RECYCLING OF MG CHIPS WITH THE HELP OF COMMERCIALLY PURE MG POWDER

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Abstract

In the present study, the feasibility of recycling AZ91 alloy chips with the help of commercially pure Mg powders, via pressing and hot extrusion processes to produce bulk solid samples, was investigated. Furthermore, the effect of Mg powders and their amounts on the mechanical properties and corrosion behaviour of the recycled AZ91 alloy were studied. To evaluate the quality of the products, their microstructures were studied using optical microscope, their mechanical properties were determined by hardness, compression and wear tests. The corrosion behaviour was evaluated by immersion tests and potentiodynamic polarisation measurements in 3.5 % NaCl solution. The results from the hardness, compression and wear tests, immersion tests and potentiodynamic polarisation measurements showed that the hardness, strength and corrosion resistance of the recycled AZ91 alloy increased progressively by increasing the chips wt.% in the products. However, the wear resistance of the recycled AZ91 alloy decreased along with the reduction of electrical conductivity by adding Mg chip.

1 Introduction

The expanding use of Mg alloys in the automotive, aerospace and electronic industries, mainly due to a number of wonderful properties such as high specific strength, excellent die castability, superior machinability, good ductility and damping capacity leads to an increasing quantity of chip and scrap [1-3]. In order to meet the increasing usage of Mg alloys aimed at weight savings, it is necessary not only to consume relatively low energy, but also to develop useful recycling processes. Since the high surface-to-volume ratio of Mg chips leads to melt losses, hazards during transportation, high oxide, inclusion and impurity contents, recycling with liquid state approaches may not be efficient to overcome in Mg recycling because of the needs of special protective environment and extra caution. Moreover, the main concern associated with using the secondary Mg is the high level of Fe content and oxide inclusions that are detrimental to the corrosion and mechanical properties of the secondary alloy. In recent years, recycling by solid state approaches has been shown to possess a number of technological advantages over the traditional casting processes because its cost is relatively low, also favorable for environment protection. In the solid state recycling, chips and scraps are recycled by consolidation using plastic deformation processes such as cold or hot pressing followed by hot extrusion without melting [1–5].

In this work, to obtain better metallurgical bonding, the effect of Mg powders added to composite composition as a binder in recycling of AZ91 chips was investigated. The recycling process was carried out by cold compaction followed by hot extrusion process. Simultaneously, the effect of the additional amount of Mg powders on quality, microstructure, mechanical and corrosion properties of the two phase produced products was studied.

2 Materials and processing

AZ91 machined chips with the size of 220 μm and pure Mg powder with the average particle diameter of about 16 μm were used as the starting materials. After preparing the chips, they were mixed with different amounts of Mg powders in the range of 25–75 wt.%. In order to consolidate the prepared mixtures, first they were filled into a cylindrical container with a diameter of 30 mm and then cold pressed in a steel die by applying 700 MPa pressure. Then, the cold pressed samples were extruded into a 10 mm rod at 350 °C. To evaluate the quality of the extruded rods, their microstructures were investigated using an optical microscope. Also their densities were measured based on the Archimedes method. Theoretical density of the recycled Mg alloys was supposed to be 1.74 g/cm^3. Mechanical properties of the produced two phase samples were determined by carrying out hardness measurements and compression tests. Hardness survey was measured on Shimadzu HMV2 microhardness tester by applying indentation load of 200 g with a Vickers indenter. At least, ten successive measurements were made for the recycled Mg alloys including different amounts of chips. Round samples with 20 mm of length and 10 mm of diameter were tested by a Dartec Universal testing machine at a crosshead speed of 1 mm/min to determine the compression behaviour of the samples. The results of the compression tests were compiled by averaging the decision of three samples.

Wear tests were carried out under normal atmospheric conditions (20±2 ^{o}C and 30±5 %RH) on a reciprocating wear tester. All experiments were carried out under a constant normal load of 1 N using a 10 mm diameter steel ball, and sliding speeds from 0.0128 to 0.0567 ms^{-1} with increments of 0.014 ms^{-1}. After the wear tests, the wear tracks formed on the recycled Mg alloys were detected by a Veeco Dektak 6M profilometer to quantify the test results in terms of wear track area. Wear track area values were then converted into Relative Wear Resistance (RWR) by assuming the RWR of the recycled Mg alloy containing 100 wt.% Mg chip under a sliding speed of 0.0375 m/s as 1.0. After the wear test, wear tracks formed on surfaces of the recycled Mg alloys were examined using a scanning electron microscope (SEM).

