

## CRYOGENIC BURNISHING OF AZ31B Mg ALLOY FOR ENHANCED CORROSION RESISTANCE

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### Abstract

Poor corrosion resistance is limiting applications of Mg alloys. However, the corrosion performance of an Mg alloy can be enhanced through modification of its microstructure. It has been reported in the literature that the microstructure, especially grain size of AZ31 Mg alloy, has a significant influence on its corrosion resistance. In this study, AZ31B discs were subjected to a novel mechanical processing method—cryogenic burnishing; the surface of AZ31B work piece was burnished with a custom tool under a liquid nitrogen spraying condition. The processing led to a more than 3 mm thick surface layer with remarkably changed microstructures formed on the disc surface. Significant grain refinement occurred within this surface layer due to dynamic recrystallization induced by severe plastic deformation and effective cooling by liquid nitrogen. Both electrochemical method and hydrogen evolution method indicate that the corrosion resistance of the burnished surface was significantly improved.

### Introduction

Mg alloys are potential lightweight materials for automotive applications and the use of the alloys may remarkably improve vehicle fuel economy. However, the poor corrosion resistance of Mg alloys significantly limits their wide application. The corrosion performance of Mg AZ31 alloy is among the poorest when compared with other common cast Mg alloys, such as AZ91 or AM60.

Although various approaches, such as coating and alloying, have been extensively studied, the potential of grain refinement to improve the corrosion resistance of Mg AZ31 alloy has not been well investigated. Hot rolled AZ31 Mg alloy samples were reported to have increased corrosion resistance compared with squeeze cast samples, which was attributed to grain refinement from 450  $\mu\text{m}$  to 20  $\mu\text{m}$  [1]. Alvarez-Lopez et al. [2] reported that grain refinement from 25.7  $\mu\text{m}$  to 4.5  $\mu\text{m}$  after equal channel angular pressing (ECAP) led to better corrosion performance for Mg AZ31 alloy. Aung and Zhou [3] considered the grain boundary as physical corrosion barriers and claimed that smaller grain size improved the corrosion resistance. However, Song and Xu [4] reported that there was no evidence to support that changes in grain size and twin density by heat-treatment were principal causes of the reduced corrosion resistance in AZ31 Mg alloys.

While more efforts are needed to further investigate the relationship between grain size and corrosion of Mg alloys, there is a great demand for novel surface processing techniques which

can lead to improved corrosion resistance. Most of the researchers use severe plastic deformation (SPD) processes, such as equal channel angular pressing (ECAP), friction stir processing and high pressure torsion, to introduce grain refinement in Mg alloys. However, these processes are often very slow and heating may be required, such as for ECAP, due to limited ductility of Mg alloys at room temperature. Also, only small samples with simple geometry have been processed. Burnishing is a widely used process in industry to reduce surface roughness, increase hardness and/or introduce compressive residual stresses. Although there have been many publications on the beneficial effects of burnishing on fatigue life and/or wear of various materials, the corrosion performance of burnished materials have rarely been reported. Recently, Al-Qawabeha and Al-Rawajfeh [5] reported that the weight loss of galvanized steel samples after roller burnishing was reduced to only 6.5% of the value in the initial material. This is a significant indication that burnishing may also lead to improved corrosion resistance of a material.

In this study, a novel SPD method based on burnishing is used to change the surface microstructure of the AZ31B Mg alloy in order to understand the influence of the novel surface processing technique on corrosion resistance.

### Experimental Work

#### Work Material

The work material studied was the commercial AZ31B-O magnesium alloy. The work material was received in the form of a 3 mm thick sheet. Disc specimens having 130 mm diameter were cut from the sheet and subsequently subjected to burnishing.

#### Burnishing Experiments

The burnishing experiments were conducted on a Mazak Quick Turn-10 Turning Center equipped with an Air Products liquid nitrogen delivery system, which sprays liquid nitrogen to the processing zone for cooling. The experimental setup is shown in Figure 1.

