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APPLICATION OF NEUTRON DIFFRACTION IN CHARACTERIZATION OF TEXTURE EVOLUTION DURING HIGH-TEMPERATURE CREEP IN MAGNESIUM ALLOYS

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Abstract

A good combination of room-temperature and elevated temperature strength and ductility, good salt-spray corrosion resistance and excellent diecastability are frequently among the main characteristics considered when developing a new magnesium alloy. Unfortunately, much less effort has been expended developing wrought-stock alloys for high temperature applications. Extrudability and high temperature performance of wrought material becomes an important factor in an effort to develop new wrought alloys and processing technologies.

This paper shows some results obtained from creep testing and studies of in-creep texture evolution, for several wrought magnesium alloys developed for use in elevated-temperature applications.

Introduction

Despite the enormous efforts expended to develop high-strength, high-temperature magnesium alloys, the most typical applications of magnesium in the automotive industry are still limited to a few select applications such as instrument panels, steering wheels, and valve covers. Limited success was attained in the use of magnesium in powertrain applications such as transmission cases and engine blocks [1-9]. These applications experience service conditions within the temperature range of 150-200°C under 50-70 MPa of tensile and compressive loads. In addition, metallurgical stability, fatigue resistance, corrosion resistance, and castability requirements need to be met. More than a decade of research and development has resulted in a number of creep-resistant magnesium alloys that are potential candidates for elevated-temperature automotive applications. These alloys are mostly based on rare-earth and alkaline-earth element additions to magnesium. A number of alloys are based on additions of Si, Sr, and Ca.

Although the majority of the material used in the automotive industry is in the form of castings, the use of wrought products in automotive applications is on the rise. Extruded sections provide opportunities for the mass-efficient design of structural and interior automobile components.

One of the main criteria for acceptable material performance in high temperature automotive applications is its resistance to creep. However, low resistance to creep deformation at elevated temperatures has been one of the main restricting factors in applying magnesium alloys.

Depending on the manufacturing route and resulting grain/crystallographic matrix, wrought magnesium alloys can exhibit quite different creep behavior, compared to similar cast alloys. Several creep-resistant wrought alloys have been studied in [10, 11]. It was shown that magnesium exhibits different creep properties under tension and compression. These studies also presented data gathered from the analysis of crystallographic texture and creep-induced residual stress –factors that would most certainly affect service properties of the material. The targeted alloying groups were magnesium-aluminum-rare earth, magnesium-aluminum-strontium, magnesium-aluminum-calcium, and magnesium-zinc-rare earth. The chemical compositions of the analyzed samples correspond to the following alloy designations: AE42, AE33, AX30, AZX310, AJ32, EZ33, and ZE10. The seven alloys targeted in this study were produced by the magnesium division of Timminco Corporation, now Advanced Magnesium International (AMI). The material was cast using a unique controlled-cooling static casting process, followed by hot extrusion. All alloys exhibited exceptionally good castability, as well as formability in the extrusion process.

The current analysis is part of a continuing effort to develop low-cost, wrought magnesium alloys with improved castability and formability, suitable for high temperature applications, and to add to the understanding of material behavior during high-temperature creep.

High-Temperature Creep Testing

The wrought material received from AMI was subsequently subjected to a tensile creep test at 150 and 175°C and then to a 200-hour compressive creep test at 150°C under a load of 50 MPa. All the samples were creep-tested along the extrusion direction. The following charts (Figure 1) illustrate some selected results of these tests.