Evaluation of the corrosion was determined by weight loss-measuring test and by potentiodynamic polarization measurement. For the weight loss-measuring test, square shape samples with an average size of 2 mm x 2 mm were ground up to 1200 grit SiC emery papers and polished 1 μm diamond paste. After cleaning with acetone, these samples were weighed and then immersed in 3.5% NaCl solution in a Pyrex glass cell exposed to atmospheric air for 36 h. The amount of solution in the baker was estimated by taking into account the surface area of the samples as 0.6 ml/mm^2. After the test, the samples cleaned/dried were weighted by an electronic balance having a resolution of ±0.1 mg. The normalized weight loss values of the investigated alloys were calculated in the unit of g/cm^2 by dividing the weight loss of the each sample by their initial total surface area.

For the potentiodynamic polarization measurement, machined samples (square cube shape samples with an average size of 2 mm x 2 mm) were mounted on copper rod using epoxy resin for electrical connection, and open surface of all samples were ground with 1200 mesh SiC abrasive paper and then cleaned with deionized water followed by rinsing with methanol and dried. The potentiodynamic polarization measurement was carried out using a Gamry model PC4/300 mA potentiostat/galvanostat controlled by a computer with DC 105 Corrosion analysis software. All the corrosion experiments were performed at the room temperature in a glass cell containing 3.5% NaCl solution. Each polarisation experiment was carried out holding the electrode for 45 min. at open circuit potential (E_o) to allow steady-state to be achieved. Potentiodynamic polarization curves were generated by sweeping the potential from cathodic to anodic direction at a scan rate of $1 mVs^{-1}$, starting from −0.1 up to +0.4 V. Each data point for both immersion and potentiodynamic polarisation tests represents at least average of 3 different measurements.

3 Results and discussion

Figure 1 contains the optical microscopy images of the recycled Mg alloys showing the overall microstructures in the extrusion direction. The white regions correspond to the Mg chips; while the gray regions are the Mg powder. The recycled Mg alloys including different amounts of chips possessed a lamellar structure composed of alternating layers of Mg powder and Mg chip aligned parallel to the extrusion direction.