The AZ31B Mg disc was fixed in the lathe chuck and rotated during processing. A roller made of high speed steel was pushed against the discs at certain feed rate. Different from the traditional burnishing method, the roller used here was fixed and does not rotate in order to introduce more severe plastic deformation. During processing, liquid nitrogen was sprayed to the processing zone as shown in Figure 1. The application of

liquid nitrogen was to remarkably reduce the temperature during processing and suppress the growth of ultrafine/nano grains introduced by dynamic recrystallization (DRX).

The burnishing speed refers to the linear speed at the contact point between the fixed roller and the disc. It was 100 m/min. The feed rate was 0.01 mm/rev. The process was stopped when the final diameter reduced to 125 mm.

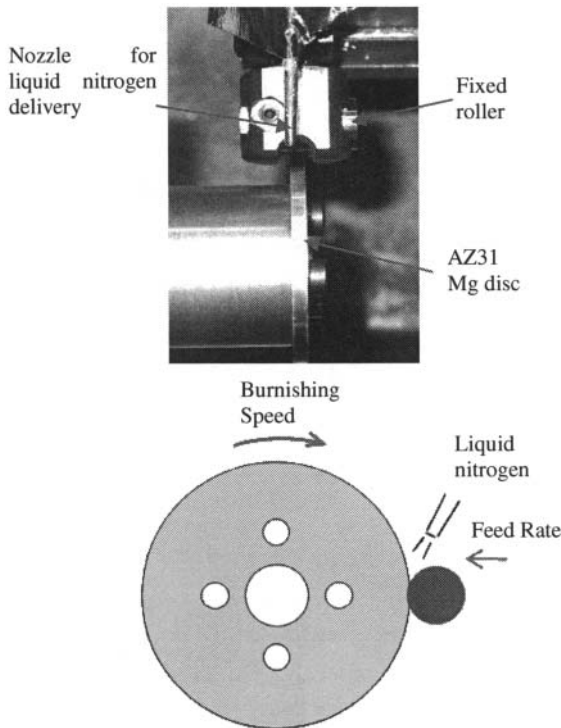


Figure 1 Burnishing setup with an Air Products liquid nitrogen delivery system

#### Grinding Treatment

To eliminate the possible influence of surface roughness on corrosion resistance [4], the unprocessed AZ31B Mg samples were ground successively using 4000 grit sand paper. Samples after grinding serve as the reference for corrosion resistance comparison.

#### Characterization Method

After burnishing, metallurgical samples were cut from the burnished discs. After cold mounting, grinding and polishing, acetic picric solution was used as an etchant to reveal the grain structure. A KEYENCE digital microscope VHX-600 was used to observe and record the microstructures of the burnished samples.

Surface roughness after grinding and burnishing were measured using a ZYGO New View 6000 measurement system which was based on white light interferometry.

The hardness of the samples was measured using a Hysitron TriboIndenter. The load used was 8 mN.

#### Electrochemical Measurement

A Solatron 1280 potentiostat system was used for polarization curve and AC impedance measurements. Only the processed surface was exposed to the testing solution and all the other surfaces are protected by a thick layer of MICROSTOP lacquer. The exposed area was 1.5 cm<sup>2</sup>. The testing solution was 5 wt. % NaCl. A platinum gauze was used as a counter electrode and a KCl-saturated Ag/AgCl electrode was used as a reference in the cell. During AC impedance measurements, the frequency ranged from 17,777 Hz to 0.1 Hz with 7 points/decade, and the amplitude of the sinusoidal potential signal was 5mV with respect to the OCP. Potentiodynamic polarization curve measurements were performed at a potential scanning rate of 0.1 mV/s from -0.3 V vs. OCP to -1.0 V vs. reference.

#### Hydrogen Evolution Measurement

In addition to electrochemical methods, hydrogen evolution method [6] was also used to compare the corrosion rates of samples after cryogenic burnishing and after grinding. The samples were mounted in epoxy resin and only the processed surface was exposed to 5 wt. % NaCl. The exposed area was 1.5 cm<sup>2</sup>. Pipettes with 0.1 mL interval were used to collect the evolved hydrogen from the samples.

