



# Evolution of the microstructure and hardness of a rapidly solidified/melt-spun AZ91 alloy upon aging at different temperatures

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#### ABSTRACT

The effect of aging at different temperatures on a rapidly solidified/melt-spun AZ91 alloy has been investigated in depth. The microstructures of as-spun and aged ribbons with a thickness of approximately 60  $\mu m$  were characterized using X-ray diffraction, transmission electron microscopy and laser optical microscopy; microhardness measurements were also conducted. It was found that the commercial AZ91 alloy undergoes a cellular/dendritic transition during melt-spinning at a speed of 34 m/s. A strengthening effect due to aging was observed: a maximum hardness of 110 HV/0.05 and an age-hardenability of 50% were obtained when the ribbon was aged at 200 °C for 20 min. The  $\beta$ -Mg17Al12 phase exhibits net and dispersion types of distribution during precipitation. The dispersion of precipitates in dendritic grains or cells is the main source of strengthening.

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### 1. Introduction

Magnesium-based alloys are some of the lightest structural metallic materials. Automobile manufacturers want to replace denser materials, such as steel, cast iron, copper-based alloys and aluminum alloys, by magnesium-based alloys [1]. The requirement to reduce the weight of components in automobiles, following from legislation limiting emissions, has triggered renewed interest in magnesium [2]. There is also a major effort in the aircraft construction industry to reduce part weight [3]. The structural aspects of the design of high-performance Mg alloys is one side of magnesium technology [4,5], and the processing technologies are another [6].

One of the promising processing routes involves rapid solidification (RS). Olsen and Hultgren used a classical rapid-solidification (RS) method [7], in which a small droplet of

molten alloy was injected into a liquid quenching bath, to study the effect of cooling rate on the homogeneity of solid solutions. Duwez et al. obtained a continuous series of metastable solid solutions in silver–copper alloys by means of melt-spinning [8]. Matsuda et al. carried out a study of rapid-solidification processing of some Mg–Li–Si–Ag alloys that they had chosen and designed [9], and found that the microstructure consisted of a fine dispersion of a Mg<sub>2</sub>Si phase in a fine-grained body-centered cubic Mg–Li solid solution, resulting in desired improvements in thermal stability and mechanical properties.

Cai et al. examined the influence of rapid solidification and copper mold casting on the microstructure of the AZ91HP alloy and obtained a microhardness of 854 MPa [10]. Inoue et al. published a paper about a novel hexagonal structure in ultrahigh-strength magnesium-based alloys made by melt-spinning

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and powder metallurgy (PM) with a yield strength ( $\sigma_y$ ) of 610 MPa, and compared the results with other Mg-based alloys [5], including an RS/PM-processed AZ91 alloy. However, this AZ91 alloy was only one of the reference materials used for comparison and was made by RS/PM. The subject of the present investigation was the examination of rapidly solidified ribbons of a commercial cast AZ91 alloy.

# 2. Experimental Procedure

The experimental material, consisting of AZ91 alloy from an industrial ingot, was remelted at 720 °C in a graphite crucible under a protective  $CO_2$ – $SF_6$  atmosphere and recast into a bar of dimensions 190 mm×95 mm×20 mm in a steel mold. An ascast specimen and small cuboids for preparing rapidly solidified ribbons were prepared from the bar. Cuboids for processing pure Mg ribbons were prepared from a Mg ingot.

Rapidly solidified AZ91 ribbons were prepared by use of a melt-spinning apparatus at a speed of 34 m/s [11]; the AZ91 ribbons prepared were about 7 mm wide and 60  $\mu$ m thick. One ribbon was then selected and divided into pieces for subsequent experiments. Pieces were aged at 100, 150, 200, 250, 300, and 400 °C for 20 min under a protective CO<sub>2</sub>–SF<sub>6</sub> atmosphere and cooled outside the furnace at room temperature; the total heating and holding time was appropriately 30 min.

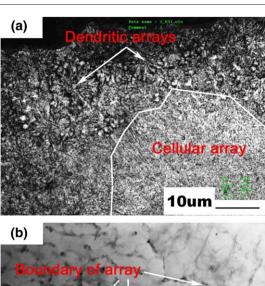
The as-cast AZ91 alloy taken from the bar was characterized using an X-ray diffractometer (D/MAX 2000/PC, Rigaku). A Brinell hardness test was also conducted on the as-cast AZ91 alloy.

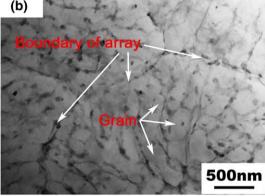
A sample of the as-spun AZ91 alloy taken from a ribbon was processed into an inset specimen for metallographic observation of a transverse section under a laser optical (focusing and scanning) microscope (LOM). Some of the asspun and aged samples were polished with very fine abrasive paper and ion-thinned in a precision ion-polishing system. The thinned specimens were investigated using a transmission electron microscope (JEM-2000EX, JEOL). Some other asspun and aged ribbons were polished with very fine abrasive paper on only the interface with the roller for phase identification using the above-mentioned X-ray diffractometer, and measured on a Vickers hardness testing instrument at a load of 50 g with a holding time of 15 s. A pure Mg ribbon was characterized by TEM, and its hardness was also tested for comparison with the data from the alloy.

#### 3. Results and Discussion

#### 3.1. Evolution of Microstructure

The microstructural morphology of the as-spun AZ91 alloy is shown in Fig. 1. The general morphology of a transverse section of the as-spun ribbon is characterized as a whole in Fig. 1(a), in which the top side is the interface with the roller of the melt-spinning apparatus and the bottom side is the free surface of the ribbon. The microstructure consists of dendritic arrays near the interface and cellular arrays near the free surface. The sizes of the dendritic arrays are about 10  $\mu$ m, and





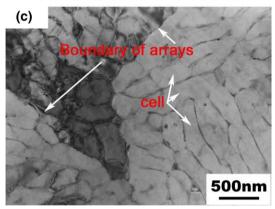


Fig. 1-Morphological images of the as-spun AZ91 ribbon: (a) general LOM image of a transverse section of the ribbon, (b) TEM image of dendritic arrays, (c) TEM image of cellular arrays.

the spacing of the secondary dendrite arms or the size of the dendritic grains is approximately 250 nm, from Fig. 1(b). The diameter of the cells or the cell spacing in the cellular arrays is about 200 nm, from Fig. 1(c). A second phase is distributed on the boundaries of the dendritic grains and cells, mainly along the edges of the boundaries (see Fig. 2).

The evolution of the microstructure of the as-spun and aged ribbons is displayed in Figs. 1–4. The second phase is distributed during solidification among and within the dendritic and cellular arrays. The total morphology of the ribbon aged at 100 °C and the dendritic and cellular arrays in it are shown in Fig. 2. The general morphology has hardly altered, as can be seen from Fig. 2(a). Fig. 2(b), (c) and (d) present TEM images showing

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