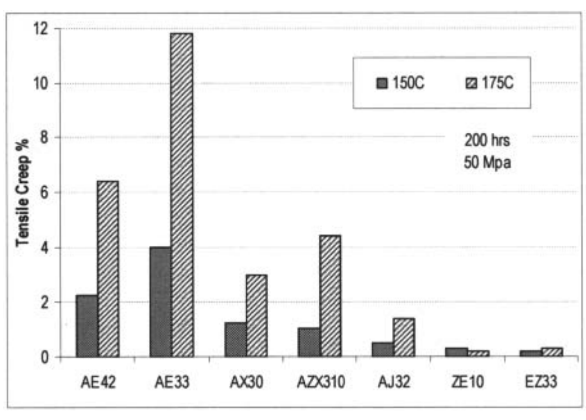


Figure 1. Resistance to creep for selected alloys at 150 and 175°C

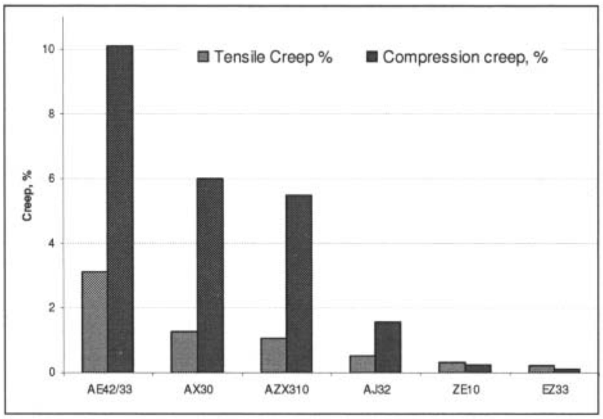


Figure 2 Tension-compression asymmetry in resistance to creep, at 150°C

Data shown in Figure 1 illustrate the comparison between results obtained in the tensile tests at 150 and 175°C, which included both primary and secondary creep. As expected, the 25°C temperature increase lead to significantly reduced resistance to creep, typically a reduction by a factor of three for all alloys, except for ZE10 and EZ33. Evidently, the manufacturing route applied in this study (permanent mould casting + hot extrusion) resulted in superior high temperature creep properties for the Mg-Zn-RE alloying system.

This finding was also confirmed in the compressive creep test, performed at 150°C. It is known that HCP crystallographic systems typically exhibit greatly reduced resistance to creep in compression. This can also be observed in Figure 2, which shows the tension/compression asymmetry in creep resistance for the alloys studied. Figure 2 indicates that for identical applied loads and test durations, resistance to creep was reduced by a factor of 3.5 to around 4 for all of alloys, with the exception, again, of ZE10 and EZ33. It can be concluded from these observations that, compared to the other samples studied, the applied manufacturing route favourably affected the ability of the Mg-Zn-RE alloying system to resist high-temperature creep, both in tension and in compression. In addition, almost no tension-compression asymmetry was observed for the ZE-group samples (in all tests, the resulting creep was within 0.25 to about 0.3% for the ZE10 samples and within 0.1 to about 0.2 for EZ33).

Figures 1 and 2 also show that in all tests the AE-group samples exhibited inferior creep properties, compared to the other three alloying systems. This was partially explained in [11] by the presence of β-phase $Mg_{17}Al_{12}$, along the grain boundaries in the magnesium matrix, which significantly reduced creep resistance at elevated temperatures. A reference was also made to the possible effect of initial texture (i.e., texture prior to creep).

Texture Evolution during Creep Deformation

It was suggested in [11] that the strength of the initial extrusion-type crystallographic texture may be another factor affecting the material resistance to creep. It can be assumed that, considering the amount of creep deformation obtained in this study (up to 10~12% strain), there could be noticeable texture evolution or modification during creep testing. These two assumptions were verified by texture analysis performed in the HIPPO (High-Pressure-Preferred-Orientation) time-of-flight spectrometer at Los Alamos Neutron Science Center. The time-of-flight method employed by HIPPO for texture analysis is well described in earlier study [12].

Similar studies have earlier been performed at the Canadian Neutron Beam Centre at Chalk River, ON, on pure magnesium and Mg-1.5wt.%Mn samples [13, 14]. It was confirmed that the tested material underwent significant texture modification during creep testing at 150°C and under a load of 50MPa. A typical "rolling" texture could be observed on the crept specimens. Important conclusions were reached regarding creep deformation mechanisms for the studied materials.