### Results and Discussion

#### Microstructure

Figure 2 shows an overview of the microstructure after cryogenic burnishing. There is a clear interface between the processing-influenced zone and the bulk. This interface is also shown in Figure 3 under  $\times 1000$  magnification. The total thickness of the processing-influenced layer is  $3.40 \pm 0.01$  mm.

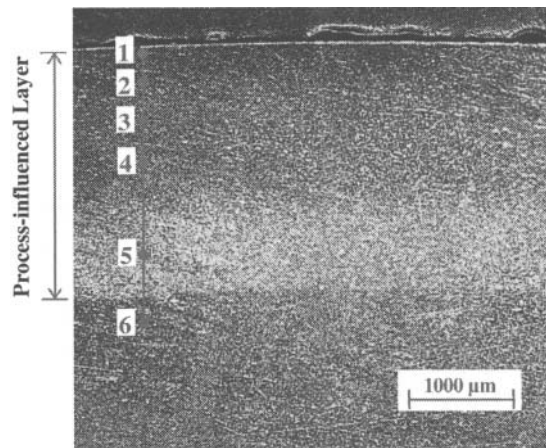


Figure 2 Microstructure after cryogenic burnishing ( $\times 30$  magnification)

While no twinning can be seen in the initial material, there is a high density of deformation twins above the interface as shown in Figure 3. The location of twinning is near the bottom of the processing-influenced layer. Twinning gradually disappears as

one moves from the bulk to the top surface. The deformation twinning indicates that the temperature near this interface is lower when compared with the top portion of the layer.

A clear evidence of dynamic recrystallization (DRX) is observed in Figure 4. The microstructures at different points in Figure 2 were shown at a  $\times 5000$  magnification using the VHX-600 digital microscope.

The image at Point 6 in Figure 4 represents the initial microstructure and Point 1 is the microstructure near the surface after cryogenic burnishing. It is clear that significant grain refinement occurred near the surface. As shown in Figure 5, the grain size after cryogenic burnishing is reduced to  $1.03 \pm 0.26 \mu\text{m}$  from the initial grain size of  $11.88 \pm 4.54 \mu\text{m}$ . Not only is the grain size reduced, but also the distribution of grain size becomes more uniform (smaller in scatter).

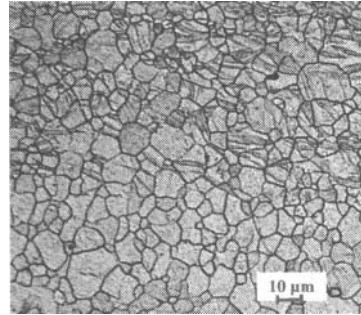


Figure 3 Interface between initial material and process-influenced layer after cryogenic burnishing ( $\times 1000$  magnification)

