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Materials and Design



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Microstructure and mechanical properties of Al-Si-Fe-X alloys

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ARTICLE INFO

Article history: Received 23 February 2016 Received in revised form 30 May 2016 Accepted 16 June 2016 Available online 18 June 2016

Keywords: Aluminium alloys Rapid solidification Melt spinning Spark plasma sintering Thermal stability

ABSTRACT

One of the current problems concerning the re-use of aluminium alloys is the recycling of aluminium alloys with increased content of iron. One of the recent trends is the processing by powder metallurgy, producing completely new grades of aluminium alloys. The aim of the present work was to test the properties of Al–Si–Fe alloy, which can be manufactured from the high-irony scrap, as a possible material for elevated temperature applications. Since chromium and nickel accompany the iron in the scrap when stainless steels are admixed, the effect of chromium and nickel on thermal stability and mechanical properties at room and elevated temperature was also studied. Al–Si–Fe–X alloys were rapidly solidified by using melt spinning technique. The rapidly solidified ribbons were pulverized by planetary ball mill and the obtained powders were used for compaction by means of Spark Plasma Sintering. It was found that nickel improves the thermal stability as well as the mechanical properties at televated temperature. On the other hand, the effect of chromium on the mechanical properties at elevated temperature.

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1. Introduction

Al–Si alloys are desired for use in severe conditions, which require high wear-resistance and thermal stability at elevated temperature [1]. Rapid solidification allows the refinement of microstructure [2] and its significant modification and extended solid solubility of constituting elements. Furthermore, it is possible to produce metastable phases by cooling of the melts by rates about $10^{4-}10^{6}$ K·s⁻¹ by rapid solidification [3]. So, the cooling rate plays an important role in controlling the morphology of primary silicon, eutectic silicon and other phases [4]. These effects lead to better mechanical properties, because new metastable phases are produced during this process. The rapid solidification powder metallurgy technology allows manufacture of powders, foils or ribbons [5,6,7].

Most transition metals exhibit a low solubility and diffusivity in solid Al. The low diffusivity is useful in stabilization of the microstructure at elevated temperature, slowing down the recrystallization processes [8]. Many of them also form thermally stable phases, which pin the grain boundaries and prevent grain growth.

Combining rapid solidification with the addition of alloying elements could result in the structure refinement and excellent mechanical properties at elevated temperatures. These alloys can be used at temperatures up to about 250 °C and allow providing weight reduction and high efficiency in automotive and aerospace industry [9,10,11]. The addition of transition metals significantly improved the hardness and compressive strength of rapidly solidified Al–Si–Fe alloys [10]. Thus, new aluminium alloys with transition metals made by rapid solidification processing are promising materials for applications in automotive and aerospace industries.

This present study is concerned with the effect of Cr and Ni additions on microstructure and thermal stability of the Al–Si–Fe base alloys processed using rapid solidification technique (melt spinning) and rapid consolidation by Spark Plasma Sintering (SPS) process.

Iron, as one of the main constituents of the tested alloys, is usually taken as an impurity in aluminium alloys, because it forms hard and brittle Al–Fe–Si phases. A majority of aluminium waste is contaminated by certain level of iron. All wrought aluminium alloys always contain at least minor iron amount. Growing concentration of iron is caused by an increasing use of recycled aluminium, where iron comes for example from the iron-containing die casting alloys, from austenitic stainless steel parts which cannot be magnetically separated from aluminium. In rapidly solidified alloys, iron increases the thermal stability due to precipitation of intermetallic compounds with iron [5,10,12]. Formation of intermetallic phases during solidification depends on the levels of Fe and Si concentration and cooling rate [13,14].

Nickel in aluminium alloys is known to improve the thermal stability due to low diffusivity in aluminium. Therefore it is added to the Al–Si based alloys for the manufacture of the pistons of internal combustion engines. Chromium is a relatively cheap alloying element and has one of the lowest diffusion coefficients in aluminium. However, its solubility in aluminium is extremely low [15]. Cr modifies the morphology of the iron-containing intermetallic compound and refines the primary silicon particles [10].