The pictures presented in Figures 3 and 4 show the results of texture calculation using the E-WIMF algorithm of the MAUD texture-analysis software [12], based on neutron measurements for several selected alloys.

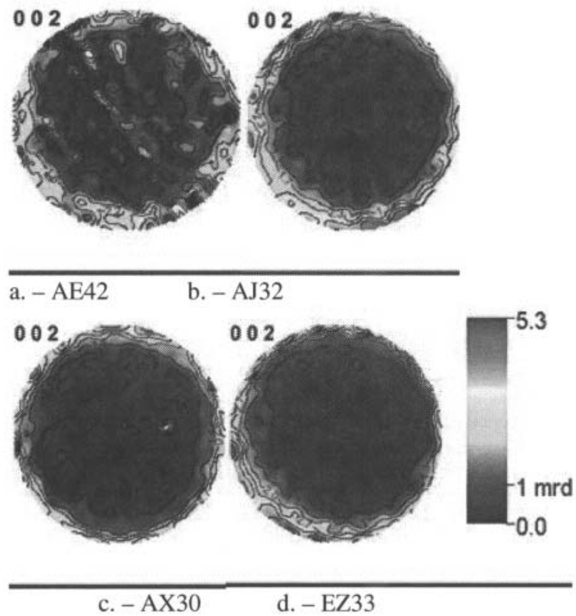


Figure 3 As-extruded texture for selected alloys, (0002) reflection

As expected, Figure 3 indicates that the selected alloys exhibited typical magnesium extrusion texture, with the (0002) poles aligned preferentially normal to the extrusion axis. The basal pole figures have similar strength for the basal texture, ranging from 4.5 m.r.d. (i.e. multiple of random distribution) for AE42 to 5.4 m.r.d. for EZ33.

Figure 4, however, shows somewhat different intensities, for the prismatic $\{10\bar{1}0\}$ orientation, for the initial texture, and for modified textures as a result of creep deformation. All samples in Figure 4 were identified in the following manner:

- As-extruded (i.e. not creep tested) – all *a.* samples;
- After the compression-creep test, 150°C – all *b.* samples
- After the tensile-creep test, 175°C – all *c.* samples