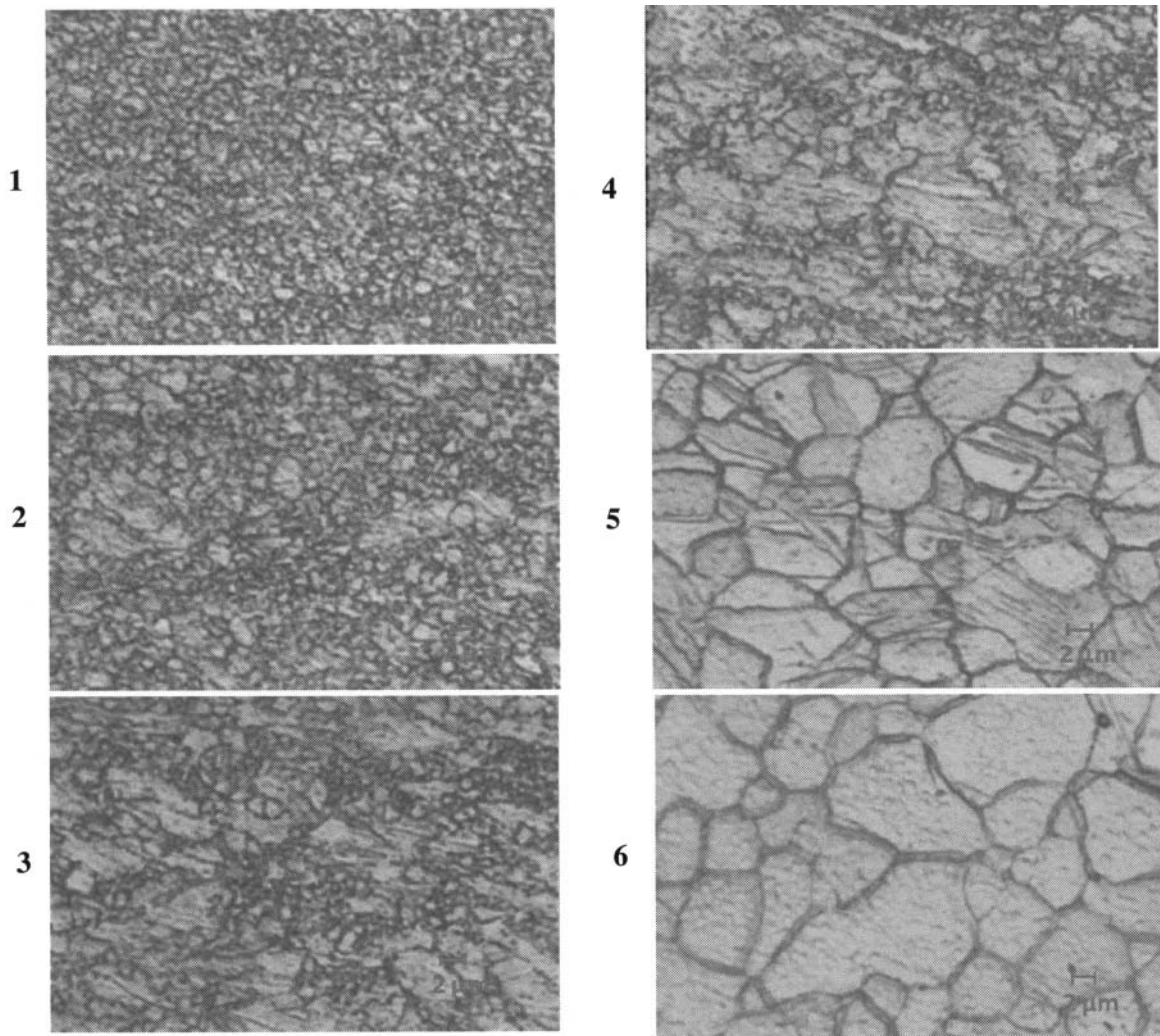


Figure 4 Microstructures at different depths after cryogenic burnishing ( $\times 5000$  magnification)

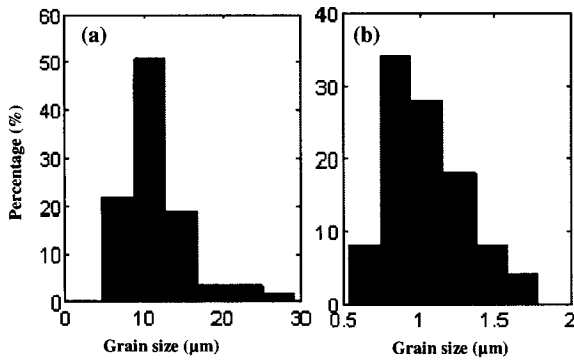


Figure 5 Distribution of grain size: (a) initial; (b) after cryogenic burnishing at Point 1.

From Point 2 to Point 4, there is a clear trend that the amount of ultrafine grains is decreasing. Fatemi-Varzaneh et al. [7] investigated the effects of temperature, strain and strain rates on dynamic recrystallization of AZ31 Mg alloy in detail and reported that the amount of dynamically recrystallized grains increased with strain in a sigmoidal form. The strain induced by cryogenic burnishing should decrease from the surface to the bulk material where the material was not influenced by the process. This agrees with the literature that the amount of dynamically recrystallized grains will decrease when the strain becomes smaller.

