Materials Science & Engineering A 621 (2015) 212–217

Contents lists available at ScienceDirect

Materials Science & Engineering A

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

The quantitative relationship between microstructure and mechanical property of a melt spun Al–Mg alloy

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ARTICLE INFO

Article history:
Received 23 July 2014
Received in revised form
16 October 2014
Accepted 17 October 2014
Available online 24 October 2014

Keywords:
Rapid solidification
Melt spinning
Aluminum alloys
Microstructure
Microhardness

ABSTRACT

This paper reports a study on the microstructure and hardness of an as-melt spun Al–Mg alloy as a potential material used for micro-manufacturing. Our results show that the microstructure is characterized by a nearly single-phase fine-grained Al-based supersaturated solid solution containing a relatively high density of dislocations and the as-melt spun Al–Mg alloy exhibits a much higher microhardness relative to that of the coarse-grained counterpart. The quantitative relationship between microhardness and microstructure was established by quantifying the contributions of various strengthening mechanisms.

1. Introduction

Over the past few decades, rapid solidification techniques, including atomization methods (e.g., gas, water and centrifugal atomization), chilling methods (e.g., melt spinning and melt drag), etc., have been widely studied [1–3]. For the alloys with compositions specifically designed using the empirical rules proposed by Inoue [4] (i.e., multiple components (≥ 3), significant differences in atomic sizes ($> 12\%$) and sufficiently negative heats of mixing), namely, 'amorphous compositions', sufficiently high cooling rates during rapid solidification (e.g., melt spinning) lead to the formation of fully amorphous materials. Rapidly solidified (RS) alloys with conventional (i.e., 'non-amorphous') compositions usually exhibit the following microstructural features [1]: refined microstructures (including grains and second-phase particles), minimized macro- and micro-segregation, substantially extended solid solubility, the formation of non-equilibrium or metastable phases, etc.

Given the metastable nature of the aforementioned amorphous alloys and RS crystalline alloys, their thermal stability, including (i) the prevention of full or partial crystallization in the amorphous alloys, and (ii) the retention of refined grains and second-phase particles, supersaturated solid solutions and non-equilibrium/metastable phases in the RS crystalline alloys, has long been one of the major concerns. Consequently, a vast number of research efforts have been attempted to improve the thermal stability, concentrating on the optimization of the compositions [5–22]. In an effort to stabilize the amorphous alloys, a series of strategies have been developed, aiming at minimizing the diffusion ability of the component atoms, which is considered as a prerequisite condition for the occurrence of crystallization: (i) enhancing the differences between atomic sizes by introducing atoms with much larger and/or much smaller sizes in order to improve the packing density of the component atoms, e.g., adding Y to $Fe_{63}Zr_8Co_6Al_1$-Mo_7B_{15} [5], Sc to $Zr_{52.5}Cu_{17.9}Ni_{14.6}Al_{10}Ti_5$ [6], O to $Zr_{80}Pt_{20}$ [7], Si to $Cu_{47}Ti_{34}Zr_{11}Ni_8$ [8], and B, Si and Pb to $Zr_{52.5}Cu_{17.9}Ni_{14.6}Al_{10}Ti_5$ [9], (ii) increasing the magnitude of negative heats of mixing between component elements in order to promote chemical short-range ordering and thus to limit long-range diffusion of atoms, e.g., adding Pd to Al–Y–Ni-based amorphous alloys [10], increasing B concentrations in Fe–Zr–B–Nb amorphous alloys [11], and adding Si to Al–Fe–La amorphous alloys [12], (iii) introducing component elements with very large atomic sizes and thus extremely low diffusion ability, e.g., selecting Hf as a component element in the Co–Hf–B system [13], and (iv) increasing the number of components as much as possible, namely, 'confusion principle' as suggested by Greer [14]. In order to achieve high thermal stability in the RS crystalline alloys, the elements that exhibit very low diffusivity in the matrix are usually selected as alloying elements. For example, in Al-based RS alloys, transition metals (e.g., Cr, Fe, Ti, Mn, Ni, etc.) are often used as alloying elements [15–22] and sometimes elements with extremely low diffusivity in Al (e.g., Mo

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http://dx.doi.org/10.1016/j.msea.2014.10.047
0921-5093/

[15]) are added in order to further reduce the diffusion ability of the alloying elements. In addition, some specific elements are also chosen to stabilize the metastable phases, e.g., using Ti, V, Nb and Ta to stabilize the icosahedral topology of the quasicrystalline icosahedral phases in the Al–Fe–Cr-based alloys [16,17].

Among the various rapid solidification techniques, melt spinning has attracted considerable interest, which is primarily motivated by a relatively higher cooling rate (10^5–10^7 K/s [1]) during melt spinning and the ability of melt spinning to produce a large quantity of rapidly solidified materials (ribbons). Due to the lightweight attribute, melt spun Al alloys (both amorphous and crystalline) have been paid particular attention and extensive experimental studies have been conducted on this class of melt spun materials [10,12,16,23–32], which involve characterization of microstructures and testing of mechanical properties. The available experimental results reveal brittleness of amorphous ribbons [30–32], which limits their widespread applications. In contrast, the crystalline ribbons generally exhibit high strength and moderate ductility [23–25], rendering them a class of promising materials. Inspection of the published literature, however, indicates that few research efforts have been made to establish the quantitative relationship between microstructures and mechanical properties of melt spun crystalline ribbons, e.g., the quantitative contributions of various strengthening mechanisms associated with microstructural features to the overall strength, which hinders our ability to tailor the mechanical properties of the melt spun crystalline alloys by optimizing their microstructures. In particular, the emergence of micro-manufacturing in recent years [33,34], e.g., forming of micro-sheet-metal components used in electronics products and micro-electro-mechanical-systems (MEMS) [35], enables the melt spun crystalline ribbons to be directly utilized as micro-components following appropriate micro-manufacturing procedures, rendering it increasingly important to develop quantitative relationships between microstructures and mechanical properties of the crystalline ribbons. In view of the aforementioned discussion, it is the objective of the present study to quantitatively correlate microstructures with mechanical properties (specifically microhardness) of the melt spun crystalline ribbons.