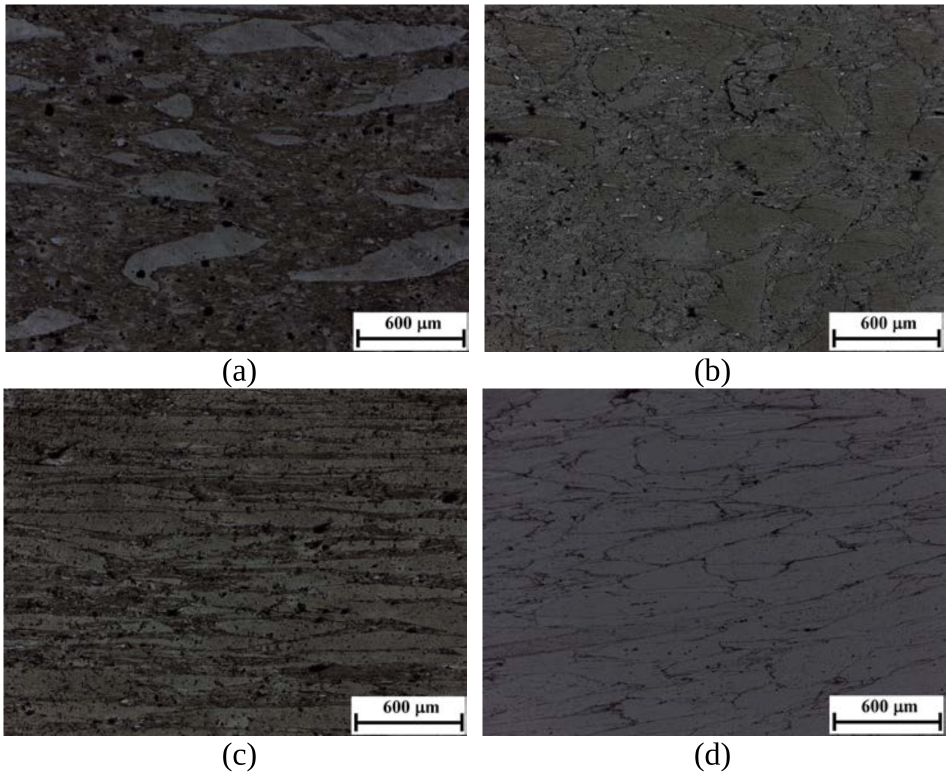


Figure 1. The longitudinal structures of the recycled Mg alloys including (a) 25% chips, (b) 50% chips, (c) 75% chips and (c) 100 % chips.

Figure 2 showed the variation of the density and electrical conductivity of the recycled Mg alloys versus wt.% of chips. As shown in Figure 2, the density of consolidated mixture was not altered notably by Mg chip addition and the recycled Mg alloys indicated close results to the theoretical density (1.74 g/cm^3). While, the electrical conductivity of the recycled Mg alloys decreases by increasing wt.% of chip, which is due to decreasing easily deformable Mg powder phase. Microstructural examinations conducted on the recycled Mg alloys revealed that a decrease in Mg chip content leaded to a substantial improvement in the integrity of microstructure (Figure 1) along with the increment of electrical conductivity (Figure 2). Besides, void and interface between chips in the recycled Mg alloys disappeared with decreasing wt.% of chip by hot extrusion.

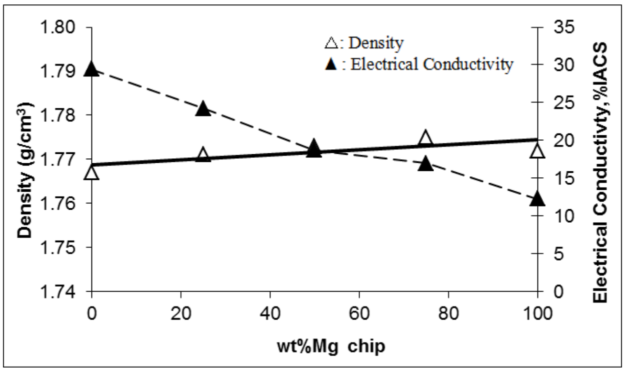


Figure 2. The variation of (a) density and (b) electrical conductivity as a function of Mg chip content in the recycled Mg alloys.

Figure 3 shows the dependence of the hardness and compression strength of the recycled Mg alloy on its Mg chip content. As it can be expected, since the hardness of chips is higher than powder regions, the hardness and compression strength of the recycled Mg alloys increase by increasing wt.% of chip. Especially interesting is that the hardness and compression strength of the recycled Mg alloy increased from 50 to ~ 70 $HV_{0.2}$ (i.e. a 40% increase) and from 250 to ~ 370 MPa (i.e. a 48% increase) by 100 wt. % Mg chip addition, respectively.

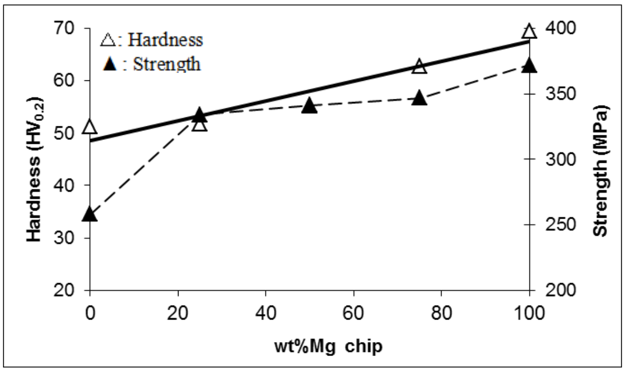


Figure 3. Variation of (a) hardness and (b) compression strength as a function of Mg chip content in the recycled Mg alloys.

The relative wear resistances of the recycled Mg alloys including different amounts of chips are plotted against sliding speed as shown in Figure 4. It is evident from Figure 4 that the wear resistances of the recycled Mg alloys are distinctly different; in fact, the samples with the best mechanical properties, the recycled Mg alloys with 75 and 100 wt.% Mg chip, generally performed the worst. However, the recycled Mg alloys with 25 and 50 wt.% Mg chip with the increase in sliding speed had a significant improvement in wear resistance.

