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MICROSTRUCTURE AND MECHANICAL PROPERTIES OF Mg – Sn ALLOYS SYNTHESIZED BY DISINTEGRATED MELT DEPOSITION TECHNIQUE

1 Introduction

Magnesium (Mg) and its alloys are suitable for aerospace and automotive applications, due to their low density and high specific strength. Mg – Al – Zn alloys and Mg – Al – Mn alloys are the widely used commercial alloys as they possess nominal strength and ductility at ambient temperature (27 °C) [1]. However, owing to the presence of the $Mg_{17}Al_{12}$ phase that is thermally unstable at temperatures > 120 °C, these alloys have relatively poor microstructural stability and low strength-retention characteristics at elevated temperatures [2, 3]. For high temperature applications, such as in power-train components of automobiles, magnesium alloys are expected to exhibit both microstructural stability and mechanical stability (i.e. retention of strength) at elevated temperatures [2]. Although alloys such as those containing zirconium, and those incorporated with rare earth metals are being used/developed for enhanced thermal stability, these elements are expensive and hence are suitable only for critical applications.

In the context of the need of magnesium based materials with good high temperature properties, Mg-alloys with Sn addition are being developed, as addition of tin forms Mg_2Sn phase which has better thermal stability than $Mg_{17}Al_{12}$ phase. From the binary phase diagram. the only secondary phase that is formed in the Mg-Sn system is the Mg_2Sn phase [4]. The Mg_2Sn phase has higher melting point (~770 °C) [4] when compared to the $Mg_{17}Al_{12}$ phase (420 °C) [2], and is therefore expected to exhibit a higher degree of thermal stability. In the current investigation, binary Mg – Sn alloys with Sn = 5, 10 and 15 (wt. %) were developed considering the high solid solubility (~14.5 wt. %) of tin in magnesium at the eutectic temperature (561 °C) [4, 5]. The binary Mg – Sn alloys were synthesized using the technique of Disintegrated Melt Deposition (DMD) followed by hot extrusion. This paper reports on the processing, composition, microstructure and mechanical properties of the developed Mg – Sn alloys in comparison with pure magnesium.

2 Experimental Procedure

2.1 Primary Processing

Mg – Sn binary alloy system with varying tin content (5 %, 10 % and 15 %) was produced using magnesium turnings of 99.9 + % purity and tin (Sn)-powder (325 mesh, supplied by ACROS Organics, New Jersey, USA) using the disintegrated melt deposition (DMD) technique (Fig. 1, *a*). The DMD technique involves bottom pouring of the melt. It is a high yield process, which uses simultaneous vortex stirring of the melt and its disintegration in an argon (Ar) gas inert environment.

In the DMD process (Fig. 1, *a*), initially the Mg turnings along with the Sn particles were heated in a graphite crucible to 750 °C in an electrical resistance furnace in an atmosphere of inert argon gas. In order to facilitate a near uniform distribution of the Sn particles in molten Mg, the superheated molten slurry was stirred for 5 minutes at 460 rpm using a twin blade (pitch 45) mild steel impeller (coated with Zirtex 25). The molten alloy was then disintegrated by two jets of argon gas oriented normal to the melt stream placed at the bottom of the crucible, which was deposited onto a steel substrate. The resultant alloy ingot was about 40 mm in diameter.

2.2 Secondary Processing

The Mg – Sn binary alloys obtained from the DMD process were machined to 36 mm diameter and soaked at 400 °C for 1 hour, and hot extruded using a 150-ton hydraulic press at 350 °C using an extrusion ratio of 20.25:1. The extruded rods were about 8 mm in diameter (Fig. 1, *b*). The extruded rods were used for microstructural and mechanical property characterization.