The microstructural features at Point 6 in Figure 4 further shows that deformation twins are dominant in the transition layer from the processing-influenced microstructure to the initial microstructure.

#### Hardness Measurement

As shown in Figure 6, the hardness far away from the surface, which is not influenced by the processing, is about 0.9 GPa. After cryogenic burnishing, the hardness near the surface reaches 1.35 GPa. The relationship between hardness and grain size in AZ31 Mg alloys has been frequently reported in literature [8]. The large increase in hardness agrees with the previous finding that significant grain refinement occurs near the surface after cryogenic burnishing.

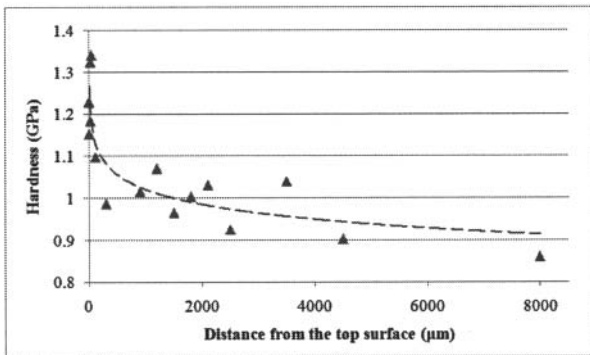


Figure 6 Hardness variation from the top surface to the bulk material

#### Surface Roughness

Figure 7 shows a comparison of surface roughness (Ra) between grinding and cryogenic burnishing. It shows that grinding creates slightly better surface roughness and should result in better corrosion resistance.

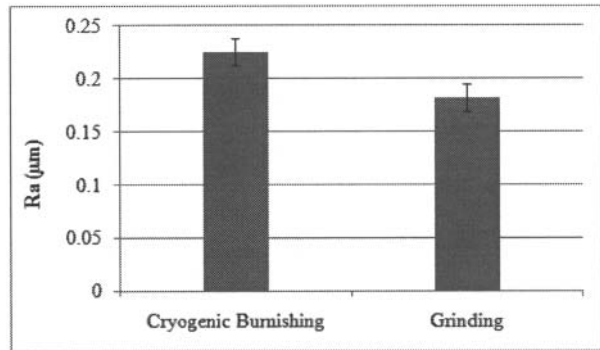


Figure 7 Comparison of surface roughness between cryogenic burnishing and grinding

#### Electrochemical Measurement

The polarization curves of samples after grinding and after cryogenic burnishing are presented in Figure 8. It shows that the cathodic polarization current density after cryogenic burnishing is smaller than the one after grinding, which suggests that cryogenic burnishing leads to improved corrosion resistance. However, there is a large shift in corrosion potential from -1.44 mV after grinding to -1.53 mV after cryogenic burnishing. Similar findings were reported by Balakrishnan et al. [9] where ultra-fine-grained (238 nm) Ti has a lower corrosion potential than coarse grained Ti (15.2 micrometers). While in general, metals with lower potential are prone to more corrosion; both the literature and the current study show the opposite trend. There is another possibility that quicker passivation of the surface layer may retard the corrosion process [10].

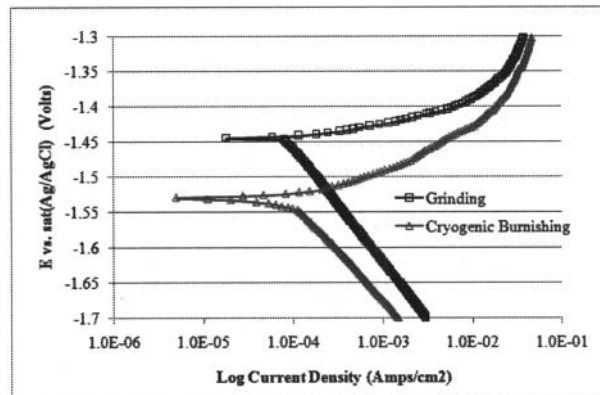


Figure 8 Polarization curves of AZ31B Mg samples after grinding and cryogenic burnishing in 5 wt. % NaCl

