Available online at www.sciencedirect.com
ScienceDirect
Trans. Nonferrous Met. Soc. China 27(2017) 747−753
Transactions of Nonferrous Metals Society of China
www.tnmsc.cn

Microstructure evolution during homogenization of Al−Mg−Si−Mn−Fe alloys: Modelling and experimental results

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Received 3 May 2016; accepted 8 December 2016

Abstract: Microstructure evolution during the homogenization heat treatment of an Al−Mg−Si−Fe−Mn (AA6xxx) alloy was investigated using a combination of modelling and experimental studies. The model is based on the CALPHAD-coupled homogenization heat treatment model originally developed for AA3xxx alloys (i.e., Al−Mn−Fe−Si). In this work, the model was adapted to the more complex AA6xxx system (Al−Mg−Si−Mn−Fe) to predict the evolution of critical microstructural features such as the spatial distribution of solute, the type and fraction of constituent particles and dispersoid number density and size distribution. Experiments were also conducted using three direct chill (DC) cast AA6xxx alloys with different Mn levels subjected to various homogenization treatments. The resulting microstructures were characterized using a range of techniques including scanning electron microscopy, electron microprobe analysis (EPMA), XRD, and electrical resistivity measurements. The model predictions were compared with the experimental measurements, and reasonable agreement was found.

Key words: AA6xxx alloy; homogenization heat treatment; mathematical modelling; CALPHAD; diffusion

1 Introduction

The aluminum alloy, AA6082, is commonly used as extrusion alloy due to its good combination of formability and corrosion resistance. Billets are mainly produced by direct chill (DC) casting and may have a range of Mn and Fe contents. The non-equilibrium nature of solidification (i.e., relatively high cooling rates and growth velocities) leads to micro-segregation and formation of a wide range of intermetallic phases [1]. These ingots have to undergo a homogenization treatment before extrusion to eliminate micro-segregation, modify the intermetallic phase type and morphology to improve extrudability [2]. It is apparent that microstructure changes such as the transformation of large constituent particles, the formation of dispersoids and the precipitation/dissolution of precipitates during homogenization influence the downstream extrusion behavior and the subsequent microstructure development [3−5]. The development of microstructure and the effect on final properties are influenced by both alloy additions and heat treatment parameters [6].

In 6xxx alloys, α-Al(MnFe)Si, β-AlFeSi and Mg_2Si constituent particles form during solidification [7,8], and can be characterized using EDX and EBSD in the as-cast condition [9]. The first phase to form is α-Al(MnFe)Si which may or may not contain Mn or Fe depending on the chemistry. COOPER et al [10,11] examined the crystal structure of this α-Al(MnFe)Si phase in the Al−Mn−Si system where this phase was found to have a simple cubic structure [10]. However, the phase changes to a BCC structure when Fe replaces Mn [11]. In contrast, the β-AlFeSi phase has sharp boundaries and is poorly bonded with the Al matrix which leads to poor hot workability [12]. A transition from plate like β-AlFeSi phase to a more spheroidized α-Al(FeMn)Si phase has been reported after homogenization [13]. It is believed that the presence of the α-Al(FeMn)Si phase improves surface finish and extrudability [14]. ZAJAC et al [14] found that Al−Mg−Si alloys alloyed with Mn greatly accelerate this transformation during homogenization. The transformation mechanism from β-AlFeSi phase to spheroidized α-Al(FeMn)Si phase was proposed to

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DOI: 10.1016/S1003-6326(17)60085-2

initiate with the nucleation of the α phase on top or side of the β phase during homogenization [15,16]. Finally, the Mg_2Si phase dissolves very quickly when the alloy is held above its solvus temperature dependent on the composition [17], but may act as nucleation sites for dispersoids during the ramp heating to the homogenization temperature [18].

