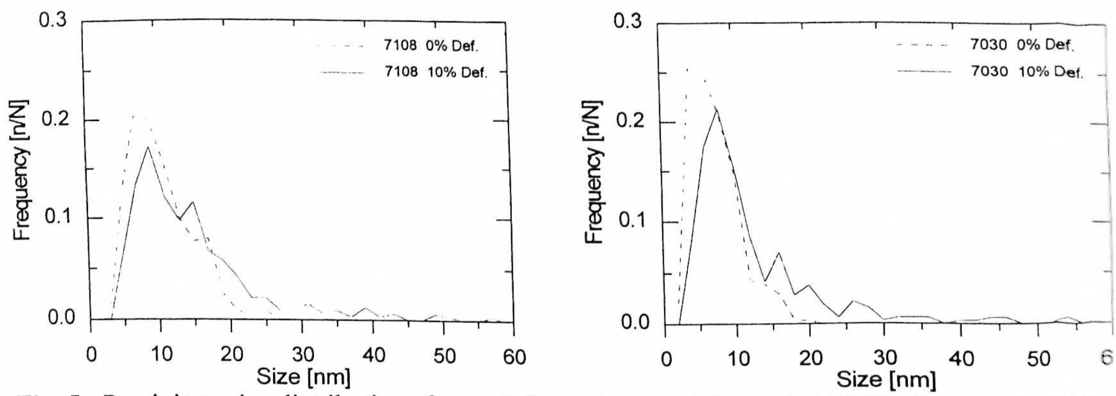


Fig. 5: Precipitate size distributions for predeformed and undeformed a) 7108-alloy and b) 7030-alloy, aged at 100°C/5 hours followed by 70 hours at 150°C.

Table 2 Precipitate sizes after ageing at 100°C/5h + 150°C/70h.

Condition	Avg. size [nm]	STD [nm]	Max. size [nm]	Avg. size/STD
7030 0% def.	6.3	3.3	19.3	1.9
7030 10% def.	11.3	8.5	53.2	1.3
7108 0% def.	9.4	4.3	24.7	2.2
7108 10% def.	13.4	8.6	56.5	1.6

4. Discussion

For the age hardening curves in Fig. 1, the present results can be compared with the one obtained by Poole and Shercliff [1], who also studied the influence of predeformation on two step ageing. In both set of experiments a similar behaviour is found for the ageing kinetics, with one exception: In the present study the ultimate peak strength is lower for the predeformed alloy compared to the undeformed one, which is an effect not observed by Poole and Shercliff [1].

This reduction in peak strength after predeformation can be due to a smaller amount of solute in the present material compared to the alloy studied in [1]. Since the driving force for homogeneous nucleation is lower due to the lower level of supersaturation, the role of hetereogeneous nucleation of precipitates on dislocations may be more critical for this alloy. A higher fraction of the solute therefore goes into the coarse precipitates nucleated on the dislocations. An alternative explanation takes into account that the first ageing step is shorter and takes place at a lower temperature, compared to the study by Poole et al. [1]. The GP-zones formed homogeneously during the first step of the two step ageing process have therefore not become stable (reached the critical size) and they will partly dissolve instead of growing during the second ageing step, with solute draining to the more stable precipitates formed on the dislocations.

The results from the TEM-investigations demonstrate the difference in microstructural evolution for the alloys with and without cold deformation prior to artificial ageing. Since all TEM-observations were made in the overaged material, all precipitates will be of the equilibrium η-phase with the metastable η'-phase being no longer present. One observes that in the predeformed alloy, coarse plate-shaped precipitates are nucleated on the dislocations. By studying the diffraction pattern from the <111> zone-axis, it is possible to identify the different types of η-precipitates which are found in the nondeformed and predeformed cases. One observes that there is a transition from η_1 when no dislocations are present, to η_2 and η_4 when dislocations are present in the material, which implies that the coarser precipitates nucleated on the dislocations are of these two latter types. This is in agreement with the observations by Deschamps et al. [3] in a one step aged ternary Al-Zn-Mg model alloy. Park and Ardell [4] observed a similar transition in precipitate structure when comparing an undeformed alloy in the T73 and T6-tempers. This implies that the dislocations do not act as nucleation sites for η-phases of new crystallographic orientations, they are only increasing the overall overageing kinetics of the material.

The observation that the dislocations arrange themselves into a cell structure is a bit surprising, since such a feature was not been observed in the works by Poole or Deschamps [4]. The reason why a cell structure is formed, is probably due to the lower level of solute elements (especially of Mg) in the present alloys: In 7108, the total amount of alloying elements is 7 wt%, while the one studied by Deschamps et al. [3] contained 8.5 wt% and the 7475-alloy studied by Poole et al. [1,2] had about 10 wt% of alloying elements. This implies that the amount of dynamic recovery occurring during deformation in the material studied here is larger than in the alloys studied in [1,2] and [3], which results in the present cell structure.

During ageing, the particles formed in the cell interior will differ in size from the particles nucleated on the dislocations in the cell walls. One would expect the homogeneously nucleated precipitates in the cell interiors to be of the same size as the ones formed in the undeformed alloy. This was confirmed by doing size measurements in the 7108-alloy aged at 150°C for 20 hours. In the undeformed alloy the particle size was measured to be 6.3 nm while in the cell interiors of the 10% deformed alloy, the precipitate size was found to be 6.6 nm. This indicates that homogenous nucleation is the dominating nucleation mechanism in the cell interiors.

For the size distributions, both the non-deformed and the predeformed conditions can be fitted to a log-normal distribution. Obviously, the long tail in the distribution for the deformed materials are due to the coarser precipitates formed on the dislocations. This influence of predeformation is also demonstrated if one divides the precipitate size with the standard deviation, as shown in Table 2. It is commonly said that the standard deviation is proportional to the average size, and one would therefore expect a constant value [6]. However, from Table 2 one observes that the ratio is reduced when the material is predeformed before ageing.

Comparing the two alloys, one observes that the precipitate sizes are much smaller in the 7030-alloy than in the zirconium bearing 7108. This is a bit surprising, since both alloys have undergone the same thermal history and have nearly the same chemical composition. The effect of zirconium on the early ageing stages of a ternary Al-Zn-Mg alloy have earlier been studied by Mukhopadhyay, Yang and Singh [7]. They claimed that when zirconium was added to the alloy, the free vacancy supersaturation was reduced during quenching and suppressed the formation of stable GP-zones during room temperature ageing. The present results could therefore be interpreted as the addition of zirconium reduces the total number of nuclei, which leads to fewer and coarser η-precipitates during overageing.

5. Conclusion

The present work have demonstrated the difference in ageing behaviour for Al-Zn-Mg-Cu with and without the addition of zirconium. The influence of predeformation on the ageing behaviour has also been investigated. It is found that during predeformation of the two alloys, the dislocations seem to organize themselves into a loose cell structure with broad cell boundaries. This implies that on a meso-scale, there will be local differences in the precipitate sizes. Predeformation accelerates the ageing kinetics, which is reported to be due to heterogeneous precipitation of the stable phase η_2 and η_4 on dislocations. When Zr is added to the material, the precipitates tend to be larger. This may be due to the reduced number of nuclei which results in fewer and coarser precipitates.

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