Journal of Alloys and Compounds 740 (2018) 1046–1050

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: http://www.elsevier.com/locate/jalcom

In-situ electrical resistivity monitors the annealing process for Al-Mg-Mn aluminum alloy sheet

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ARTICLE INFO

Article history:
Received 15 May 2017
Accepted 3 January 2018
Available online 6 January 2018

Keywords:
Aluminum alloys
Electrical resistivity
Annealing
Recrystallization
Avrami equation

ABSTRACT

The in-situ electrical resistivity monitors the isothermal annealing history of Al-Mg-Mn aluminum alloy sheet. The resistivity-time and the yield stress-time curves well fitted with each other helps mill engineers in optimizing both the annealing time and temperature for the mill product. The resistivity-time curves are revealed as an exponential decay and interpreted by Avrami equation with R-squared values higher than 0.98. From the extended Avrami equations, the activation energy of the recrystallization is found to be 261 kJ/mol which is a reference for the future annealing experiment. This pioneering in-situ electrical resistivity system well quantitatively correlates the resistivity with the yield stress, and therefore is a potential candidate of the commercial instrument to clarify the annealing process.

1. Introduction

Electrical resistivity has been used extensively for monitoring the annealing process including recovery, recrystallization, and grain growth for the metallic materials [1–4]. Kazemi-Choob et al. demonstrated that the in-situ electrical resistance measurements could detect the occurrence of the recrystallization process for $Ni_{50.9}Ti_{49.1}$ shape memory wires with different values of cold work [5]. Deng et al. suggested that both interface area density and lattice distortion were taken into account for analyzing the hardness and resistivity at different levels of plastic strain [2]. However, electrical resistivity applied to metal is so far only discussed in academic literature, and does not become a popular methodology universally used in the commercial instruments because of the following reasons. First, the electrical resistivity of metal is so low that the intrinsic resistivity is usually disturbed by the magnetic or electric noises of sounding. Based on the above mention, the overall decay of percentage in resistivity of the alloy caused from the annealing processes is usually less than 2.5%, so the signal-to-noise ratio is hard to meet the standard for successfully distinguishing each annealing stage. In addition, the geometrical error of resistivity, e.g., the deviation of sample sizes and the different locations of weld points, has already exceeded 2.5%. Second, people not only care if the recovery, recrystallization, and grain growth phenomena happen or not, but also need the precise annealing temperature and the accurate holding time which correspond the targeted crystallinity. Commercially, the critical annealing temperatures of these phenomena are roughly detected via the differential scanning calorimetry (DSC), the differential thermal analysis (DTA), or the thermal expansion analyzer (TMA). Unfortunately, these instruments are basically the rapid nonisothermal tests which cannot truly reflect the real-time crystallinity on the isothermal production line of mill. One of the solutions to overcome the above issues is a high-resolution in-situ electrical resistivity measurement.

For the production line, the aluminum sheets are manufactured in a number of procedure steps like remelting, casting, hot rolling, cold rolling, and temper annealing. The 3104 aluminum alloy mainly consisting of Al, Mg, and Mn elements is a kind of work-hardening alloy, so the temper annealing step plays an important role on the mechanical properties of final products. For satisfying the customer specification, the mill engineer needs to apply the proper annealing temperature and annealing time under the presupposition of energy saving. Traditionally, the laboratory investigator anneals numerous samples for various times at different temperatures in order to yield an optimized annealing parameter for the mill engineer. However, this work needs a lot of time,

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https://doi.org/10.1016/j.jallcom.2018.01.028

human resources, and experiences. Therefore an in-situ monitoring is desired to overcome these issues. Although DSC is widely used to detect the recrystallization temperature of the material, but the featured temperature is not precise enough and the isothermal annealing is not available. Besides, DSC method can only specify the rough trend of the annealing processes, while an in-situ measuring which can precisely reflect the real-time mechanical property is eagerly wanted. This study is aimed to develop a high-resolution in-situ electrical resistivity measurement which can provide the annealing parameters for producing the products qualified within industrial standard.

2. Experimental

The 2.2-mm hot-rolled 3104 plate with composition of Al–1.20Mg–1.01Mn–0.47Fe–0.21Ti–0.20Si–0.17Cu (wt%) is fully annealed and consequently cold rolled to 0.29 mm. The 0.29-mm aluminum sheets are cut to the size of 200 × 2.5 × 0.29 mm for in-situ electrical resistivity measurement, 28 × 245 × 0.29 mm for tensile test, 4 × 4 × 0.29 mm for DSC analysis. The instruments of tensile test and DSC are ZWICK-ROELL 010 and Perkin Elmer-Pyris 1, respectively. The homemade in-situ electrical resistivity measuring system is composed of a four-point AC-impedance analyzer and a furnace designed for accurate temperature control. The sample is welded with four isolated tungsten wires, and measured on the holder which is shielded around with the electrical steel, in order to avoid the magnetic disturbance. The optical image of microstructure is obtained via an Olympus-BX53M.

