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OXIDE FILM AND POROSITY DEFECTS IN MAGNESIUM ALLOY AZ91

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Abstract

Porosity is a major concern in the production of light metal parts. This work aims to identify some of the mechanisms of microporosity formation in magnesium alloy AZ91. Microstructure analysis was performed on several samples obtained from gravity-poured ingots in graphite plate molds. Temperature data during cooling was acquired with type K thermocouples at 60 Hz at three locations of each casting. The microstructure of samples extracted from the regions of measured temperature was then characterized with optical metallography. Tensile tests and conventional four point bend tests were also conducted on specimens cut from the cast plates. Scanning electron microscopy was then used to observe the microstructure on the fracture surface of the specimens. The results of this study revealed the existence of abundant oxide film defects, similar to those observed in aluminum alloys. Remnants of oxide films were detected on some pore surfaces, and folded oxides were observed in fracture surfaces indicating the presence of double oxides entrained during pouring.

Introduction

Magnesium cast alloys, such as AZ91, are gaining increasing attention in the struggle for weight saving in the automobile industry [1]. However, in many cases the consistent production of sound AZ91 castings is marred by the stubborn persistence of some defects that are difficult to remove: porosity, macrosegregation, oxide entrainment, irregularity of microstructure, etc. The formation of microporosity in particular is known to be one of the primary detrimental factors controlling fatigue lifetime and total elongation in cast light alloy components.

Many efforts have been devoted to investigate the mechanisms of porosity formation in the last 20 years. More recently, new mechanisms of pore formation based on entrainment of oxide films during the filling of aluminum alloy castings have been identified and documented [2-7]. Oxide film defects are formed when the oxidized surface of the liquid metal is folded over onto itself and entrained into the bulk liquid. A layer of air is trapped between the internal surfaces of the oxide film, which leads to the porosity formation in the solidified castings. The entrainment process due to surface turbulence is usually rapid, in the order of milliseconds; therefore the time is very limited to form new oxide film on the fresh surface, so that the entrained oxide film can be very thin, in the order of nanometers [2].

Oxide film defects may be contained in most reactive liquid metals such as Al and Mg due to surface turbulence during the melting, pouring and transfer processes in casting. These defects have been observed on the fracture surfaces of tensile test specimens and the oxides have been identified by SEM-EDX analysis [7-9]. In contrast with the efforts devoted to Al-based cast alloys, few studies have been done in Mg alloy castings. Griffiths and Lai [8] investigated the nature of the oxide film defects in pure Mg castings. They found double oxide film defects comprised of folded MgO films on the fracture surface of tensile test bars taken from the castings. Mirak et al. [9] recently studied the characteristics of oxide films in AZ91 alloys, where the formation of oxide films was induced by the impingement of bubbles.

In this study, we examined the microstructure of magnesium alloy AZ91 ingots gravity-poured in plate graphite molds. Temperature data during cooling was acquired with type K thermocouples at 60 Hz in two locations of each casting. The microstructure of samples extracted from the regions of measured temperature was then characterized using optical metallography, tensile tests, four point bend tests and Scanning Electron Microscopy (SEM) of the fracture surfaces. The nature of oxide film and porosity defects in AZ91 was investigated.

Experimental Procedure

Design of castings

The cast ingots or slab castings were produced in a graphite plate mold at the facilities of Oak Ridge National Laboratory (Oak Ridge, TN). The mold was rectangular and the thickness of the wall was 0.5 in (12.7 mm). The width, height, and thickness dimensions were 5.5×11×2.25 in (140×279×57 mm), respectively, as shown in Figure 1. Three thermocouples were placed in the empty molds at distances of approximately 2.5, 5 and 8 in (64, 127 and 203 mm) from the casting end per each casting.

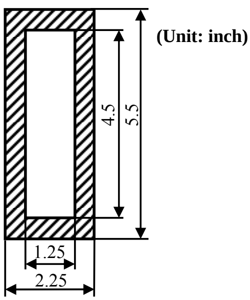

Figure 1. Cross section of plate graphite mold used for AZ91 castings. Height was 11 in (279 mm).

Figure 2. Pouring of casting type C.