Turning to the dispersoid phase, DOWLING and MARTIN [6] studied two Fe-free Mn-bearing 6xxx alloys with different Mn contents and found that the dispersoids are incoherent α-$Al_{12}Mn_3Si$ particles. In the case of Fe-containing alloys, the crystal structure of these dispersoids depends on the mole ratio of Mn/Fe [6,19], i.e., simple cubic structure is found for higher Mn/Fe mole ratios while a lower Mn/Fe mole ratio favors the BCC structure. LODGAARD and RYUM [18] studied the sequence of precipitation in Al−Mg−Si alloys with different Mn contents. They found that α-Al(MnFe)Si dispersoids heterogeneously nucleate on "u-phase" which is an intermediate or transition phase precipitate of Mg_2Si during heating to the homogenization temperature.

The above observations indicate that the transformation of primary constituent particles (particles of 0.5−5 μm) and the formation of dispersoids (20−200 nm) are both affected by the alloying and homogenization scenarios. Thus, it would be desirable to have a chemistry dependent homogenization model predicting the evolution of these particles. Such a model was reported by DU et al in Ref. [20] where a CALPHAD-coupled homogenization model was developed and validated for AA3xxx (Al−Mn−Fe−Si alloys). The current study is an extension of the model to the more complex AA6xxx (Al−Mg−Si−Mn−Fe) alloys. Our aim is to examine the predictive power of the model for the design of 6xxx alloys and heat treatment parameters using a detailed comparison to experimental measurements.

2 Model description

The multi-scale homogenization model which has been adapted to the current study was previously described in Ref. [20] and as such only a brief summary is given below. It consists of a macroscopic 1D solute diffusion equation for the growth of constituent particles and their transformation and a microscopic based model to describe dispersoid nucleation and growth. The finite volume method described in Ref. [21] is employed to solve the diffusion equation at the scale of the dendrite arm spacing:

$$\frac{\partial x_i^m}{\partial t} = \nabla \cdot (D_i \nabla x_i^m) \quad (1)$$

where x_i^m is the solid solution level of solute i, and D_i is the diffusion coefficient of the diffusion species including Mg, Si, Mn and Fe. An interface cell always separates the matrix and the inter-granular mixture cell.

The microscopic model is the KWN model described in Ref. [22]. It is applied to each volume element of the 1D domain to capture the precipitation kinetics resulting from the variation in local chemistry. The main assumptions adopted in the extended model are as follows.

1) Mn-bearing dispersoids are of spherical shape and their growth/dissolution is solely controlled by diffusion. Mg_2Si particles are not considered, i.e., they are assumed to dissolve at homogenization temperatures of interest.

2) Local equilibrium modified by the Gibbs−Thomson effect prevails at the precipitate−matrix interface and in interdendritic regions.

3) The diffusion field surrounding each dispersoid is at quasi-steady state.

These two models are tightly coupled and involve two length scales. The splitting method adopted in Ref. [20] was employed in this work to deal with the coupling. Readers are referred to Ref. [20] and the references therein for the complete details. The model is also extended to study the effect of Cr on homogenization in AA3xxx alloys [23].

The input parameters for the model are the alloy composition, the thermal history, the phase diagram (thermodynamic database Thermocalc 4.1 with TTAL6), thermo-physical parameters such as diffusivities and the interfacial energy and the number density of nucleation sites. The model can then predict the evolution of dispersoids free zone (DFZ), constituent particle type and fraction, solid solution solute levels and size distribution of dispersoids. The diffusivity of the alloying species (Mn, Fe, Si and Mg) was taken from the work of DU et al [24]. The interfacial energy used in the simulation is 0.08 J/m^2. After several runs of fitting, the number of heterogeneous nucleation sites was chosen as 2.5×10^{19} m^{-3}. The secondary dendrite arm spacing is about 20 μm based on measurements in various as-cast samples with different Mn contents.

3 Experimental

3.1 Materials and heat treatment

Three as-cast Al−Mg−Si alloys were prepared as billets with 100 mm in diameter and 300 mm in length by the Rio Tinto Aluminum Research and Development Center (ARDC) in Jonquiere, Quebec. The chemical compositions of these alloys are listed in Table 1. The chemical compositions for alloys 1, 2 and 3 are designed to investigate the effect of Mn on microstructure changes