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Homogenization Model for 7xxx Aluminum Alloys

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A model for homogenization has been developed for 7xxx aluminum alloys based on extensive experimental work evaluating the effects of heat-up rate, soak temperature and soak time on the progress of homogenization. Special attention was paid to the heat-up rate effect on the occurrence of non-equilibrium eutectic melting. Constituent phases and microstructure were characterized by Optical Microscopy and Scanning Electron Microscopy. The amount of residual soluble constituents was quantified by Differential Scanning Calorimetry; solute microsegregation was quantified by Electron Microscopy Probe Analysis. An important feature incorporated in the model is its capability to capture the evolving "critical length" scale from dendritic arm spacing, early in the homogenization process when the kinetics are dominated by solute redistribution, to the grain dimension during the latter stage when the dissolution and coarsening of constituents becomes the dominant factor. The homogenization model enables computer-aided design and optimization of homogenization practices and reduction of production cost and has been implemented at Alcoa plants. On-going work is being performed to extend this model to other heat-treatable aluminum alloys.

Keywords: *Heat-Treatable Aluminum Alloys; Homogenization; Phase Transformation; Computer Model; Microstructure Characterization.*

1. Introduction

The microstructure of most alloys in the as-cast condition is a heterogeneous cored dendritic structure, with eutectic intermetallic phases concentrated at grain and dendritic boundaries. Because of the relatively low ductility of the intergranular and inter-dendritic constituent particles, as-cast structures generally have poor workability. One of the principle objectives of ingot preheating or homogenization process is to dissolve intermetallic phases so as to improve the workability for the downstream hot working process.

Basic thermodynamic concepts about homogenization are presented with reference to a schematic binary phase diagram [1] as shown in Fig.1. The solubility of a constituent phase increases with temperature until it reaches maximum solubility at the eutectic temperature. If the solute content is within the maximum solubility, it is possible to completely dissolve all the constituent phase by homogenizing at a temperature between the solvus and solidus. To avoid the possibility of nonequilibrium eutectic melting (EM), either the upper soak temperature should be below the eutectic temperature or the time spent below the eutectic temperature should be sufficient to dissolve the constituent phase. If the solute content exceeds maximum solubility, it is impossible to completely dissolve all the constituent phase. For commercial 7xxx aluminum alloys, the multi-component phase diagram is more complicated than the binary one shown in Fig.1, but the basic thermodynamic concepts still apply. The kinetics of the homogenization process are determined by the dissolution rate of soluble constituent phases and the diffusion rate of solutes across dendrites and grains, both of which are favored by high temperature. It is desirable to achieve high temperatures as quickly as practicable in planning the thermal paths for homogenization practice development. However, the metallurgical phenomenon of eutectic melting imposes restrictions on the metal heatup rate and the soak temperature.

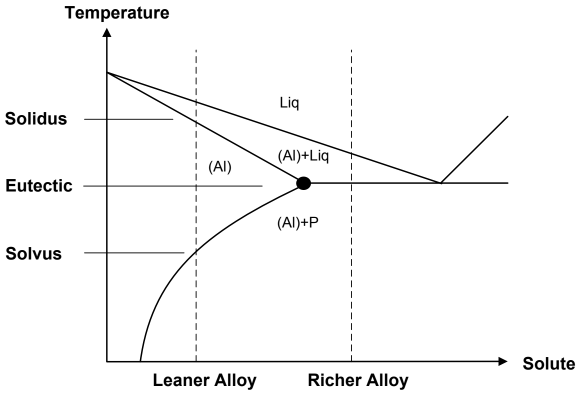


Fig. 1 Schematic phase diagram of a binary alloy.

