# Modelling Al<sub>3</sub>Zr Precipitation in An AA7050 Alloy

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#### Abstract

A physical model for predicting precipitation reactions in aluminium alloys has been developed at Corus Research, Development & Technology. The model is based on the classical nucleation theory and diffusion-controlled growth. In this work the model was applied to predicting Al<sub>3</sub>Zr precipitation in AA7050 alloy. To determine parameters in the model, samples taken from a DC-cast AA7050 ingot, were isothermally annealed at different temperatures. The number density and the size of Al<sub>3</sub>Zr-precipitates in the annealed samples were measured using transmission electron microscopy. A good agreement between the measured microstructural parameters and these predicted by the model was found.

#### 1. Introduction

The aircraft industry is constantly striving for improved materials that enable higher performance at a reduced cost. With the development of high speed milling machines and the improved properties of thick plates, large structures machined from a thick plate can now replace forged components or built-up structures, which significantly reduces the cost by minimising the number of components and joints. This in turn results in a demand for even thicker plates that are mainly made from AA7050 alloy.

The thicker the plate, the more difficult it is to obtain a favourable grain structure. For a thick plate, the important processing steps for controlling the grain structure are homogenisation and hot rolling. During homogenisation of AA7050, the Al<sub>3</sub>Zr-dispersiods are formed that are needed for controlling recrystallisation. The effectiveness of the dispersoids in controlling recrystallization depends on their size, spacing and distribution. By refining the homogenisation conditions the effectiveness of the dispersoids may be increased.

In this paper we modeled the  $Al_3Zr$  dispersoid precipitation as a function of temperature. The quantitative knowledge on the  $Al_3Zr$ -dispersiods will help us to optimize the grain structure in the final product.

#### 2. Model Description

The precipitation process consists of two simultaneously occurring physical processes: nucleation and growth of second phases. The nucleation rate can be described by the following equations based on the methodology of Kampmann and Wagner<sup>[1]</sup>:

$$\frac{dN}{dt} = (N_{tot} - N_t) \cdot \frac{D}{a^2} \cdot \frac{C_m^t}{C_p} \cdot \exp(-\frac{\Delta G^*}{kT})$$
(1)

Where N<sub>tot</sub> is the total number of nucleation sites, N<sub>t</sub> is the number of precipitates at the time t, D = D<sub>0</sub>\*exp(-Q/RT) is the diffusion coefficient, *a* is the lattice parameter of the matrix,  $C_m^t$  is the instantaneous average solute concentration, C<sub>p</sub> is the solute concentration in the precipitate and  $\Delta G^*$  is the critical energy barrier for the nucleation.

The critical energy barrier for the nucleation,  $\Delta G^{*}$ , is given by

$$\Delta G^* = \frac{4}{3} \cdot r^{*2} \cdot \gamma \tag{2}$$

where  $r^*$  is the critical radius of nucleus and  $\gamma$  is the interfacial energy of the precipitate-matrix interface.

The critical nuclei radius, r\*, is calculated with

$$r^* = \frac{2 \cdot \gamma}{(\Delta G_c - \Delta G_s)} \tag{3}$$

Where  $\Delta G_c$  is the chemical driving force for precipitation and  $\Delta G_s$  is the misfit strain energy.  $\Delta G_c$  can be calculated using the thermodynamic database COST507 and  $\Delta G_s$  is neglected here.

The growth of precipitates is described together with <sup>[2]</sup>

$$\frac{dr}{dt} = \frac{D}{r} \cdot \frac{C_m^t - C_i}{C_p \cdot \frac{\rho_p}{\rho_m} - C_i}$$
(4)

where  $C_i$  is the solute concentration at the interface and  $\rho$  is the mass density. The equation above can be used to model the coarsening of the precipitates as well. From this equation, all precipitates larger than the average one will grow whereas precipitates smaller than the average one will shrink once the growth process is completed.

The solute concentration at the interface can be calculated by

$$C_{i} = C_{m}^{e} \cdot \exp(\frac{2\gamma \cdot V_{at}}{k \cdot T} \cdot \frac{1}{r})$$
(5)

Where  $C_m^e$  is the equilibrium matrix solute concentration and V<sub>at</sub> is the atomic volume. In this work  $C_m^e$  was calculated using the thermodynamic database COST507. Since the growth rate of each precipitate depends on its radius, it is necessary to separately calculate the growth rate of every individual precipitate. To do this, the radii of particles that nucleate in each time interval are tracked separately.

$$C_m^t = C_m^0 - \frac{\rho_p}{\rho_m} \cdot f_p \cdot C_p \tag{6}$$

where  $f_{\text{p}}$  is the volume fraction of precipitates and can be calculated by

$$f_p = \sum n_i \cdot \frac{4\pi}{3} \cdot r_i^3 \tag{7}$$

## 3. Experimental

Samples used for this work were taken from an industrially cast ingot AA7050 with 0.12%Zr. The average composition of AA7050 according to the Aluminium Association is: 6.2%Zn, 2.3%Mg, 2.3%Cu, 0.12%Si and 0.15%Fe.