The tested AZ91 alloy composition was Mg, 9.0%Al, 0.7%Zn, 0.2%Mn. The furnace charge was in the form of pre-alloyed ingot. The weight of the melt was 8 kg and the alloy was melted in an electrical resistance furnace. For protection, Ar and CO_2+3%SF6 were used as cover gases. The pouring temperature was approximately 700 ^{o}C. No degassing procedures were used. All castings were poured from one melt. The melt was poured directly from the crucible to minimize temperature decrease during pouring (Figure 2). The mold was not preheated and was coated with boron nitride. In order to assess the reproducibility of the results, two molds were used. Temperature data was acquired with thermocouples type K at a sampling rate of approximately 60 Hz. The measured cooling curves are shown in Figure 3. The cooling curves are labeled in the following format: xn_m, where x – is a letter, indicating the mold type, n – indicates casting number (1 or 2), and m – indicates thermocouple location (b-bottom of casting, c-center of casting). The cooling curves show an excellent reproducibility. The data measured by the thermocouple near the top of the casting was discarded because of turbulence in this region. As shown in Figure 3, the cooling rate during solidification for the AZ91 alloy castings was approximately 3.0 ^{o}C/s. In this article, results for only one mold type, denoted as type C, are reported. Analysis of castings in other graphite and ceramic molds of different dimensions will be reported elsewhere.

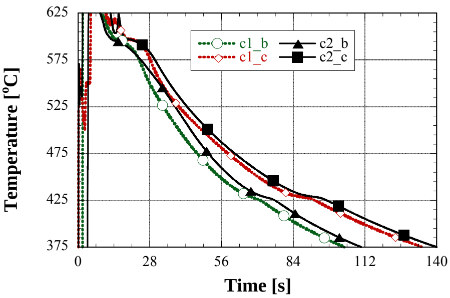

Figure 3. Cooling curves for AZ91 Mg alloy castings in mold type C.

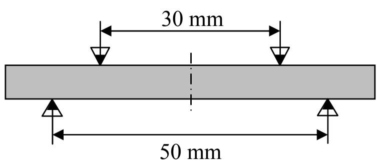

Figure 4. Sketch of four point bending test geometry.

Sample preparation for optical metallograhy

The samples were cut near the location of the thermocouple for each as-cast ingot and then hot-mounted in phenolic resin, with one side of the plate flush with the mounted surface. The samples were then polished using a machine disc grinder. The silicon carbide abrasive papers of grade 500 and 2400 μm grits were used successively. In between papers the samples were cleaned by ethanol thoroughly. The samples were then cleaned in a sonic bath before being examined by optical microscope. Approximately 20 to 30 images were taken for each sample.

Sample preparation for tensile test and Four Point Bend (FPB) test

Two tensile test strip specimens and two Four Point Bend (FPB) test specimens with dimensions of 115 mm long, 10 mm wide, and 3 mm thick, were cut from each of the cast samples to characterize the casting mechanical properties. These specimens were tested using an EM Model 5869 Instron machine at a strain rate of 0.001/s for the tensile test and a cross-head speed of 0.05 in/min for the FPB test. Figure 4 depicts the sample arrangement employed in the FPB test. The fracture surfaces of the test specimens were examined using a field-emission gun scanning electron microscope (FEG-SEM) equipped with an energy dispersive x-ray spectrometry (EDX).

Results and Discussion

Porosity was the major defect observed in the tested specimens. Pores ranging in size from 100 μm to 500 μm were found in many of the polished surfaces. Figure 5 shows typical pore morphology at a location close to the thermocouple in the AZ91 C1 sample. A magnified view (Fig. 5(b)) reveals dendrites protruding into the pore as well as pieces of oxides on the surface of the pore. EDX spectroscopy shows a three-fold increase of the oxygen content inside the pore compared with the surrounding matrix. This pore was most probably caused by interdendritic shrinkage, however, the presence of oxides might suggest also a pore formed by an entrained double oxide that was torn apart by shrinkage-induced shear forces.

Long pieces of oxide films, some longer than 1 mm, were observed in AZ91 samples through optical microscopy and in SEM images. Figure 6 shows a "dragon-shaped" oxide film found on a polished surface of the specimen. The distinct precipitation upon both sides of the film might suggest the former existence of a double oxide that was later torn open, with the higher precipitation occurring on the wetted side.

Tensile tests performed on strip specimens at a strain rate of 0.001/s confirmed that oxides and porosity had a significant effect on the mechanical properties of AZ91. Figure 7 shows that the yield strength and ductility of two C1 samples of the gravity-poured ingots are considerably smaller than those of a AZ91D die cast plate of 3 mm thickness. The relatively good properties of the AZ91D sample is thought to be caused by the high velocity of the die casting process which possibly breaks the oxide films into very small parts [10].

The details of fracture surfaces of tensile test AZ91 samples are shown in Figures 8-10. A distinct interface between the dendritic matrix and an oxide region can be observed (Figure 8). Figure 9 shows two symmetrical oxide films on either side of a fracture surface. This agrees well with the observation by Griffiths and Lai [8] for pure Mg castings. A magnified view of the oxide region (Figure 10) reveals a pleated surface, similarly as observed in double oxide films in aluminum alloys.

