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Preparation and Structural Characterization of Rapidly Solidified Al–Cu Alloys

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Rapidly solidified Al_{100-x}–Cu_x alloys (x = 5, 10, 15, 25, 35 wt%) were prepared and analyzed. High cooling rate increased the Cu solubility in α-Al matrix. The influence of the cooling rate on Cu solubility extension in Al was experimentally simulated. Thus the pouring was performed in metallic die and by melt spinning-low pressure (MS-LP) technique. Melt processing by liquid quenching was performed using a self-designed melt spinning set-up which combined the cooling technology of a melt jet on the spinning disc with the principle of the mold feeding from low pressure casting technology. The thickness of the melt-spun ribbons was in the range of 30–70 μm. The cooling rate provided by MS-LP was within 10^5–10^6 K/s after the device calibration. The obtained alloys were characterized from structural, thermal and mechanical point of view. Optical microscopy and scanning electron microscopy were employed for the microstructural characterization which was followed by X-ray analysis. The thermal properties were evaluated by dilatometric and differential scanning calorimetric measurements. Vickers microhardness measurements were performed in the study. In the case of the hypereutectic alloy with 35 wt% Cu obtained by MS-LP method, the microhardness value increased by 45% compared to the same alloy obtained by gravity casting method. This was due to the extended solubility of the alloying element in the α-Al solid solution.

KEY WORDS: Al–Cu alloy; Melt spinning; Fractographs; Extended solubility

1. Introduction

Nowadays, the reductions of energy consumption and emission of greenhouse gasses are very important issues in many applications. The most commonly used light materials for manufacturing components for automotive and aeronautical parts are aluminum, magnesium, and their alloys. The research communities in cooperation with manufacturing industries are oriented toward new products using innovative materials and/or novel procedures. According to the results obtained till today, it has been possible to enlarge the series of the alloys jointly with the manufacturing routes[1–5].

At the same time, materials with metastable structures play an important role in the infrastructure of modern civilization[6]. Research in this area has been intensified after 1960, when Duwez et al.[7] discovered the possibility of obtaining metastable structures by applying high cooling rates during the liquid–solid phase transformation. By intensifying the cooling rate, both structure refinement and solubility extension of alloying elements into the matrix have been achieved. Both wrought and foundry Al–Cu alloys have been widely used in such techniques due to their high mechanical properties maintained still after aging treatment application. Al–Cu alloys with higher Cu contents are limited due to the θ-Al_2Cu intermetallic phase formation which causes the alloy fragility. Following liquid quenching process, some structural changes have been produced with higher influence on the properties of Al–Cu alloys rich in Cu[8,9].

Recent studies have extensively been reported on the investigation of rapidly solidified materials with modified microstructures, compositions and properties[10–12].

Melt spinning is a commonly used technique to produce rapidly solidified alloys, developing a fine-grained microstructure with a minor content of segregation. The precipitation of a supersaturated solid solution obtained by melt spinning technique is usually accompanied by the recrystallization stage and influences the aging microstructures and the related properties. In

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addition to Al-based alloys, the mutual actions between precipitation and recrystallization have also been investigated for Mg, Fe, Cu and Ni-based alloys[13,14].

According to some studies on Al−Cu alloys[15−18], many processing parameters are very important during the melt spinning procedure. The most important of them are the melting crucible type, the cavity environment, the melt superheat, the diameter of the melt ejection nozzle, the pressure and head of the melt expulsion, etc. The heat transfer between the wheel and the ribbon together with their properties, like thickness, roughness, hardness, structural homogeneities of the ribbons, gas turbulence around the pool, the wheel substrate material, the wheel rotation speed, as a function of the solidification condition play a specific role in the final properties of the obtained material. Other researchers reported the possibility to produce nanoporous copper ribbons starting from Al−Cu bands by free corrosion de-alloying procedure prepared by single-roller melt spinning equipment for precursor samples[19]. Moreover, an expansion of the alloys compositions through the metastable structures formation has been given at industrial levels[6].

In this work, hypo- and hypereutectic Al−Cu alloys have been considered and the experimental results obtained by melt spinning-low pressure (MS-LP) processing are presented and analyzed. Structural changes caused by the solubility extension of Cu in α-Al solid solution after liquid quenching are discussed. The alloys obtained by high cooling rate are studied by X-ray diffraction (XRD) analysis, optical microscopy (OM), scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDS) technique, dilatometric and differential scanning calorimetry (DSC) measurements followed by the comparison of the data with those obtained by using metallic die.