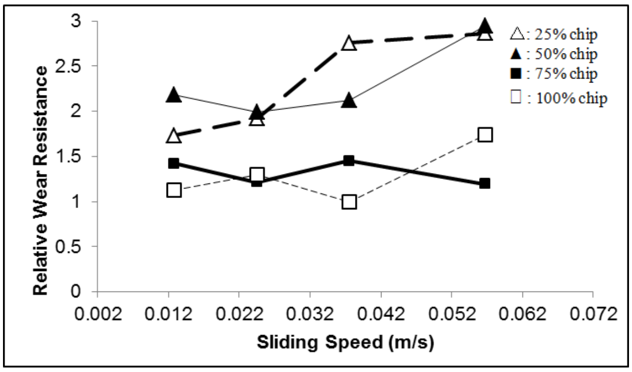


Figure 4. Effect of Mg chip content on the wear resistance of the recycled Mg alloys.

SEM observations of worn surfaces of the recycled Mg alloys under the low sliding speeds (0.0128 and 0.0245 m/s) find numerous parallel ridges and grooves running parallel to the sliding direction, as shown in Figure 5. However, the large differences in the strength and electrical conductivity of the recycled Mg alloys might result in different abrasive wear modes among the various materials, and thus affect the amount of material removed from the worn surfaces. Also, the increment of electrical conductivity by decreasing wt.% of chip (Figure 2) means that the binding between chips in the recycled Mg alloys is good. It may be seen from Figure 2 and 3 that the recycled Mg alloys with 25 and 50 wt.% Mg chip are relatively lower strength values and higher electrical conductivity than the recycled Mg alloys with 75 and 100 wt.% Mg chip. The lower strength and higher electrical conductivity of the recycled Mg alloys with 25 and 50 wt.% Mg chip favor the ploughing abrasive wear mode, in which material is primarily pushed to the sides of the scratches without being removed [5]. In contrast, the higher strength and less electrical conductivity of the recycled Mg alloys with 75 and 100 wt.% Mg chip are more likely to wear by micro-cutting [5], so the material from the groove is removed by a chip.

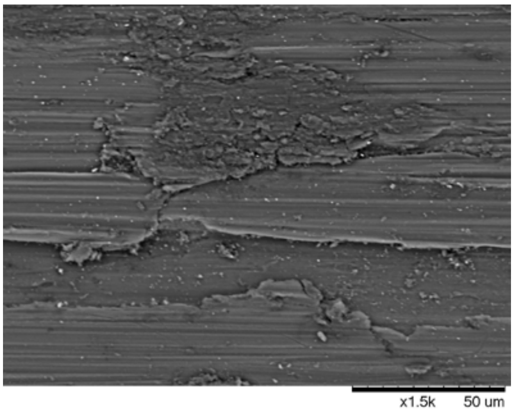
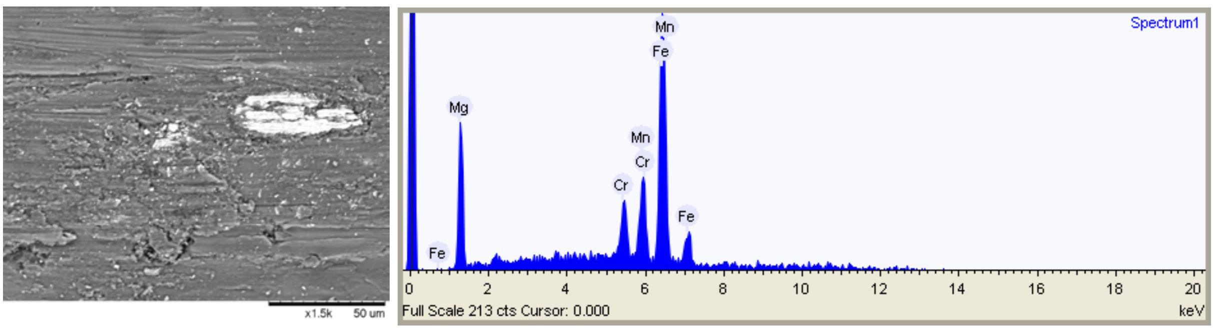


Figure 5. Grooves and scratch marks on the worn surface of the recycled Mg alloys with 100 wt.% Mg chip under a sliding speed of 0.0128 m/s indicating abrasion.