The cast sample was evaluated for the segregation of Zr and other elements during solidification in a Cameca SX50 electron microprobe (EPMA) operated at 15 kV. Quantitative compositional measurements were made by line scans across the solidification dendrites.

The cast samples were isothermally homogenized at 3 temperatures (475, 500 and 525°C) for up to 10 hours. Following the heat treatment the samples were quenched in cold water to prevent any precipitation of other phases during cooling. Note that 500°C and 525°C are above the temperature normally used in industry.

For TEM investigation, 3 mm diameter discs were cut from the bulk samples by spark erosion machining. Thin foil specimens were prepared by electro-polishing using the Tenupol-3 and followed by a further thinning using Gatan PIPS (Precision Ion Preparation System). The TEM examinations were carried out on a Philips 400EM interfaced with an EDA analytical facility. Since the  $AI_3Zr$  precipitates are coherent with the AI matrix, dark field images using the extra reflection from the precipitates were used for the measurement of the precipitate size and density.

### 4. Results and Discussion

### 4.1. Electron Microprobe Analysis

EPMA mapping of the polished sample showed that the as-cast material consisted of a dendritic microstructure (see Figure 1). Cu, Zn and Mg-levels are higher at the inter-dendrite regions while Zr is enriched in the centers of the dendrites. Quantitative line scans indicated that the Zr concentration at the centers of the dendrites (0.23 wt.%) is about 2 times as high as the nominal one (0.12 wt.%). The Zr level is reduced significantly near to the grain boundaries or inter-dendritic regions.



Figure 1: EPMA X-ray map (0.512mmx0.512mm) and quantitative line scan (Line 1).

### 4.2 TEM Precipitation Study

It is well known that homogenization of commercial 7xxx alloys leads to the precipitation of the metastable L1<sub>2</sub> Al<sub>3</sub>Zr phase as fine, spherical dispersoids <sup>[3-5]</sup>. Thin foil TEM examination revealed that in the homogenized samples the dispersoids were inhomogeneously distributed within grains due to the Zr segregation. The grain centers contain a high density of small, spherical Al<sub>3</sub>Zr precipitates while areas near to grain boundaries have fewer or no precipitates. Therefore, only the center parts of the grains were selected to measure the size and density of the dispersoids. On each sample the average size of the precipitates and the precipitate number density were measured from at least five grains. The thickness of the TEM-foils was estimated to be about 250 nm.

Figure 2 shows three dark field images taken at the centre of the grains in samples homogenized at 500°C up to 9hr40min. The average size of the Al<sub>3</sub>Zr precipitates remained almost constant while the particle number density increased steadily with the homogenisation time.

Precipitation study indicated that homogenization at 525°C resulted in a similar particle size and number density of dispersoids as at 500°C for a similar period of time. However, homogenization at 475°C resulted in smaller and much less precipitates than at 500°C and 525°C for a similar period of time.

# 4.3 Model Calibration and Prediction

In the model many parameters need to be included. The values for these parameters used for the modelling of Al<sub>3</sub>Zr-precipitation are given in Table 1.

D <sub>0</sub>	Q	а	V <sub>at</sub>	ρm	ρρ	
M²/s	J/mol	m	m <sup>3</sup>	kg/m <sup>3</sup>	kg/m <sup>3</sup>	
7.28E-2	242000	4.08E-10	1.25E-29	2702	3200	

Table 1: Values for the parameters used in the model



Figure 2: TEM dark field micrographs showing the size and distribution of the dispersoids homogenised at 500°C.

The values for the D<sub>0</sub> and Q were taken from reference 6. The values for C<sub>0</sub> and  $\Delta H$  were obtained as follows: firstly the equilibrium Zr concentration as a function of temperature was calculated using FacSage and the thermodynamic database COST507 and then equation 4 was fitted to the data.

In the precipitation model there are two tuning parameters, the total number of nucleation sites N<sub>tot</sub> and the interfacial energy of the nuclei  $\gamma$ . The values of these two parameters were found by fitting the model to the measured precipitate radius and the number density in the samples homogenized at 500°C for the three different times. The best agreement between the predicted and measured particle radius and number density at 500°C was obtained with N<sub>tot</sub> = 8.5E20/m<sup>3</sup> and  $\gamma$  = 0.18J/m<sup>2</sup> (Figure 3). To check the predicting capability of this model, the previously found values of the two tuning parameters were used to predict the precipitate radius and number density in samples homogenized at the two other temperatures. The reasonable agreement between the measured and predicted values indicates the good predicting capability of this model (see Figure 4).

#### 5. Conclusions

The precipitation of  $AI_3Zr$  in as-cast AA7050 during homogenisation has been studied using electron microprobe analysis and TEM. A physical model has been developed to describe the kinetics of the precipitation. The results from TEM have been compared with the model prediction. The following points can be made:

- 1. There is a strong Zr segregation towards the dendritic centres.
- 2. The Zr-segregation leads to inhomogeneous distribution of Al<sub>3</sub>Zr-dispersoids within a grain.
- 3. A physical model has been developed to describe the kinetics of the precipitation, the evolution of precipitate size and number density during homogenisation treatment. Once properly tuned, the model has a good predicting capability.



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