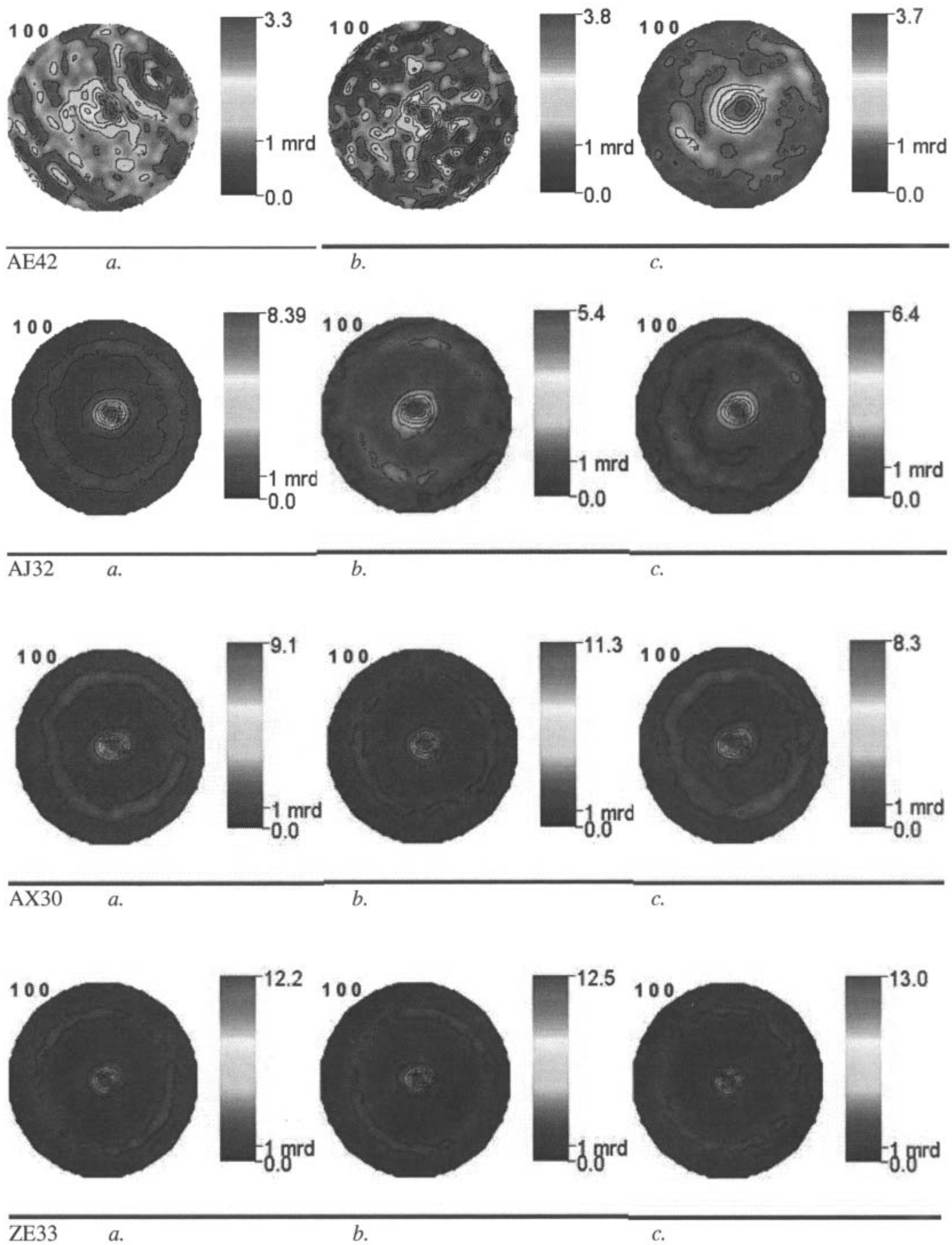


Figure 4 Texture evolution in creep tests for selected alloys, $\{10\bar{1}0\}$ reflection

Figure 4 shows that the strength of the initial as-extruded texture for the studied samples can vary for the prismatic reflection $\{10\bar{1}0\}$ pole figures, ranging from about 3 to about 12 m.r.d. Interestingly, the alloys that had relatively weak extrusion texture typically did not fare well in the creep testing (see Figure 1), an observation made in earlier studies [10, 11]. Fugure 4 also shows obvious texture modification on the AE42 specimen as a result of creep testing, particularly for the tensile creep specimen (see Figure 4, AE42, *c*.). Although the strength of the texture remains relatively the same, the grains exhibit further extrusion-type reorientation as a result of tensile creep deformation.
The other three alloys shown in Figure 4 exhibited much less texture evolution under creep, which is consistent with the observation that these alloys deformed much less than AE42. It can be suggested that the presence of the $Mg_{17}Al_{12}$ β-phase only partially explains the inferior performance of AE, AJ, and AX alloys compared to the EZ-group material.
It would be of interest to characterize in more details the phases present in the best-performing EZ33 alloy.

EZ33 – Second Phase Analysis

Unlike the other alloys, the EZ-group alloys clearly show the presence of a second phase in the diffraction pattern (see Figure 5). Along with easily identifiable peaks of α-magnesium, Figure 5 also depicts several [circled] peaks of another phase. This was further analyzed with application of SEM.