3. Results and discussion

3.1. DSC analysis

In order to establish a reference for the identical cold-rolled sample, DSC measurement is applied to detect the characteristic temperature which is associated with the three stages of annealing processes before we formally perform the experiment of in-situ resistivity. Fig. 1 shows a compilation of DSC curves with the heating rates of 20, 10, 5, and 2 °C/min, respectively. Observed from the data, there are three points worth discussing. First, the characteristic peaks occurs at 329 and 318 °C when the heating rates = 20 and 10 °C/min, respectively. The peak temperature shifts rightward with the increased heating rate. For an identical sample, these two peaks are believed to result from the same consequent annealing processes in the lattice, i.e., recovery, recrystallization, and grain growth. When heating rate ≤ 5 °C/min, one can see that the characteristic peaks disappear in the sinusoidal background. For the mill application, the aluminum coils are always produced with an isothermal soaking. From DSC analysis, the peak shifting with ascending temperature and vanishing at constant temperature make it difficult to find out a proper temperature for the isothermal annealing production. Second, the cold-rolled 3104 sheets reveal the recovery behavior when annealing temperature ≥149 °C, while show the recrystallization and the grain growth behaviors when annealing temperature ≥204 °C in isothermal annealing [6]. However, one can only identify the characteristic temperature initially around 320 °C via DSC method (See Fig. 1). It implies that the annealing kinetics of non-isothermal and isothermal conditions are such different that applying the annealing temperature verified from DSC into mill production line is not robust. Third, the percentage of crystallization is the important issue which the manufacturers of aluminum alloy sheets care about. Even for the same type of aluminum alloy, the customers usually request different strength based on various tempers. In addition, manufacturers always try to lower the annealing temperature as possible as they can to anneal the product with the required tempering because of the issue of energy consumption. Besides the temperature, the soaking time is also the key parameter for annealing process. Unfortunately, DSC measurement can only inspect if the recrystallization occurrence would happen, but the information of the degree in crystallinity is still lacking. In summary, the precise annealing temperature, the isothermal testing environment, and the accurate soaking time for a specific tempering are the challenges need to be overcome via in-situ resistivity measurement.

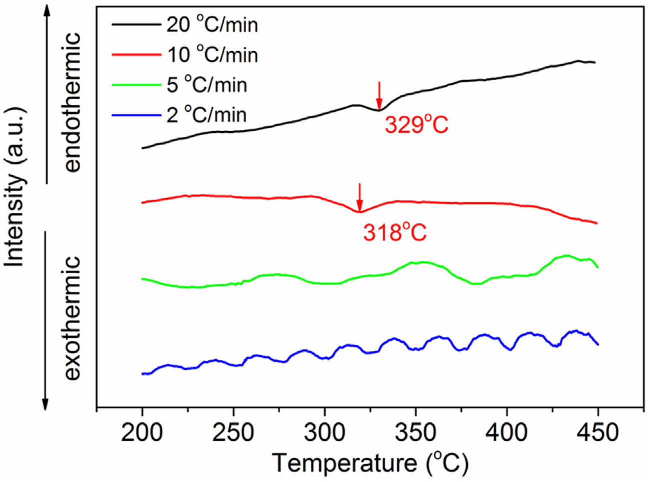

Fig. 1. DSC profiles measured with the heating rates of 20 °C/min (black), 10 °C/min (red), 5 °C/min (green), and 2 °C/min (blue), respectively. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

3.2. In-situ electrical resistivity

The sample arranged with four-point measurement in the chamber is heated to the targeted temperature then operated with an isothermal soaking process. The example of the electrical resistance history is shown as Fig. 2. Attributed the nature of positive temperature coefficient of resistance (TCR) in metal material, the resistivity increases with annealing time in 0–290 s. Based on the heating rate of 60 °C/min, TCR value is estimated to be 0.00193 $(^{\circ}C)^{-1}$. Illustrated together with related 3xxx and 5xxx series

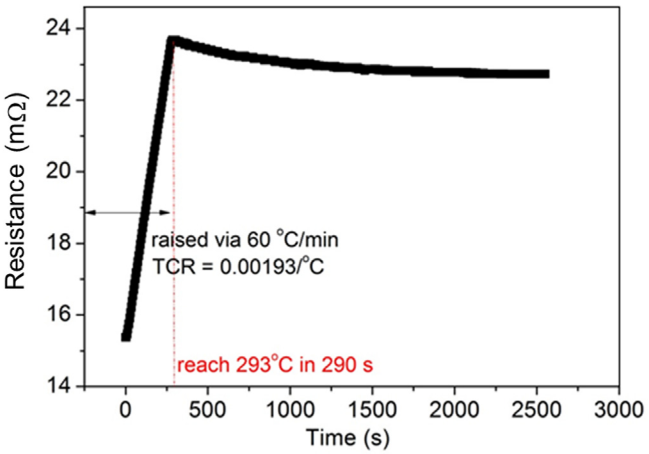

Fig. 2. The example of the electrical resistance history annealed at 293 °C.