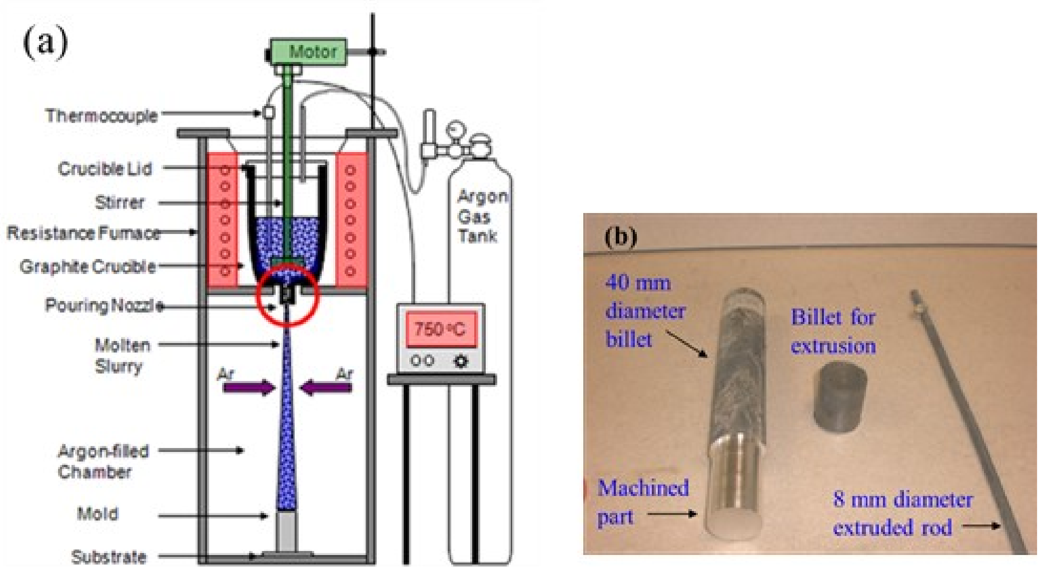

Fig. 1. Schematic of the Disintegrated Melt Deposition (DMD) process (*a*) and extruded rod after secondary processing (*b*)

2.3 Materials Characterization

Microstructural investigation of finely polished and etched Mg – Sn alloy samples was conducted using a Field Emission Scanning Electron Microscope (Hitachi FESEM-S4300) coupled with Energy Dispersion Analysis (EDS). X-ray diffraction analysis was conducted on the samples using an automated Shimadzu LAB-XRD-6000 X-ray diffractometer (Cu K_α; κ = 1.54056 Å) at a scanning speed of 2 degree per minute. Microhardness measurements were performed on polished flat specimens using a Shimadzu HMV automatic digital microhardness tester with a Vickers indenter (load: 25 gf and dwell time: 15 seconds). More than 10 measurements were performed on each test specimen, and the average microhardness value has been reported. Uniaxial tensile tests were performed at room temperature (27 ^{o}C) on smooth bar tensile specimens (ASTM E8M-08) of 5 mm diameter and a gage length of 25 mm, using a Materials Test System (MTS 810) at a constant strain rate of 1.69×10^{-4} s^{-1}. A minimum of five tests were conducted on each alloy composition. Fractographic analysis was performed using the FESEM.

3 Results and Discussion

3.1 Microstructure

From the representative microstructure of Mg – Sn binary alloys shown in Fig. 2, *a*, it could be observed that the grains were equiaxed, and showed significant grain refinement with grain size varying between 2 to 5 microns for all the three compositions. The grain size was one order lower than that of pure magnesium (grain size: ~29 microns). Fig. 2, *a* also shows the presence of second-phase particles in the magnesium matrix. The Mg-Sn alloy binary phase diagram indicates that Mg_2Sn phase is the only secondary phase (eutectic phase) present in the phase diagram [4]. Hence, the particles present in the microstructure are Mg_2Sn particles. This was further confirmed by XRD analysis, which shows the Mg_2Sn peaks (Fig. 2, *b*). Furthermore, in the XRD profile, an increase in the intensity of Mg_2Sn peak with an increase in tin content was observed indicating the increased formation of the Mg_2Sn phase. As seen from Fig. 1, *a*, Mg_2Sn particles vary in their morphology (also see Fig. 2, *a*), such as: (i) polygonal-shaped particles of size 2 to 4 μm (marked 'A'), and (ii) submicron-sized lath/rod-like particles (of length 500 nm to 1 μm; diameter < 200 nm, marked 'B'). Reports on earlier works [5 – 10], show that particles of varying morphologies and with fine sizes are formed in Mg – Sn based alloy systems. The particle shape varied from nano-polygonal to nanolath/rods and was reported to be dependent on the solidification rate in rapidly solidified alloys [6, 10], whereas in extruded or aged alloys, the fine size of the particles and their morphology was influenced by the dynamic precipitation process during hot extrusion [7, 10]. Hence, in the present work, the formation of equiaxed grains and grain refinement in all the developed binary alloys can be attributed to the occurrence of dynamic recrystallization during the hot extrusion process. With regard to the varying morphologies of the Mg_2Sn secondary phase, it is identified to be controlled by the mutually interactive influences of: (a) solidification rate, (b) dynamic precipitation, and (c) alloying additions [6, 10]. While the polygonal and lath-shaped particles are formed by dynamic precipitation due to hot extrusion, the fine size of the particles are influenced by the processing method itself that plays a dominant role. During the secondary processing of hot extrusion, initially, as the alloy is homogenized at 400 ^{o}C (prior to hot extrusion), the secondary Mg_2Sn phase

