

DYNAMIC MECHANICAL ANALYSIS OF PURE Mg AND Mg AZ31 ALLOY

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Abstract

Dynamic mechanical analyses were performed on pure Mg and Mg AZ31 alloy to study the impact of thermo-mechanical state and grain structure on the complex modulus ($E^* = E' + iE''$). The storage modulus (E') of pure Mg was found to be 40.0 ± 2.5 GPa and independent of grain size. A thermally activated loss modulus (E'') peak with an activation energy of 1.39 ± 0.19 eV, which is very close to the activation energy of the self-diffusion of Mg (1.408 eV), was observed and attributed to a grain boundary relaxation mechanism. The temperature of this peak depends on orientation with respect to direction of solidification as well as on heat treatment. The behavior of Mg AZ31 alloy was similar to that of pure Mg. The storage modulus determined for this alloy was 42.0 ± 2.5 GPa and it was independent of rolling direction. The thermally activated peak in Mg AZ31 was sharper and was observed at lower temperatures (167-174°C) with an activation energy of 1.68 ± 0.04 eV.

Introduction

There is a growing demand in the automotive industry for reducing vehicle weight to increase efficiency and reduce pollution. This goal can be achieved by either replacing of various automotive components (e.g., hoods, doors and bodies) with components made from light weight metals, like Mg or Al alloys, or using higher strength alloys in thinner cross sections.

In spite of the use of advanced technologies, the application of advanced materials by the automotive industry has been hindered by the lack of experience with forming these alloys. One area of concern is the springback effect (i.e. the elastic shape changes that occur due to the residual stresses developed during stamping). It is estimated that the US auto industry spends more than \$700 million a year redesigning, and correcting dies to compensate for springback⁽¹⁾. Computer simulations are being developed to eliminate or reduce the cost of die tryout and springback compensation. However, the ability of these simulations to accomplish this objective is strongly depended on knowledge of the mechanisms that contribute to springback and the measurements used to quantify these properties for forming alloys.

Recently, the automotive industry has exhibited increasing interest in Mg alloys and the formability of new higher ductility, corrosion resistant, Mg alloys. For example, die cast magnesium components have been used for applications such as clutch housings, gearboxes; pedal brackets, instrument panel frames, integral seat frames and wheel hub cover components^(2,3). However, the range of potential applications for Mg alloys has been limited by our lack of knowledge and experience with forming of Mg alloy sheet.

To benefit from the weight saving advantages of magnesium alloys, a whole new level of formability measurement methods

and data is needed, together with better understanding of the physics behind deformation of Mg alloys. The goal of this investigation was to develop a better understanding of the properties and measurements that would required in a database of Mg alloy properties that designers can feed into computer models for designing more affective dies to compensate for springback effects. In this report we shall describe our preliminary study of Dynamic Mechanical Analysis-(DMA) of pure Mg and Mg AZ31 alloy.

Experimental

Pure cast Mg and commercial Mg AZ31 H24 alloy were used for Dynamic Mechanical Analysis experiments. The pure Mg alloy was cast in a metal permanent mold and solidified with low cooling rates (less than several mm/sec). However, due to the thermal conductivity of the mold and the low heat capacity of the Mg, platelets about 2 mm thick, 3 to 5 mm wide, and several cm long were formed. On the top surface and up to 6 mm from the top surface, equiaxed grains approximately 5 mm in diameter were observed. For the AZ31 alloy, commercial H24 plates were used with a small grain size of approximately 10 μ m.

Rectangular specimens were manufactured from different area of the pure Mg and the AZ31 alloy. The specimens had of the following dimension: 2-3 mm thick, 10 mm wide and 60 mm long. To remove defects introduced by specimen preparation, the specimens were annealed for 3-0 min. at 350°C. It was found that this heat treatment eliminates defects while not changing the original grain size significantly.

In order to find the impact of heat treatment on the storage and loss moduli the specimens were heat treated for various times and temperatures in air.

Dynamic Mechanical Analysis

Dynamic mechanical analysis is a thermal mechanical analysis technique that measures the properties of materials as they deformed under periodic stress as a function of temperature (or time). In this technique a sinusoidal displacement is applied to the specimen which induces a sinusoidal force. In materials that exhibit viscoelastic behavior, which includes most materials, a phase difference (δ) forms between the stress and strain sinusoids, which is measured. From the measured phase difference (δ) and the stress and strain amplitudes, one determines the complex modulus ($E^* = E' + iE''$) where the real component or storage modulus (E') represents the energy returned elastically by the sample and the imaginary component or loss modulus (E'') represents the ability of the material to dissipate energy in the form of heat through some relaxation mechanism in the material.