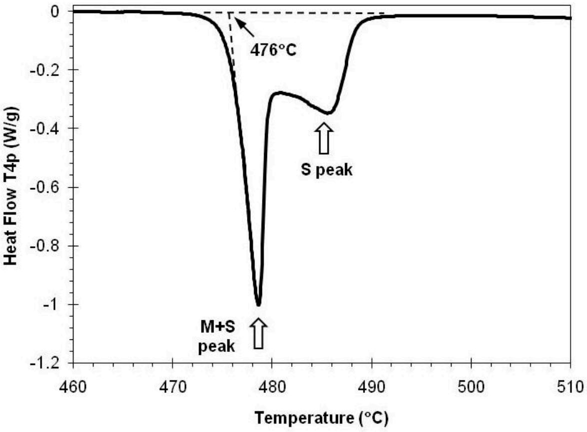


Fig. 2 DSC trace of as-cast 7050 ingot sample.

The objective of this work is to develop a computer model for the homogenization process of 7xxx aluminum alloys. This model needs to be based on physics and should have the capability to predict the evolution of constituent phases for an arbitrary thermal path so it can enable intelligent process design and optimization and can be used as a process monitor/control tool in production.

2. Homogenization Experiments

2.1 Ingot Samples

All the specimens used in the following experiments were extracted from the quarter thickness (T/4) position of a slice of as-cast and stress-relieved commercial grade AA7050 ingot produced by Alcoa. The nominal composition of 7050 alloy is shown in Table 1.

Table 1 Nominal Composition of AA7050

Element	Fe	Si	Cu	Mg	Zn	Zr	Al
Composition (wt%)	0.06	0.07	2.3	2.25	6.2	0.11	Bal.

The as-cast ingot samples were characterized by a variety of methods to provide quantitative input parameters for the homogenization model. They were examined by Optical Metallography (OM) for grain and dendritic structure and Scanning Electron Microscopy (SEM) for phase identification. As-cast grain size, measured using the linear intercept method, is ~150 μm, and dendrite arm spacing (DAS) is ~50 μm. SEM examination reveals that the as-cast structure contains the following phases: M–phase ($Mg(Zn,Cu,Al)_2$), S–phase (Al_2CuMg), Al_7CuFe, and Mg_2Si. A majority of the constituent phases are distributed as agglomerated clusters at grain vertices and the average cluster size is ~5 μm. As shown in Fig. 2, the Differential Scanning Calorimetry (DSC) trace in the as-cast condition contains two endothermic peaks associated with the following EM reactions: Al+M+S ↔ Liq, and Al+S ↔ Liq. The total enthalpy is ~18 J/g and the onset temperature of the M+S EM is ~476°C. The samples were also analyzed by Electron Microscopy Probe Analysis (EMPA) for solute distribution profiles. As shown in Table 2, the solutes are highly segregated in the as-cast condition.

2.2 Experimental Procedure

Lab-scale homogenization experiments were conducted using small 7050 ingot samples, about 25mm×25 mm×12 mm in dimension. Four separate trials were conducted, each following a different time-temperature path as shown in Fig. 3: (a) slow heatup to 482°C (the heatup time is 18 hr) and soak for 36 hr; (b) fast heatup to 482°C (the heatup time is 9 hr) and soak for 12 hr; (c) slow heatup to 477°C and soak for 36 hr; (d) slow heatup to 487°C and soak for 12 hr. Such experimental design was aimed to study the effects of heatup rate, soak temperature and soak time on phase evolution. In experiments A and B, samples were pulled out of the furnace during both heatup and soak and were immediately quenched in cold water. In experiments C and D, samples were only pulled during the soak and immediately quenched.