Figure 9 shows the Nyquist diagrams of AZ31B Mg samples after grinding and cryogenic burnishing in 5 wt.% NaCl. Both curves have a clear capacitive arc at the high frequency region. The diameter of this capacitive loop at the high frequency region is associated with the charge-transfer resistance. Makar and Kruger [11] shows that for magnesium alloys, larger diameter indicates better corrosion resistance. The diameter for the sample after cryogenic burnishing is remarkably larger than the one after grinding, which suggests the sample after cryogenic burnishing has better corrosion resistance than the ground sample. This finding agrees with the trend of cathodic polarization current densities as shown in Figure 8.

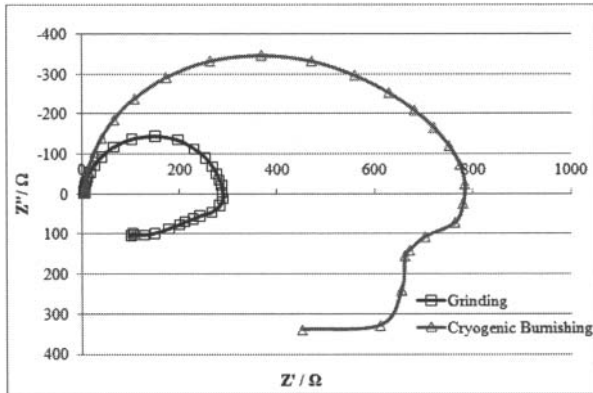


Figure 9 Nyquist diagrams of AZ31B Mg samples after grinding and cryogenic burnishing in 5 wt.% NaCl

#### Hydrogen Evolution Measurement

The hydrogen evolution of the samples in 5 wt. % NaCl after grinding and burnishing are presented in Figure 10. It shows that more hydrogen is generated from the ground samples. Also, the data scatter after grinding is larger than after cryogenic burnishing. Since cryogenic burnishing was carried out automatically on a CNC machine, it is expected that the process is more repeatable than grinding by hand. The finding from hydrogen evolution measurement further proves that the corrosion resistance of the AZ31B Mg alloy after cryogenic burnishing is improved compared with the one after grinding.

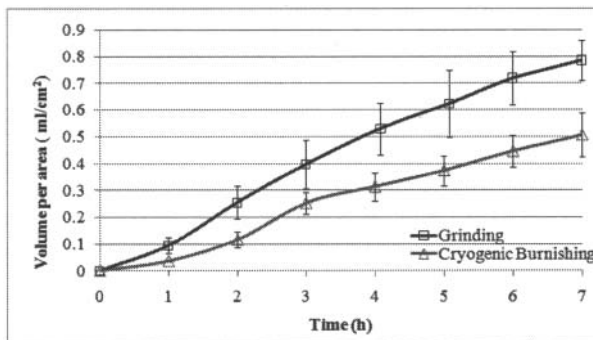


Figure 10 Hydrogen evolution of AZ31B Mg samples after grinding and cryogenic burnishing in 5 wt.% NaCl

#### Conclusion

The present study shows that significant grain refinement as well as a large increase in hardness can be achieved in the surface layer of AZ31B Mg alloy after cryogenic burnishing. The microstructure of AZ31B at depths up to 3.4 mm away from the surface can be remarkably changed by cryogenic burnishing. The mechanism for grain refinement is dynamic recrystallization.

Both electrochemical method and hydrogen evolution method show that the corrosion resistance of AZ31B Mg alloy is improved after cryogenic burnishing. This agrees with other literatures that smaller grain size leads to better corrosion resistance of AZ31B Mg alloy.

In addition, it reveals a great opportunity to improve material performance through fabricating a grain refinement surface layer by cryogenic burnishing. Not only corrosion resistance, but other properties, such as fatigue and wear resistance may also be significantly enhanced if proper processing conditions are used.

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