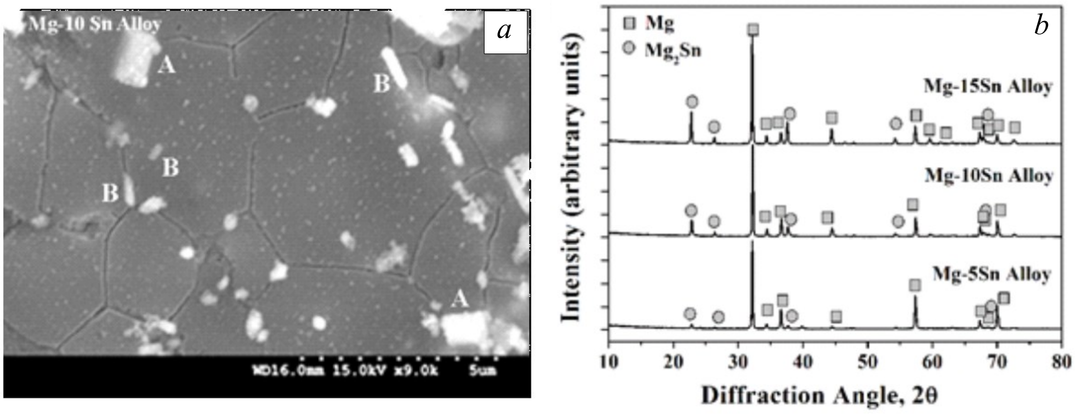

Fig. 2. Microstructure of M – 10 Sn alloy showing fine grain size and presence of second phase with two different morphologies, viz., (A) polygonal and (B) rod-like(*a*) and X-ray diffraction pattern of Mg – Sn alloys showing the presence of Mg_2Sn phase and pure Mg (*b*)

present in the alloy would dissolve in the magnesium matrix due to the high temperature. Subsequently, during hot extrusion (350 ^oC), a large number of dislocations and sub-grain boundaries would be generated within the magnesium matrix [7]. These would not only provide active sites for dynamic precipitation of the secondary phase (i.e. Mg_2Sn), but would also facilitate the formation of fine-sized particles.

3.2 Mechanical Properties

The microhardness and tensile strength properties of the developed Mg – Sn binary alloys are discussed below.

3.2.1 Microhardness

Table 1 shows the microhardness values of the developed Mg – Sn alloys with increasing tin content. It is seen that when compared to pure magnesium, all the three compositions of the Mg – Sn binary alloys show a significant increase in microhardness. For example, the micro-hardness of Mg – 15 Sn alloy is ~2.6 times higher when compared to that of pure magnesium. This significant increase in the hardness is because of the formation of the Mg_2Sn phase, which has inherent high hardness (1.2 GPa) [7]. The increase in hardness with higher Sn content is due to the increased formation and presence of the Mg_2Sn phase, as shown by the increase in the intensity of Mg_2Sn peaks with increasing Sn content in the XRD pattern (Fig. 1, *b*).

3.2.2 Tensile Properties

In Table 2, the tensile properties of the developed binary Mg – Sn alloys are listed. It can be observed that when compared to pure magnesium, the developed alloys show a significant increase in yield strength and ultimate tensile strength, accompanied by a reduction in ductility. Considering the Mg – 5 Sn alloy, it exhibits 85 % increase in yield strength and 75 % increase in ultimate tensile strength when compared to pure magnesium. Indeed, amongst the three compositions, the Mg – 5 Sn alloy showed the best combination of tensile properties. The reduction in tensile properties of the Mg – 10 Sn alloy and Mg – 15 Sn alloy is due to the increased formation of the hard Mg_2Sn phase with the increase in tin content. Particularly under tensile loading, an increased volume fraction of the Mg_2Sn phase for 10 % and 15 % Sn resulted in enhanced brittleness causing reduction in ductility and strength properties when compared to the Mg – 5 Sn alloy.