2. Experimental

The chemical composition of the investigated Al−Cu alloys is listed in Table 1. Al−35 wt% Cu alloy was acquired as a master alloy, while the other four alloys were obtained in an electric furnace using pure Al (99.5%) and the aforementioned master alloy.

Different cooling rates were forced by employing two types of pouring methods, namely gravity casting and melt spinning techniques. Gravity casting was realized by employing metallic die (14 mm × 80 mm × 160 mm). The cooling rates from liquid state were measured, based on the cooling curves obtained for each pouring step by gravity method. For recording the cooling curves an EBI-2T-1202-type-K logger was employed: a K-TPN-101 coaxial thermocouple with an outer diameter of 0.6 mm was used as sensor. The cooling rate obtained for this kind of casting method was 32 °C/s.

The liquid quenching of Al−Cu melts was realized by using a self-designed melt spinning device. The estimated cooling rate corresponded to 10^5−10^6 °C/s. The cooling rate value (v) for the modified melt spinning device was estimated based on the relation $d = A \cdot v^{-n}$[20], where d is the secondary dendrite arm spacing (SDAS), A and n are constants.

The annealing heat treatment (T_O) of the obtained ribbons was carried out for 14 h, including a heating to 500 °C, maintenance at the maximum temperature for 2 h and followed by a cooling at a slow cooling rate in furnace.

Structural and morphological characterizations were performed by OM (NIKON Eclipse MA100) and SEM (LEO 1450VP). Vickers microhardness measurements were performed using an AHOTEC FM-700 device. For each type of alloy, 30−50 measurements were performed using 0.98 mN (for global hardness measurement of gravity cast and ribbons samples) and 0.098 mN (for the determination hardness of α-Al phase from gravity cast samples), for 15 s.

Structural transformation was monitored by DSC (Netzsch Maia F3 200) and dilatometric analysis (DIL, Linseis L75) with a heating rate of 10 °C/min. Both thermal analysis devices were previously calibrated. XRD technique was used by using Brucker diffractometer with a scanning rate of 1°/min and 2θ of 20°−90° using a Co anode with a wavelength $\lambda = 0.1789$ nm.

Table 1 Chemical composition of studied alloys (wt%)

	Si	Cu	Fe	Zn	Ti	Ni	Other	Al
AlCu5	0.12	4.97	0.40	0.11	0.01	0.08	0.02	Bal.
AlCu10	0.13	9.84	0.34	0.05	0.02	0.04	0.01	Bal.
AlCu15	0.15	14.67	0.32	0.07	0.02	0.06	0.01	Bal.
AlCu25	0.11	25.31	0.41	0.13	0.03	0.11	0.02	Bal.
AlCu35	0.11	34.52	0.44	0.15	0.03	0.18	0.02	Bal.

3. Results and Discussion

3.1. Experimental melt spinning-low pressure (MS-LP) device

The liquid quenching was performed by employing an own developed and built melt spinning device. In the following, a short description of the device was realized to identify the differences between the present device and the well-known melt spinning method.

The experimental device (Fig. 1) has the advantages to combine the cooling technology of the melt jet on spinning disc with the principle of the molds feeding from low pressure casting technology. In the case of low pressure casting technology, the transfer of the liquid metal from the furnace to the die cavity sited on the top of the furnace is ensured by an overpressure which acts on the liquid alloy. In this working method the crucible has to be placed in a hermetically sealed position. Therefore the liquid metal is forced to go vertically through the connecting tube. In the case of the experimental device indicated in Fig. 1, the working gas (Ar) introduced by the cover (6) acts upon the liquid metal (4) from the crucible (3) causing the fused metal raising in the feeding tube (5) followed by the melt placement on the rotating disc. The furnace and port-crucible are denoted by (1) and (2), respectively.

The diameter of the feeding tube (5) is 1 mm and the distance to the disc is 5 mm. The disc rotation speed is 2600 r/min and the working gas pressure is 1.5 bar (1.5 × 10^5 Pa). The aforementioned parameters have an important role, since they establish the thickness of the ribbons.

The cooling rate was assessed previously during the processing of Al−Si and Al−Cu alloy systems, for which the coefficients n and A from the Eq. (1) are as follows[20,21]:

$$\log d = -n \log v + A \quad (1)$$

where d is the secondary dendrite arm spacing (SDAS, μm), v is the cooling rate (°C/s).

The value of the cooling rate is within the limits of 10^5−10^6 °C/s. Al_{100-x}−Cu_x (x = 5, 10, 15, 25, 35 wt%) alloys were prepared using the MS-LP device. Ribbons with thickness of