Thermochimica Acta 589 (2014) 174–182

Contents lists available at ScienceDirect

Thermochimica Acta

journal homepage: www.elsevier.com/locate/tca

Evaluation of formation of intermetallic compounds in Al2024 alloy using thermal analysis technique

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ARTICLE INFO

Article history:
Received 7 March 2014
Received in revised form 17 May 2014
Accepted 20 May 2014
Available online 28 May 2014

Keywords:
Al2024 alloy
Intermetallic compounds
Thermal analysis
Cooling rate
First derivative curve

ABSTRACT

The aim of this research is to determine intermetallic compounds formed in Al2024 alloy. The effect of cooling rates on solidification characteristics of intermetallic phases such as, nucleation and growth temperature, nucleation time and solid fraction have been determined via first derivative curve. The range of cooling rates used in this investigation is between 0.4 and 17.5 $^{\circ}C\,s^{-1}$. Thermal analysis was used to plot cooling curves and its derivative curves. Also, solid fraction versus time is plotted based on Newtonian technique. Microstructural evaluation and energy dispersive X-ray were used to identify intermetallic phases and their chemical composition. Formation of intermetallic compounds was interpreted through comparing the EDS analysis, binary and ternary diagrams, and the reactions detected from cooling curves. The results showed that nucleation and growth temperature of intermetallics was decreased by increasing of cooling rate. Also, the time intervals for the formation of intermetallic compounds were reduced.

1. Introduction

2xxx series wrought aluminum alloys have been used in aerospace applications due to their low density and good damage tolerance [1,2]. 2024 aluminum alloy is one of the most famous alloys in Al–Cu–Mg series which is used in high strength applications. Strength of the alloy is increased by precipitation hardening based on a dispersion of S-phase precipitates having the composition of Al_2CuMg [3]. The impurity elements, such as Si and Fe generally have a low solubility in aluminum and precipitate as coarse intermetallic compounds in Al matrix during solidification. These coarse intermetallic phases consume useful alloying elements, such as Mg and Cu, and then decrease the amount of fine precipitates of S-phase. Therefore, it is important to study the nucleation and growth temperature of intermetallic compounds formed during solidification of 2024 alloy.

A few researches were performed on solidification parameters of wrought aluminum alloys by Backerud et al. [4] and Flemings et al. [5]. The majority of thermal analysis studies were done on cast aluminum alloys by Gruzleski [6,7], Shabestari [8–10], Sokolowski [11,12] and other researchers [13–15]. The main reasons are probably: (1) since the intermetallic compounds in wrought aluminum alloys release low latent heat during solidification, detecting the peaks of these phases in the first derivative curves becomes more difficult and accuracy of devices is a very important parameter, (2) manufacturing process of wrought aluminum billets in industry such as, Al2024 alloy is direct-chill (DC) casting. Since the cooling rate condition in this process is approximately high, heat flow from system is too high and detection of intermetallics peaks becomes more difficult.

Also, the majority of researches based on thermal analysis were focused on solidification parameters such as, nucleation and growth temperature of primary α-Al, solidification time, latent heat, recalescence undercooling, and dendrite coherency point (DCP) [9,14,16]. Only a few investigations were carried out by Backerud et al. [4] and Shabestari et al. [17–19] on intermetallics formation by this technique.

The aim of this paper is to study intermetallic compounds, such as, Al_2Cu, $Al_{15}(CuFeMn)_3Si_2$, $Al_{20}Cu_2Mn_3$, Mg_2Si and Al_2CuMg formed in Al2024 alloy and to investigate the effect of cooling rate on the solidification characteristics of these intermetallics. Physical simulation of industrial manufacturing process of Al2024 alloy was designed to achieve cooling rates similar to DC casting process.

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http://dx.doi.org/10.1016/j.tca.2014.05.024
0040-6031/

Table 1
Chemical composition of 2024 aluminum alloy.

Alloy composition	Elements (wt%)					
	Cu	Mg	Mn	Fe	Si	Al
Al2024 alloy	4.33	1.45	0.63	0.23	0.16	Bal.

2. Experimental procedure

2.1. Material and melting

Commercial 2024 aluminum alloy was used and the chemical composition of the alloy is presented in Table 1. Seven types of molds having different cooling rates were used to investigate the influence of cooling rate on nucleation and growth temperature and nucleation time of intermetallic compounds formed during solidification of Al2024 alloy. Cooling rates condition applied in direct-chill casting process was simulated physically using a designed water circulated steel mold. Dimensions of the mold were 60 mm in diameter and 80 mm in height. In each experiment, 500 g of Al2024 alloy were melted in a crucible in an electrical resistance furnace and the melt was maintained at a temperature of $750 \pm 5\,^{\circ}$C. The melt was degassed with nitrogen-base degasser capsule for 5 min and was regularly stirred to achieve a homogenized melt. After melting, the oxide layer was skimmed from the surface of the melt and the molten metal was cast into the mold. Cooling rates used in this study was in the range of 0.4–17.5 $^{\circ}$C s^{-1}. In each cooling rate condition, three samples were cast, in order to check the reproducibility and the accuracy.