In the present study, an Al–Mg alloy 5083 (Al–Mg–Mn–Cr–Fe alloy) was selected as the model material, which was motivated by two factors. First, 5083 Al exhibits excellent weldability and corrosion resistance [36], rendering it a suitable material for micro-sheet-metal components in micro-manufacturing. Second, in the wrought 5083 Al, due to very low equilibrium solubility of the alloying elements Mn, Cr and Fe in the matrix Al [37], most of them are present in the form of submicrometer- to micrometer-sized second-phase particles [36], which provide very limited precipitating strengthening. In contrast, in melt spun 5083 Al, a very high cooling rate during melt spinning may result in the dissolution of most of the aforementioned alloying elements in the matrix Al, which provides appreciable solid-solution strengthening and thus further increases the strength of melt spun 5083 Al as compared to that of the wrought 5083 Al.

2. Experimental

The 5083 Al sheets were first induction melted in a quartz tube in an argon atmosphere, and then ejected onto the surface of a copper wheel of 220 mm in diameter using a pressure of 0.05 MPa through the rectangular slit of 5 mm in length and 0.3 mm in width at the bottom of the quartz tube. The copper wheel was water-cooled and rotated at the surface speed of 20 m/s. Following melt spinning, the ribbons with 50 to 90 μm in thickness and 2.5 to 4 mm in width were produced. The composition of the as-melt spun ribbons was measured using chemical analysis and is shown in Table 1 (Fe, Si and Cu are impurities originating from the raw materials used).

Table 1
Chemical composition of the as-melt spun 5083 Al ribbons.

	Mg	Mn	Cr	Fe	Si	Cu	Al
wt%	4.70	0.76	0.14	0.22	0.15	0.014	Balance
at%	5.22	0.37	0.073	0.11	0.14	0.0059	Balance

The microstructures of the as-melt spun ribbons were studied via optical microscopy (OM, model: Axiovert 200 MAT) under polarized light and via transmission electron microscopy (TEM, model: JEOL JEM 2010) equipped with energy X-ray dispersive spectroscopy (EDX) operated at 200 kV. Planar-view specimens for OM observations were prepared by mechanical grinding and polishing followed by electro-chemical etching using 60 ml fluoroboric acid in 400 ml deionized water as the electrolyte for anodic oxidation. The preparation of planar-view specimens for TEM analysis is described as follows. First, the as-melt spun ribbons were mechanically thinned to 30 to 40 μm thickness by equally grinding both the wheel and the air sides. Then, the specimens were twin-jet polished using a solution of 25 vol.% nitric acid and 75 vol.% methanol at −30 °C. X-ray diffraction (XRD) analysis was also conducted on the as-melt spun ribbons using a Rigaku D/MAX-2500 diffractometer equipped with a Cu target at a speed of 0.02° and a count time of 3 s per step. Microhardness of the as-melt spun 5083 Al ribbons was measured in 20 randomly selected points with a load of 10 g and a holding time of 10 s using a FM-ARS9000 microhardness tester.

3. Results

3.1. Microstructure

Figs. 1a and b show the planar-view polarized OM microstructures of the as-melt spun ribbons near the surfaces of the wheel and air sides (~10 μm away from the surfaces), respectively. Inspection of Figs. 1a and b reveals that the microstructures consist of fine grains. The presence of a fine-grained structure can be attributed to rapid solidification during melt spinning (cooling rate 10^5–10^7 K/s [1]), which induces the formation of a high density of nuclei. Grain sizes were measured from a series of OM images using the linear intercept method per ASTM E112 [38]: step 1, draw a series of parallel lines in each of a large number of OM micrographs; step 2, for each of the grains in these OM microhraphs, locate the line that passes through the grain, measure the length of the line segment that is intercepted by the grain, and take the measured length of the intercepted line segment as the grain size. Using the aforementioned two-step procedure, a large number of grain sizes were measured. The statistical distributions of grain size near the surfaces of the wheel and air sides are shown in Figs. 1c and d, which were obtained by randomly measuring 621 and 639 grains, respectively. The corresponding average grain sizes ($\overline{D}$) were evaluated as ~6.2 and ~7.8 μm, respectively, by fitting the statistical distributions using a lognormal probability function [39,40]. These experimental results reveal an insignificant difference in average grain size in the regions near the surfaces of the wheel and air sides albeit a higher cooling rate in the wheel side.

The TEM bright field (BF) image of the as-melt spun ribbons as shown in Fig. 2a reveals the presence of very few precipitates (volume fraction < 0.1%). EDX analysis reveals that the precipitates contain Al, Mn and Fe, and other elements, including Mg, Cr, Si and Cu, cannot be detected. Based on the previously published literature [41,42], the precipitates can be most likely identified as Al_6(Mn, Fe). After analysis of selected area electron diffraction (SAED) on each of a large number of randomly selected grains, a typical SAED pattern was taken as shown in Fig. 2b. Inspection of the SAED pattern indicates that only Al