Above the sliding speed of 0.0245 m/s, a layer of material intermittently covering the worn surface of the recycled Mg alloys with 25 and 50 wt.% Mg chip begins to be apparent, as shown in Figure 6. EDS analysis reveals the presence of O, Fe, Mg, Mn, Cr within this layer as shown in Figure 6 b indicating adhesive transfer of material from the steel counterface, as well as some oxidation. The existence of oxidation products on the recycled Mg alloys has been found to shield the underlying worn surface from metallic contact, leading to improved wear resistance in the sliding wear of the recycled AZ91 Mg alloys against steel [5].



(a) (b)

Figure 6. (a) SEM image and (b) corresponding EDS analysis of the wear track formed on the worn surface of the recycled Mg alloys with 50 wt.% Mg chip.

Figure 7 shows the effect of chip content on the corrosion rate of the recycled Mg alloys in 3.5 % NaCl solution. It was observed from the immersion test results that the corrosion was decreased considerably up to 25 wt.% chips above which the chip content resulted in a slight decrease in corrosion rate of the recycled Mg alloys. As illustrated in Figure 7, addition of 25 wt.% Mg chip to the recycled Mg alloy exhibited the corrosion rate from 0.42×10^{-3} to around 0.06×10^{-3} ($g/cm^2 xh$), however, slightly lower corrosion rate (0.022×10^{-3} $g/cm^2 xh$) was observed with 100 wt.% Mg chip.

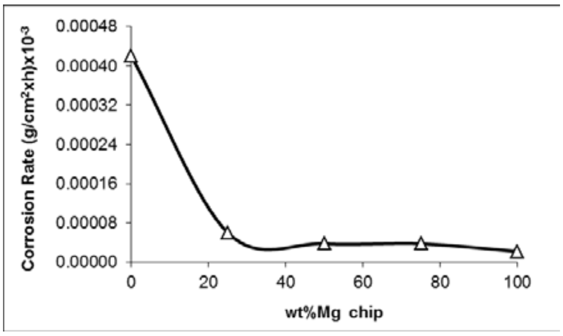
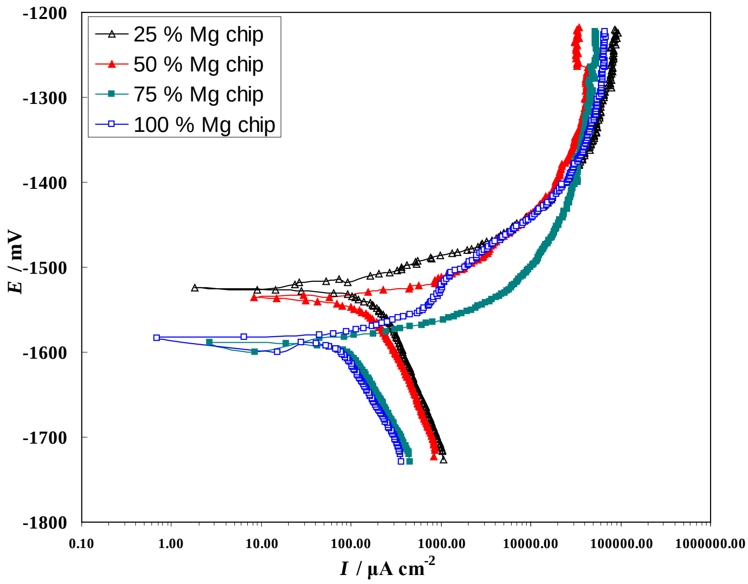


Figure 7. Effect of Mg chip content on the corrosion rate of the recycled Mg alloys in 3.5 % NaCl solution.

The polarization behaviour of the recycled Mg alloys is shown in Figure 8. The polarization results showed that the 75 and 100 wt.% Mg chip addition resulted in E_{corr} shifting negatively by ~ 50 mV. I_{corr} value increased with the 50 wt.% Mg chip addition to the recycled Mg alloy whereas above 50% Mg chip addition shifted the I_{corr} to a more lower region. These results are very much in line with the immersion tests.