2.2. Thermal analysis

K-type thermocouples (chromel–alumel) manufactured by OMEGA[1] engineering company were used. It was inserted into a stainless steel sheath and connected to a high-speed data acquisition system. To achieve a more accuracy in each test, three thermocouples were used; one located at the center of the mold, the other located near the inner wall and the third one located between these two thermocouples. Thermocouples were fixed at exactly the same depth in the melt (at a location of 20 mm from the bottom of the mold). Analog to digital (A/D) convertor used in this work has a sensitive 16-bit convertor (resolution of $1/2^{16}$ or 0.0015%), response time of 0.02 s and high accuracy detection. Thermal analysis program can simultaneously display the cooling curves, temperature and time on the monitor of the computer for an instant observation. Each test was repeated three times. Time–temperature data were recorded with the frequency of 10 readings per second and it was plotted using Origin pro.8.6 software[2]. The adjacent averaging method was applied to each data to smooth the thermal analysis curves. Solid fraction versus time was plotted using TAW software which is set based on Newtonian method. All thermocouples were calibrated with melting and solidifying high purity aluminum (99.99 wt% Al). There are some differences in defining the thermal analysis parameters in the literature. To avoid this, solidification parameters of intermetallics used in the present investigation are given in Table 2.

2.3. Microstructural evaluations

All samples were sectioned horizontally through the places that the tips of the thermocouples were located and prepared for metallographic study. They were mechanically polished, and then etched for metallographic observations. Kellers reagent was used for microstructural study. The prepared surfaces were studied using a unimet optical microscope and Tescan-Vega II scanning electron microscope (SEM). The chemical composition of intermetallic phases was determined using energy dispersive X-ray analysis linked to SEM.

[1] OMEGA Engineering Inc., Stamford, Connecticut, USA.
[2] Origin Lab Corporation, Northampton, MA.

Table 2
Solidification characteristic parameters of intermetallic compounds.

Characteristic symbols	Characteristic description
CR	Cooling rate
$\Delta T_{N,Intermetallic}$	Range of nucleation temperature of intermetallic
$T_{G,Intermetallic}$	Growth temperature of intermetallic
$t_{N,Intermetallic}$	Nucleation time of intermetallic
$f^{s}_{N,\ Intermetallic}$	Solid fraction at the start point of intermetallic nucleation

3. Results and discussion

3.1. Sequence of phase precipitation in Al2024 using thermal analysis

A typical cooling curve obtained by thermocouple located at the center of the moldand its first derivative curve for Al2024 alloy solidified with cooling rate of 0.42 $^{\circ}$C s^{-1} are shown in Fig. 1. As seen in this figure, the range of reactions happened during solidification of Al2024 alloy has been demonstrated. According to binary Al–Cu and ternary Al–Cu–Mg phase diagrams [20] and, EDX results given in Section 3.6, the main reactions can be categorized into three regions:

Region 1) formation of some intricate phases such as, $Al_{15}(CuFeMn)_3Si_2$ and $Al_{20}Cu_2Mn_3$.

Region 2) formation of Al_2Cu and Mg_2Si intermetallic compounds.

Region 3) formation of Al_2CuMg phase.

In other researches, Wang et al. [21,22] reported $Al_{12}(FeMn)_3Si_2$, Al_7Cu_2Fe, $Al_6(FeCu)$, Mg_2Si, Al_2Cu and Al_2CuMg in 2024 alloys. In other research, Backerud et al. detected $Al_{15}(CuFeMn)_3Si_2$, $Al_{20}Cu_2Mn_3$, Al_2Cu, Mg_2Si and Al_2CuMg intermetallics [4].

To calculate nucleation and growth temperature of intermetallic compounds, two methods based on first derivative curve and second derivative curve were used. These two methods represent similar results. Based on first derivative method, some researchers such as, Shabestari et al. [17,19] and Backerud et al. [4] have expressed that start and end points of intermetallics formation is accommodated to points just before and after intermetallics peak

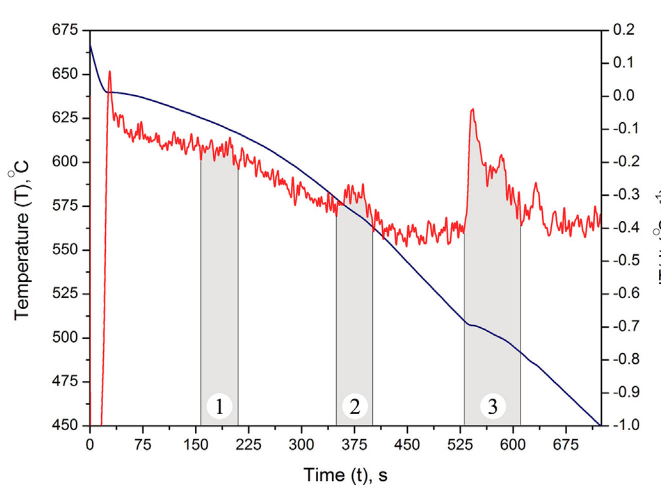

Fig. 1. Thermal analysis curves obtained from the thermocouple located in the center of the mold and the regions of the detected reactions in Al2024 alloy.