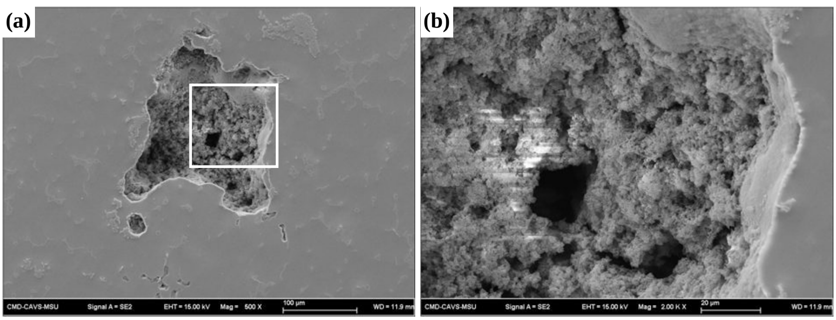

Figure 5. (a) Typical pore morphologies formed at the location close to the thermocouple in casting AZ91 C1 sample; (b) higher magnification (2000X) of image (a).

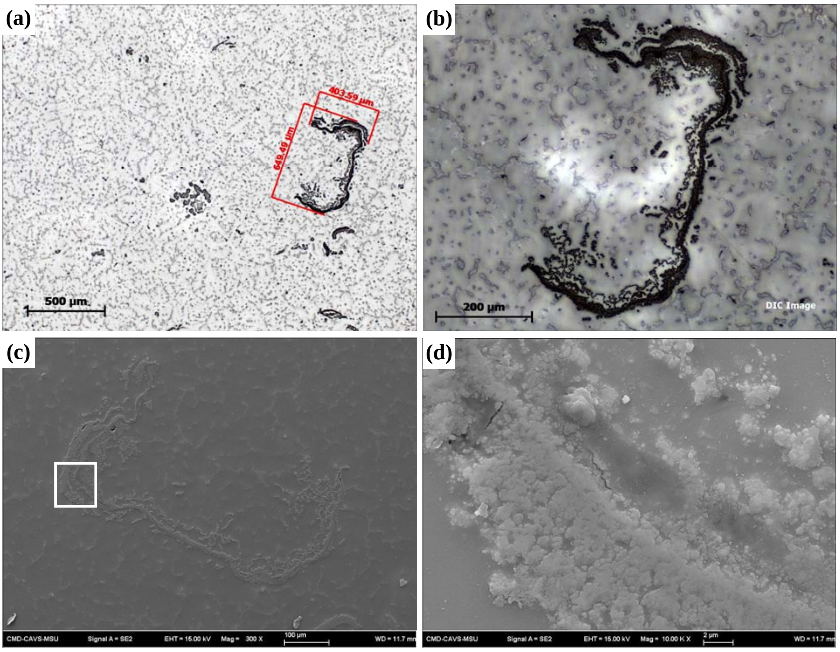

Figure 6. Oxide film in AZ91 sample C1. (a) Optical micrography; (b) Transmitted light differential interference contrast (DIC) image; (c) SEM image; (d) Higher magnification (10000X) of image (c).

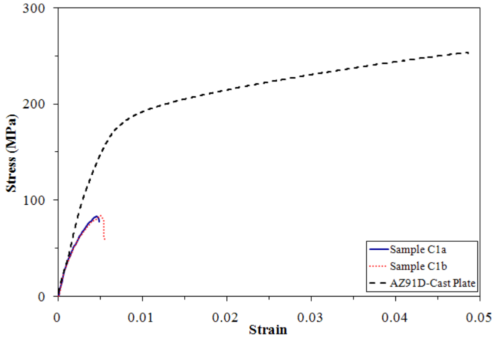

Figure 7. Tensile test results for strip specimens with a strain rate of 0.001/s; Specimen C1a and C1b were taken from different locations in AZ91 sample C1; The third specimen was taken from a AZ91D die cast plate.

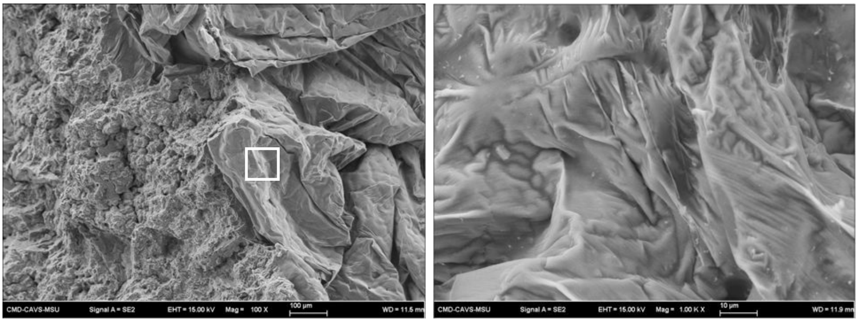

Figure 8. Scanning electron microscope images showing the interface region of the matrix and oxide film on fracture surfaces of a tensile test specimen taken from AZ91 sample C1.