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The development of recycling technologies has increased for the high-irony scrap of Al–Si alloys. One of the possible methods of processing this waste is powder metallurgy using rapid solidification methods and SPS. SPS process is a modern method of sintering, which allows consolidation of various materials by the combination of uni-axial pressing and passage of high electric current [16]. This method utilizes rapid heating and lower sintering temperatures than in conventional sintering techniques [17]. The compact specimen is obtained in a very short time. The advantages are low cost, minimal grain growth and maintaining fine-grained structure [18].

In this work, the production of Al–Si–Fe, Al–Si–Fe–Cr and Al–Si–Fe– Ni alloys by powder metallurgy of rapidly solidified particles was tested. Hardness of melt-spun ribbons and compact alloys, obtained by cryogenic milling and subsequent Spark Plasma Sintering (SPS) process, was tested. Vickers hardness was measured and differential thermal analysis was carried out in order to determine mechanical properties and thermal stability, respectively.

2. Experimental

Cast AlSi₁₂Fe₇, AlSi₁₂Fe₄Ni₃ and AlSi₁₂Fe₄Cr₃ alloys were prepared by melting in an electric resistance furnace. Commercial master alloys (AlSi₅₀, AlFe₁₀, AlNi₂₀, AlCr₅ – in wt.%) and commercial purity elements (Al, Si, Fe, Cr) were used as components for the alloy preparation. Ribbons were produced by the melt spinning process by means of casting of molten alloy onto the rotating copper wheel (CuCr₁Zr_{0.1}) with a diameter of 300 mm rotating at 30 m/s. Melting was completed under argon protective atmosphere with temperature of the melt 950 °C. The rapidly solidified ribbons were pulverized by planetary ball mill Retch PM 100 CM in a stainless steel container (ball-to-powder ratio of 15:1) for 10 min at 400 ppm. At the beginning of the milling, liquid nitrogen was added to the container. The obtained powders, with the size of the particles at less than 50 µm, were consolidated to 20 mm diameter cylindrical samples by the means of SPS. SPS was carried out in the Institute of Plasma Physics AS CR using Thermal Technology SPS 10-4 device by the pressure of 80 MPa for 10 min at 500 °C with the heating rate of 100 K/min.

The samples were ground using P80–P4000 sandpapers with SiC abrasive particles and polished using D2 diamond paste. Then the samples were etched by Kroll's reagent (5 ml HNO₃, 10 ml HF, 85 ml H₂O). Microstructure of all the samples was investigated by the optical microscope (Olympus PME3) with Axio Vision 4.8 software, as well as by the scanning electron microscope (SEM–TESCAN VEGA 3 LMU). Phase composition was determined by X-ray diffraction (PANanalyticalX'Pert Pro, CuK α radiation). The grain size of aluminium-based matrix was determined by Scherrer Calculator in X'Pert High Score Plus software. Vickers microhardness was measured by Carl Zeiss Neophot 2 metallographic light microscope equipped with Hanneman hardness testing unit with a load of 5 g (HV 0.005) on the melt spun ribbons in longitudinal and cross section, while the hardness of the cast and compacted samples was investigated by the Vickers method with the load of 5 kg (HV 5) due to the heterogeneity of the samples.

The thermal stability of the melt spun ribbons and compact alloys was evaluated as the change in hardness (HV 0.005) during the short-term or long-term annealing performed at 100–500 °C for 1 h, respectively at 300 and 400 °C for 400 h in air. Hardness was measured every 100 h.

Mechanical properties were measured on compact samples by means of the universal testing machine LabTest 5.250 SP1-VM. The device is equipped with a laser extensometer for the accurate measurement of deformation of the sample. Tests were performed at room temperature and at 300 and 400 °C on samples prepared by SPS.

Differential scanning calorimetry (DSC) was performed on the rapidly solidified ribbons. The temperature program was set to a linear increase of temperature at a heating rate of 10 K/min. DSC analyses were performed on a Setaram DSC 131 instrument under argon atmosphere in the temperature range of 25–600 °C.

3. Results and discussion

3.1. Microstructure of the cast alloys

Cast alloys are composed of eutectic structure α -Al and Si in which the coarse intermetallic phases Al₅FeSi (AlSi₁₂Fe₇ alloy and AlSi₁₂Fe₄Cr₃



Fig. 1. Light micrographs of a) AlSi₁₂Fe₇, b) AlSi₁₂Fe₄Ni₃ and c) AlSi₁₂Fe₄Cr₃.

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