The samples were tested in three-point bend with constant displacement amplitude of 15 μ m. To prevent sample lift-off during the experiments a static preforce between 0.1 N and 0.5 N

was used and this becomes the minimum load for each load cycle. The controller was set to automatically adjust during the experiment to account for changes in the sample and keep these values constant.

Results and Discussion

Pure Mg

The macrostructure of pure Mg is illustrated in Figure 1. From this figure it can be seen that there is considerable anisotropy to the grain structure of this alloy due to the solidification procedure. In order to evaluate the influence of the grain anisotropy on the storage (Young) modulus and the loss modulus of pure Mg, specimens were cut oriented with the tensile axis parallel and perpendicular to the direction of grain growth for different heat treatments. The storage modulus and loss modulus of pure Mg. For specimen parallel to grains and for specimen perpendicular to grains Figure 2a and 2b, respectively.

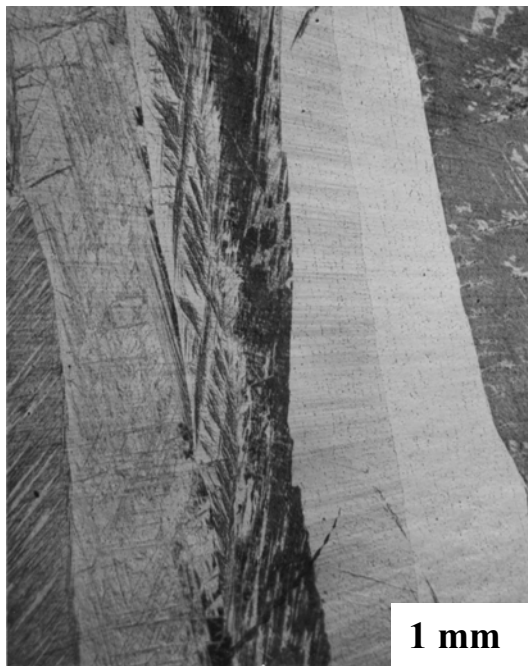


Figure 1) Macrostructure of Pure Mg illustrating the grain Microstructure

From this figure, it can be seen that the storage modulus is changing between 38 GPa to 43 GPa at 25°C for specimens perpendicular and parallel to the grains, respectively. However, the uncertainty in these measurements is of the order of ± 2.5 GPa. The storage modulus exhibited the same general behavior in all specimens tested. In contrast, the loss modulus changed with annealing temperature as shown in this figure. The peak observed around 210°C to 235°C, varied with grain size (with smaller grain producing peaks at higher temperatures). Qualitatively, it is believed to be a peak for a grain boundary relaxation mechanism. The peak of the grain boundary was sharp with the increasing of the heating temperature. This value is similar to the value of 152°C that was found for Mg AZ91

by Lambri et al⁽⁶⁾, who also attribute the peak to grain boundary relaxation.

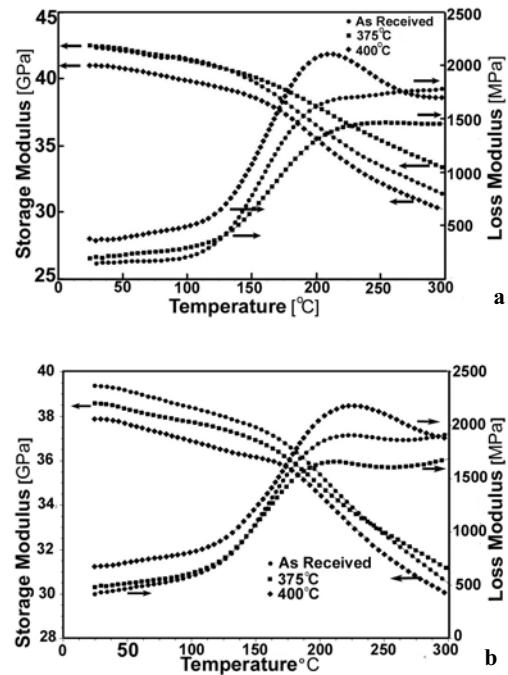


Figure 2) Storage modulus and loss modulus of pure Mg. a) Specimen parallel to grains, b) Specimen perpendicular to grains

The nature of a relaxation peak mechanism can be investigated by observing the temperature of the peak at different testing frequencies. If the mechanism is thermally activated, then the peak will shift in temperature with testing frequency (as seen in Figure 3) and the activation energy of the relaxation mechanism can be estimated by linear regression as shown in figure 4.