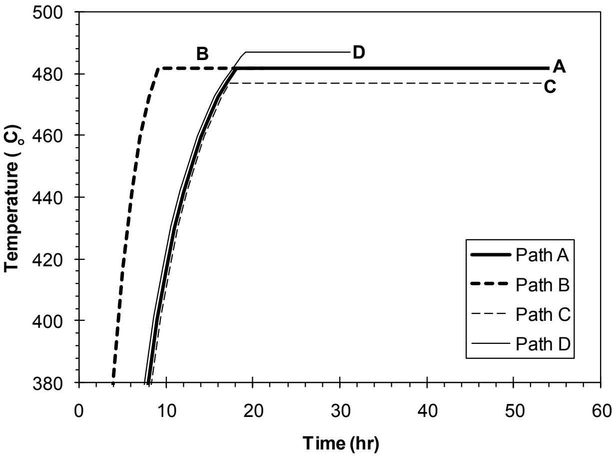
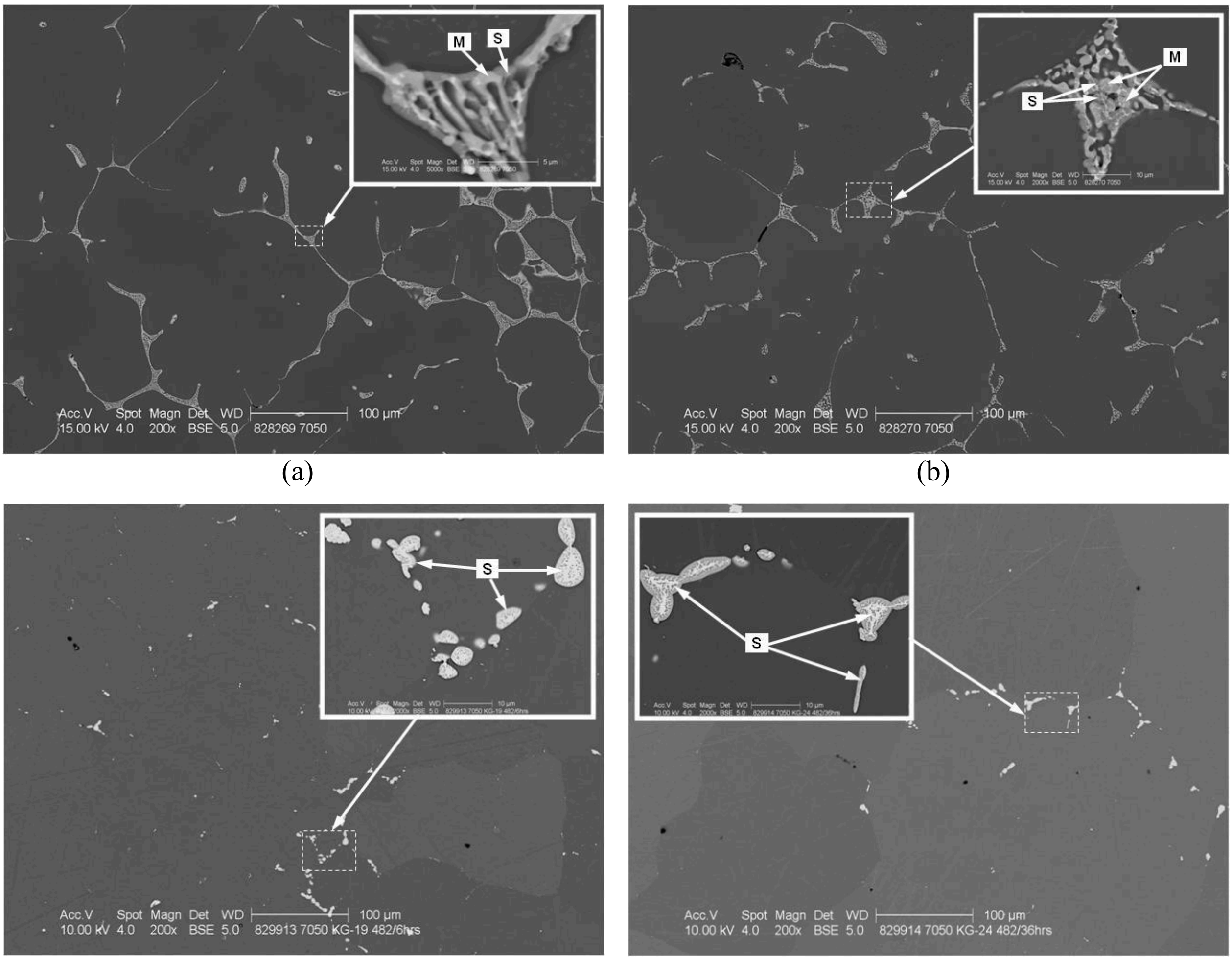


Fig. 3 Lab-scale homogenization experiments for 7050 ingot samples following different time-temperature paths.