Alloy	25 % Mg chips	50 % Mg chips	75 % Mg chips	100 % Mg chips
E_{corr}, (mv)	-1520	-1540	-1590	-1580
Icorr, ($\mu A/cm^2$)	203	490	44	148

Figure 8. Potentiodynamic polarisation curves for the recycled Mg alloys in 3.5% NaCl solution with corresponding E_{corr} and I_{corr} values.

Figure 9 shows the surface micrographs of the immersed samples in 3.5 % NaCl solution. It was noted that roughness of the corroded sample surface had been decreased by increasing the chips wt.% in the products. The results of corrosion test carried out in this study are in good agreement with the result of Chino et.al. [4], who studied corrosion behaviour of a Mg alloy recycled by solid-state process. These researchers attributed the enhancement of corrosion resistance for the recycled samples to the presence of oxide contaminants which were distributed parallel to the extrusion direction. In the present work, it is believed that a decrease in the electrical conductivity of the recycled Mg alloys by increasing wt.% of chip (Figure 2) may be due to a greater contamination of oxide with the increase in the total surface area of the machined chips per unit volume. The contamination of oxide particles is likely to be responsible for the lower corrosion rate in the solid recycled samples.

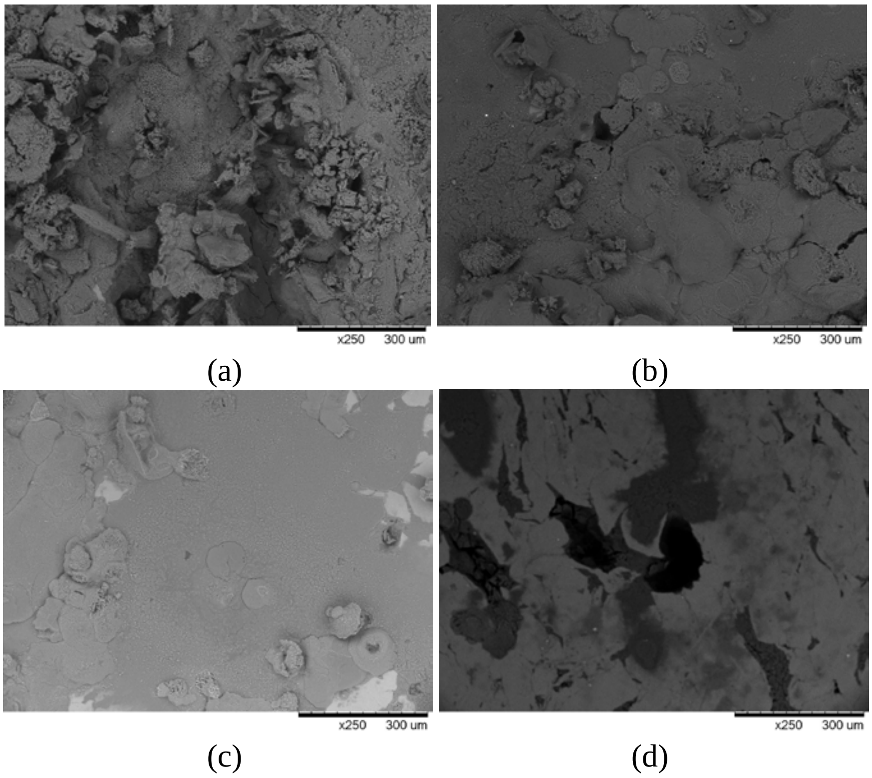


Figure 9. Corrosion surfaces of the recycled Mg alloys including (a) 25% chips, (b) 50% chips, (c) 75% chips and (c) 100 % chips.

4 Conclusion

Based on the above observations, following conclusions can be drawn on the role of Mg powder on the mechanical and corrosion properties of the recycled AZ91 alloy:

1. Addition of wt. % Mg powder to the recycled AZ91 alloy did not alter the density; however, Mg powder addition increased the electrical conductivity of the recycled AZ91 alloy considerably.
2. The hardness and compression strength of the recycled AZ91 alloy by Mg powder addition decreased by 28% and by 32% respectively. However, the wear resistance of the recycled AZ91 alloy increased with increasing Mg powder content, due to the formation of stronger bonding between chips in the recycled Mg alloys.
3. The Mg powder addition decreased the corrosion resistance of the recycled AZ91 alloy.

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