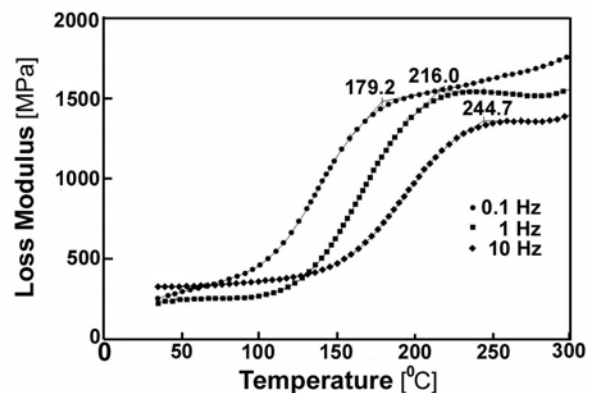


Figure 3) Loss modulus of pure Mg in the as cast conditions at 3 different frequencies

In this manner, the activation energy of this peak was determined to be $(1.39 \pm 0.19 \text{ eV})$ which is very close to the activation energy reported for self diffusion of Mg (1.408 eV) ⁽⁷⁾.

It might be that the additional element, especially Al and Zn changing the nature of the grain boundary interface.

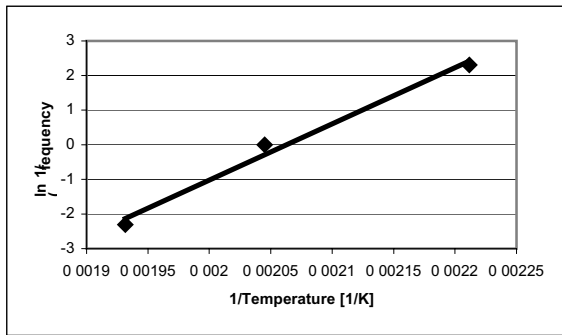


Figure 4) $\ln(1/\text{frequency})$ as a function of $1/\text{Temperature}$ for pure Mg in the as cast condition

Mg AZ31

The microstructure of Mg AZ31H24 after heat treatment of 2 hrs at 350°C is illustrated in Figure 5. Essentially this is an equiaxed microstructure, but, as is common with AZ31 alloy, this microstructure has a bimodal grain size distribution, as shown in Figure 5. The storage and loss moduli for different heat treatments are shown in Figure 6. We did not find a difference in the storage and loss moduli for different sample orientations with respect to rolling direction. The loss modulus as a function of temperature for 4 different frequencies is given in Figure 7. In the same way as described for pure Mg the loss modulus peak is created by a thermally activated process (see Figures 7 and 8) with an activation energy of 1.68 ± 0.04 eV. This value is slightly higher than that for pure Mg (1.37 eV). We attribute this increase to the additional element present in AZ31 alloy. It might be that the additional element, especially Al and Zn changing the nature of the grain boundary interface. A comparison of the loss modulus behaviors of AZ31 and pure Mg after heat treatment of 375°C for 50 hrs is shown in Figure 9. In this Figure it can be seen that the loss modulus of pure Mg usually is higher and the grain boundary relaxation peak is broader than the peak of the AZ31 alloy.

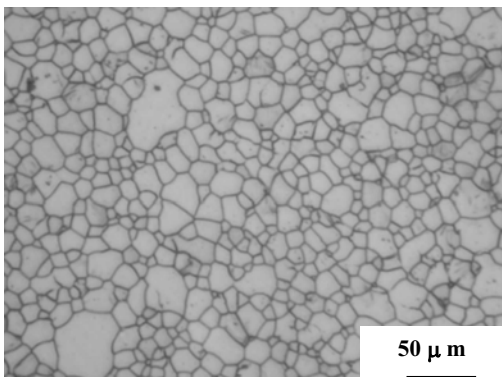


Figure 5) Microstructure of AZ31 used.

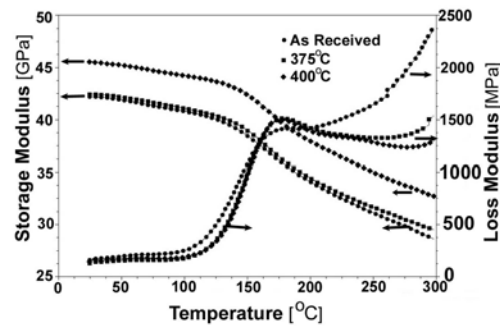


Figure 6) Storage (Young) modulus and loss (internal friction) modulus for Mg AZ31.

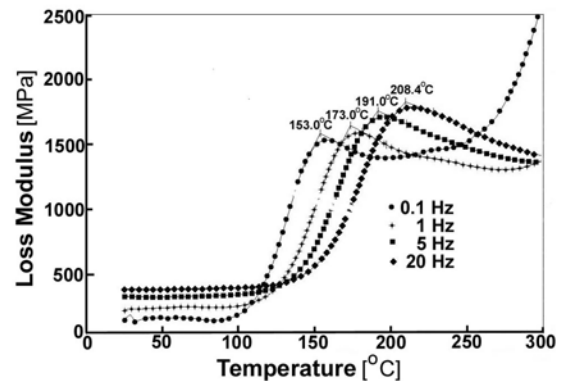


Figure 7) Loss modulus of pure Mg in the as cast conditions at 4 different frequencies.