2.3 Experimental Results

All of the quenched samples were analyzed by DSC for the amount of residual constituents. The total enthalpies of residual soluble constituents (M and S) are plotted against time in Fig. 5(a, b). As shown in Fig. 5(a), for experiments A and B, the total enthalpy decreases quickly after the temperature rises above 400°C during heatup, and decreases relatively slowly during soak and seems to approach a minimum value. The effect of soak temperature on the amount of residual enthalpy is evident in Fig. 5(b), with lower soak temperature resulting in higher residual enthalpy. The initial decrease in the total enthalpy is mainly a result of dissolution of M phase, since M phase has a lower solvus than S phase in 7050, which is reflected in the DSC trace as a reduction in the M+S peak. The dissolution rate of M phase is affected by the heatup rate: in the fast heatup experiment B, the M+S peak disappears around the time the temperature rises to ~476°C; whereas in the slow heatup experiment A it is completely gone at a much lower temperature. Since the onset temperature of the M+S peak is ~476°C, there is little chance for EM to occur during the fast heatup and no chance for EM to occur during the slow heatup. This is confirmed by OM examination of the quenched samples.

Selected samples from experiment B were examined by SEM to understand microstructural evolution during homogenization. Fig. 4 shows SEM images for the following 4 samples: (a) 427°C during heatup; (b) 466°C during heatup; (c) 6 hr soak at 482°C; (d) 36 hr soak at 482°C. The low-magnification (200×) SEM images show the overall microstructure, while the inserted small high-magnification (2000× or 5000×) images illustrate the detailed features at grain vertices. As shown in Fig. 4(a), dendritic structure and lamellar-like eutectic features at grain vertices, which are typical as-cast microstructures, are clearly visible in the sample quenched at 427°C during heatup. This is because the dissolution rate of constituents is very slow below 427°C due to low solute diffusivity. As shown in Fig. 4(b), some thin films at grain boundaries have dissolved in the sample quenched at 466°C during heatup. The most significant change occurs at grain vertices where the lamellar-like eutectic features are replaced by clusters of spheroidized particles, while the boundaries of such clusters seem to have moved little. As shown in Fig. 4(c), after 6 hr soak at 482°C the sample contains only isolated clusters of S and Al_7Cu_2Fe particles at grain vertices. These S particles seem to have coarsened further after 36 hr soak at 482°C, as shown in Fig. 4(d).



(a) (b)

(c) (d)

Fig. 4 SEM images of 7050 ingot samples quenched during the lab homogenization experiment B: (a) 427°C during heatup; (b) 466°C during heatup; (c) 6 hr soak at 482°C; (d) 36 hr soak at 482°C.

Two of the samples, soaked for 6 hr and 36 hr at 482°C, were also analyzed by EMPA for solute microsegregation, and the results are shown in Table 2. Compared with the as-cast sample, the microsegregation ratios of both Cu and Mg have reduced significantly in the homogenized samples, and the ratios decrease further towards 1 with longer soak time. The homogenization process is a coupled process of constituent dissolution and solute redistribution. The experiment results were then used to develop and validate the homogenization model.