Fig. 3 shows the representative tensile fracture surfaces of Mg – Sn alloys. The effect of the second-phase particles in determining the tensile properties and fracture behavior is evident from the tensile fracture surfaces. The alloy showed a combination of cleavage fracture and cracked second-phase particles, Fig. 3, *a* shows the representative fracture surface of Mg – 5Sn alloy, wherein matrix cracking and cracking around the second phase particles are evident (as shown by arrows in Fig. 3, *a*). Further,

Table 1

Micro-hardness of the developed Mg – Sn alloys, with increasing Sn content

Materials	Hardness, HV
Pure Mg	46 ± 1
Mg – 5 Sn	115 ± 13
Mg – 10 Sn	117 ± 9
Mg – 15 Sn	122 ± 16

Table 2

Tensile properties of the developed Mg – Sn alloys

Materials	Yield Strength, MPa	Tensile Strength, MPa	Elongation, %
Pure Mg	129 ± 11	170 ± 10.0	6.2 ± 0.7
Mg – 5 Sn	238 ± 9	291 ± 11.0	5.5 ± 1.3
Mg – 10 Sn	215 ± 4	274 ± 4.5	4.8 ± 0.4
Mg – 15 Sn	212 ± 6	257 ± 2.0	4.8 ± 1.3

with increasing Sn content (i.e. increase in Mg_2Sn phase formation), features of dominant brittle failure are observed. In Fig. 3, *b*, which shows representative fractograph of Mg – 15Sn alloy, arrows indicate extensive matrix and particle cracking. Also, isolated pockets of dimples of varying size interdispersed with fine microscopic voids are seen in the alloys. These features are reminiscent of local ductile failure mechanisms.

In all the three Mg – Sn alloy compositions developed, the significant improvement in hardness and tensile properties obtained due to the addition of Sn results due to one or more of the following strengthening mechanisms [7]: (i) solid-solution strengthening, (ii) precipitation strengthening (Mg_2Sn phase formation), (iii) grain boundary strengthening (grain refinement), (iv) increase in the dislocation density due to residual stress (arising from the mismatch of coefficient of thermal expansion between magnesium matrix and Mg_2Sn phase) during processing, and (v) Orowan strengthening, which arises due to the interaction between the dislocations and second-phase particles.

4. Conclusions

Mg – Sn alloys were developed with varying Sn content using disintegrated melt deposition technique. The developed alloys were investigated for their microstructure and mechanical properties. The following are the conclusions from the present work:

1. The processing method used (disintegrated melt deposition technique) followed by hot extrusion facilitated the formation of fine Mg_2Sn phase having varying morphologies.

2. The addition of Sn content to pure magnesium resulted in fine-grained alloys with equiaxed microstructure, due to dynamic recrystallization during processing.

3. The increase in hardness of the Mg – Sn alloys with increase in Sn content is attributed to the inherent high hardness of the Mg_2Sn phase.

4. When compared to pure Mg, the developed Mg – Sn alloys showed higher values of microhardness and significant improvement in tensile properties.

5. Under tensile loading, the Mg – 5 Sn alloy showed the best strength properties among the developed alloys. The relatively lower strength of the Mg – 10 Sn and Mg – 15 Sn alloys is due to the formation and presence of a higher volume fraction of the Mg_2Sn phase, which resulted in second-phase particle fracture during tensile loading.

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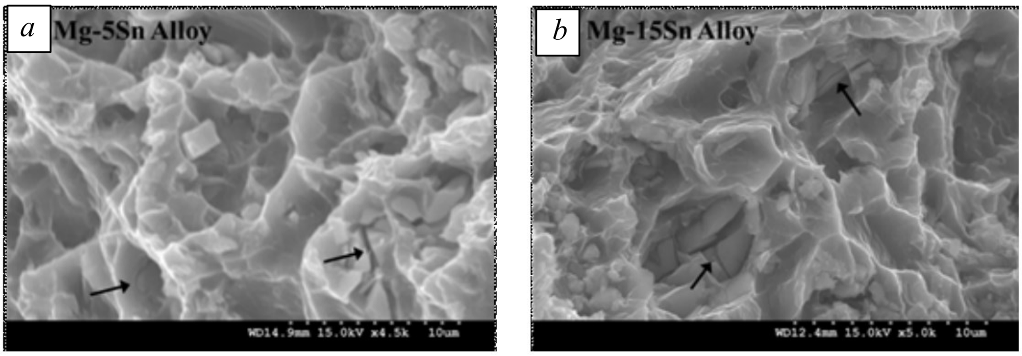

Fig. 3. Representative tensile fracture surface of Mg – Sn binary alloy showing:
a – matrix cracking and cracking around particles in Mg – 5Sn alloy; *b* – dominant brittle morphology and second phase (particle) fracture with increasing Sn content, in Mg – 15 Sn alloy.
Dimple features that is reminiscent of locally ductile failure mechanism can also be seen

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