A comparison of the loss modulus behaviors of AZ31 and pure Mg after heat treatment of 375°C for 50 hrs is shown in Figure 9. In this Figure it can be seen that the loss modulus of pure Mg usually is higher and the grain boundary relaxation peak is broader than the peak of the AZ31 alloy. It might be that the additional element, especially Al and Zn changing the nature of the grain boundary interface.

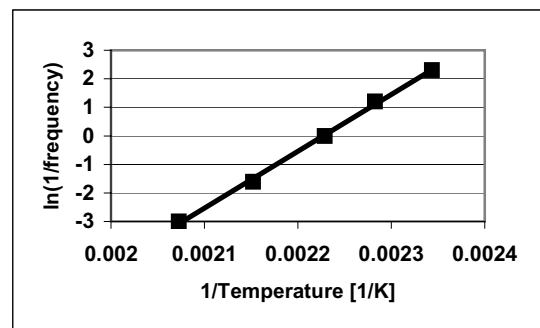


Figure 8) $\ln(1/\text{frequency})$ as a function of $1/\text{Temperature}$ for AZ31.

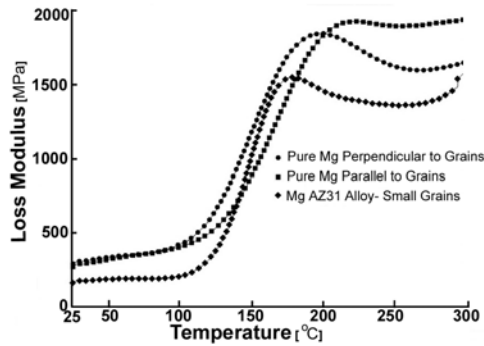


Figure 9) Comparison between the loss modulus of pure Mg perpendicular and parallel to grain growth compare to that of Az31 Alloy after heat treatment of 375°C for 50 hrs.

It should be mentioned that from Figure 9 it seems that the grain boundary relaxation peak is actually a superposition of two peaks one around 174°C and the second one around 243°C. The source of the second peak is still unknown and it is under investigation. From Figures 2, 6 and 9 one can find the temperatures of both peaks as it summarized in Table 1.

Table 1: Summary of temperature (in °C) of the grain boundary relaxation peak and the second Peak for pure Mg (perpendicular and parallel to grain growth and for Mg AZ31 alloy.

Alloy State	Pure Mg				AZ31 Alloy	
	Parallel to Growth		Perpendicular to Growth		Small Grains	
	1 st	2 nd	1 st	2 nd	1 st	2 nd
As Received	201	264	203	263	167	Not clear
After Heat treatment of 400°C for 50 hrs	207	>300	221	>300	174	243

Summary

Dynamic mechanical analyses were performed on pure Mg and Mg AZ31. The following results were obtained:

Pure Mg

- The storage modulus of pure Mg, under our experimental conditions was found to be 40.0 ± 2.5 GPa at 25°C. It should be noted that there are indication that the storage modulus of specimens cut perpendicular to growth direction was slightly lower than that of specimens parallel to growth direction but that the difference was of the uncertainty in these measurements. This subject is currently under investigation.

- The loss modulus relaxation peak was essentially composed of two peaks. The origin of the second peak is still unknown and is currently under investigation. The first peak, that we attribute to grain boundary relaxation is similar for both directions (201-203°C) in the as cast condition. However, heat treatment shifted the peak of the specimen to higher temperatures (207, 221°C for specimen manufacture parallel and perpendicular, respectively). The second peaks temperatures increase too with annealing and its temperature increases above 300°C.

Mg AZ31 alloy

- The storage modulus was found to be 42.0 ± 2.5 GPa at 25°C, and was independent of rolling direction and annealing temperature. The role of cold rolling, the effect of grain size, and microstructure on the storage and loss moduli of Mg AZ31 alloy is currently under investigation.
- The loss modulus was similar to that of pure Mg, but with lower values. The peak also appeared to be a combination of two peaks, but at lower temperatures 167°C and 174°C for as received and annealed at 400°C for 50 hrs, respectively. The second peak can be revealed only after heat treatment and appeared at 243°C. However, mathematical analysis show that this peak is also present in the as received condition.
- The grain boundary relaxation peak was thermal activated for Pure Mg as well as Mg AZ31 alloy. The activation for the pure Mg 1.386 eV, which is very close to the activation energy of the self-diffusion of Mg (1.408 eV). The activation for the AZ31 is 1.68 eV.

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