Table 2 EMPA Data of Solute Compositions in 7050

Process Condition	**Cu**			**Zn**			**Mg**
	Grain vertex	**Grain center**	**Ratio**	**Grain vertex**	**Grain center**	**Ratio**	**Ratio**
As-cast	1.77 wt%	0.49 wt%	3.61	6.33 wt%	3.92 wt%	1.61	1.76
6 hr 482°C	2.43 wt%	1.88 wt%	1.29	-	-	-	1.05
36 hr 482°C	2.45 wt%	2.18 wt%	1.12	-	-	-	1.03

3. Homogenization Model

A state-variable approach [2, 3] was adopted in developing the homogenization model, in which the alloy microstructure is described by a set of state variables (e.g., amount and size of constituents) and the microstructural evolution is governed by a set of physics-based mathematical equations.

3.1 Model Algorithm

A few assumptions were made to simplify the homogenization model. First, 7050 alloy is simplified as a quaternary Al-Cu-Mg-Zn system. Effective Cu and Mg solutes are used in the model; this is to account for the reduction in effective solutes due to the formation of Al_7Cu_2Fe and Mg_2Si phases which are insoluble at the typical homogenization temperatures for 7050 alloy. Only two soluble phases, M and S, are included in the system besides the aluminum matrix phase. Second, it is assumed that the as-cast grain structure has an equi-axed shape. Third, it is assumed that all of the constituent phases are distributed as spherical clusters at grain vertices, and the cluster size does not change during homogenization.

The dissolution rate for the ith constituent phase is

$$\frac{df_i}{dt} = 3f_i \frac{D_j}{r^2} \frac{C_j - C_j^e}{C_j^p - C_j^e} \text{ (for i = 1 to 2, j = 1 to 3),} \tag{1}$$

where f is the volume fraction of constituent phase, D is solute diffusivity, r is the cluster size, C is the solute concentration in the matrix phase, C^p is the solute concentration in the constituent phase, C_e is the equilibrium solute concentration at the surface of the constituent phase, and the sub index j refers to solute elements. Solute diffusion in the matrix phase is calculated by solving the following equation using the finite difference method:

$$\frac{dC_j}{dt} = D_j \nabla^2 C_j \text{ (for j = 1 to 3).} \tag{2}$$

The grain volume is divided into 5 equal-volume concentric spheres, represented by V_k, where k = 1 to 5. As constituent particles dissolve, solutes are dumped in the grain boundary volume V_5 and diffused across the grid to the center volume V_0.

It is often suggested that the time for solute homogenization is determined by solute diffusivity and the DAS. In the present case this may be true initially, but in the long term the rate of homogenization is determined by the grain size rather than by the DAS. After initial homogenization, the wavelength of solute segregation is determined by the spacing of dissolving eutectic constituents rather than the initial microsegregation within dendrites. As shown in Fig. 4, the spacing of eutectic clusters is at the grain scale. EMPA analysis also confirms the wavelength of solute segregation is at the grain scale in the late stage of homogenization. We believe the control of homogenization by grain size rather than by the DAS is a general characteristic of high-solute alloys. To capture this phenomenon of changing diffusion scale, a critical diffusion distance L is introduced. The evolution equation of the critical diffusion distance is expressed as:

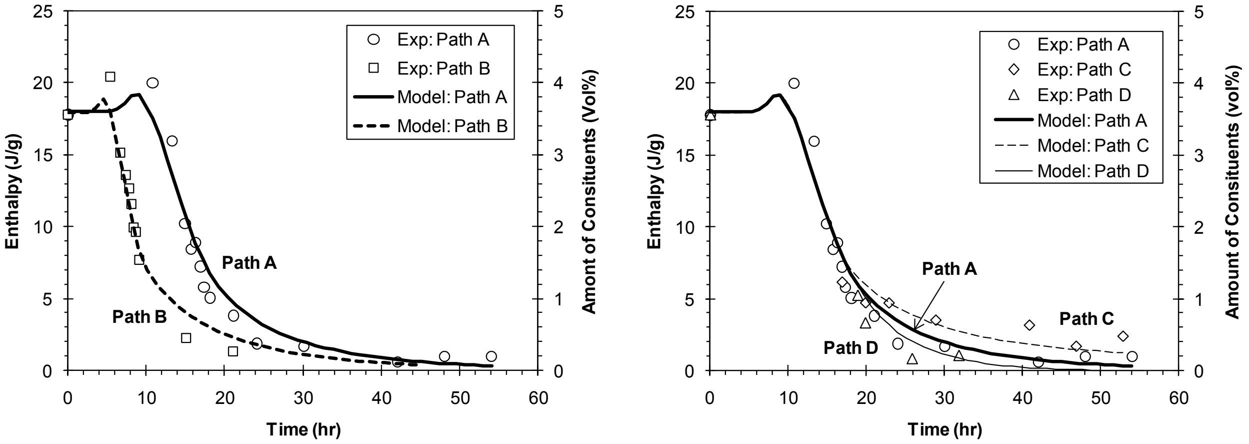
$$\frac{dL_j}{dt} = 4\pi^2 (d - L_j) D_j \left(\frac{1}{\lambda^2} - \frac{1}{d^2} \right) \text{ (for j = 1 to 3),} \tag{3}$$

where d is grain size and λ is DAS. Initially, $L_j = \lambda$, which means the diffusion scale of all solute elements equal the DAS. During homogenization, the diffusion scale increases with time, but at a rate which is affected by the solute diffusivity. Eventually, all of the diffusion distances will evolve towards the grain size d.

The model has the following evolving state variables: f_i (i = 1 to 2), $C_{j,k}$ (j = 1 to 3, k = 1 to 5) and L_j (j = 1 to 3). For simplicity, the cluster size r is kept constant. The model also needs inputs of overall alloy composition and as-cast microstructure (e.g., grain size, dendrite arm spacing, constituent cluster size and solute microsegregation profiles) as the initial state. The model reads in an arbitrary time-temperature profile and predicts the evolution of constituent phase amount vs. time. The homogenization model is coupled with commercial software ThermoCalc®, to compute equilibrium composition at the matrix/constituent interface for a given temperature. Use of this allows easy expansion of the model to other 7xxx alloys and other alloy series.

3.2 Simulations and Comparison with Experiments

Most of the input parameters for the homogenization model are derived from the experimental characterization results of the as-cast 7050 ingot samples, such as grain size, DAS, eutectic cluster size, and initial solute microsegregation profiles. An empirical conversion formula (5 J/g ~ 1 vol%) is used to estimate the initial volume fraction of soluble constituents. The total enthalpy of the as-cast sample is ~18 J/g and it is assumed that the initial total amount of constituents is ~3.6 vol%: ~1.6 vol% M and ~2.0 vol% S. Simulations were conducted using the time-temperature paths used in the experiments, except that the soak time is extended to 36 hr in all four cases. The simulation results are shown in Fig. 5, overlaid on the experimental data points. The model predictions of phase evolution history generally have good agreement with the experiments. As shown in Fig. 5(a), the model correctly predicts the effect of different heatup rate on phase dissolution during heatup. The model slightly over-predicts the amount of residual constituent in the early soak stage in both cases. As shown in Fig. 5(b), the model captures the effect of different soak temperatures on the amount of residual constituents.



(a) (b)

Fig. 5 Homogenization experimental results compared with model predictions on evolution of constituent phases versus time following different time-temperature path: (a) slow heatup path A vs. fast heatup path B; (b) same heatup rate but different soak temperatures.

4. Conclusion

A homogenization model has been developed, which can predict the changes of soluble constituent phases (M and S) during homogenization of 7050 ingot, and is suitable for process control by integration of time-temperature data. The model has been developed and validated using lab-scale homogenization experiments. It has been demonstrated that this model is capable of predicting the effects of heatup rate, soak temperature and time on the homogenization process. Such a model could be used as a process monitor/control tool in production, thereby enabling reduction in time and energy for 7xxx ingot homogenization. This model is being extended to other heat-treatable aluminum alloys.

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