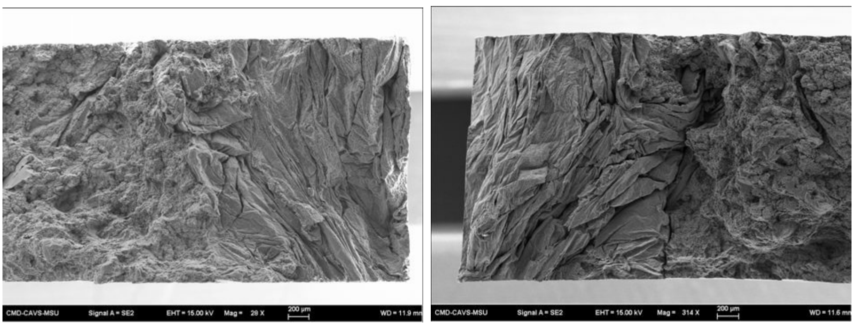

Figure 9. Scanning electron microscope images of oxide films on the two sides of the fracture surfaces of a tensile test specimen taken from AZ91 sample C1.

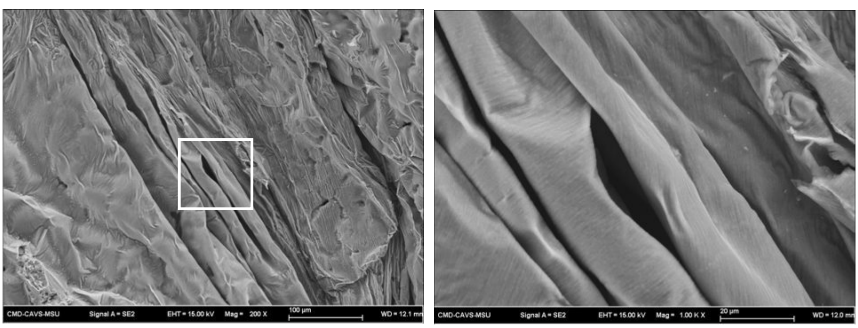

Figure 10. Higher magnification views of the oxide film found on the fracture surface shown in Figure 9.

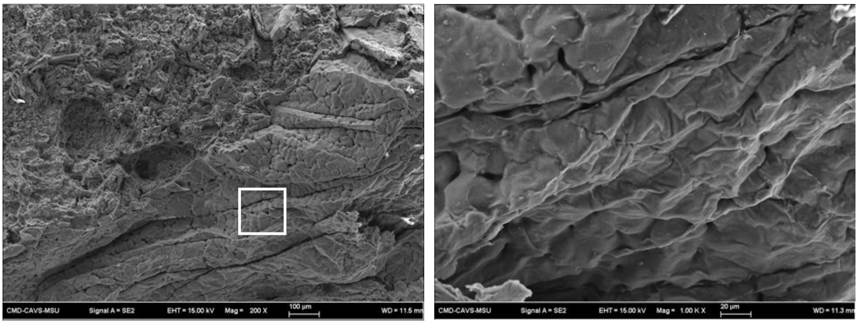

Figure 11. Scanning electron microscope images show the interface region of matrix and oxide film on fracture surfaces of a four point bending test specimen taken from AZ91.

SEM images of the fracture surfaces of a four-point bend specimen are presented in Figure 11. Again, an interface between the matrix and an oxide region can be detected. However, these surfaces look different than the fracture surfaces of the tensile test samples. They are darker and the oxide film is not pleated as the one in the tensile test fracture surfaces; instead, an apparently thinner film is folded over dendrites. Possibly, the different states of stress in comparison to the tensile test samples are in part responsible for these features.

Conclusions

The microstructure of plate-shaped ingots of magnesium alloy AZ91 obtained by gravity-pouring in a graphite mold was analyzed by EDX spectroscopy, SEM and optical metallography. Abundant porosity was found throughout the ingots, with some pores as large as 500 µm. Pieces of oxide film, some of them 1 mm or longer, were also detected in many polished cross sections. Distinct features on both sides of the films suggest that they might be remnants of torn double oxide films or bifilms entrained during the pouring. This fact is supported by the analysis of the fracture surfaces of tensile and four point bend samples, which revealed pleated layers of oxides similar to those observed in aluminum alloys. Oxide fragments were also detected inside some pores, indicating that they might have formed from oxide bifilms that were torn by shrinkage shear or dendrite action.

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