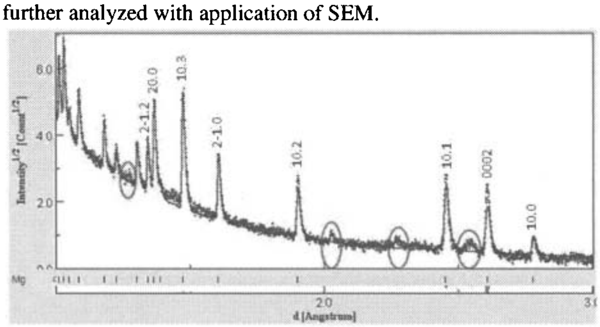
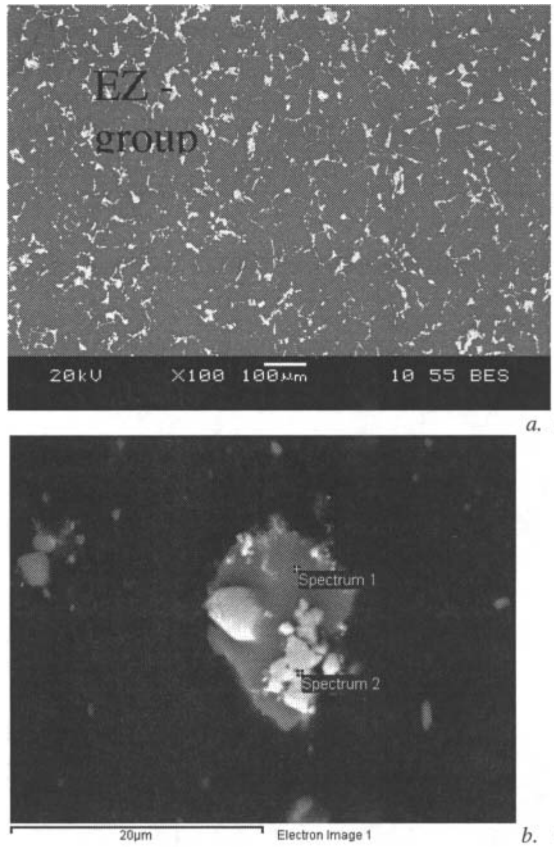


Figure 5 Neutron diffraction pattern obtained for EZ33 specimen

Figure 6*a* shows that a second phase is formed along the grain boundaries. Higher magnification (see Figure 6*b*) revealed that the second phase mostly consists of two different intermetallics, with most of the phase volume taken by magnesium-rich (up to about 85at.%Mg) complex $Mg_xZn_xZr_xRE_x$ intermetallic. The second (minority-phase) intermetallic that is formed inside this phase is mostly-binary Mn-Zr ($ZrMn_2$) inclusions, as seen in Figure 6*b* and Table 1. Diffraction peaks, however, associated with the $ZrMn_2$ crystallographic unit cell are different than the ones present in Figure 5, and are likely hidden in the background "noise", which could be expected considering that the volume fraction of this phase is minimal.

On the other hand, a significant volume fraction of the complex Mg-Zn-Zr-RE phase gives rise to the diffraction peaks detected in the neutron diffraction pattern in Figure 5.



a. 100X

b. 1000X

Figure 6 SEM studies of EZ33 specimen

Table 1
X-ray phase analysis, EZ33, spectrum 1 and 2 (Figure 6*b*)

EZ33 Specimen 1 Element	Wt. %	At. %	EZ33 Specimen 2 Element	Wt. %	At. %
Mg K	48.2	76	Mg K	1.9	5.4
Mn K	12.3	8.6	Mn K	39.2	49.0
Zn K	10.2	6.0	Zn K	-	-
Zr K	9.0	3.8	Zr L	52.8	39.7
La L	5.5	1.5	La L	-	-
Ce L	11.2	3.2	Ce L	-	-
Nd L	2.1	0.6	Nd L	-	-

Conclusions

1. Crystallographic texture studies confirmed that there is a notable difference in the strength of extrusion-type texture for the extruded samples of studied alloys, which represent four alloying systems: Mg-Al-RE, Mg-Al-Ca, Mg-Al-Sr, and Mg-Zn-RE. Two alloys representing the last system developed the strongest texture in the extrusion process (12.3 mrd for the $\{10\bar{1}0\}$ reflection). These alloys also performed the best during high-temperature creep testing, at temperatures of 150 and 175°C, both under compressive and

tensile loading. In contrast, two alloys from the AE group developed the weakest texture in the extrusion process (3.3 mrd for the $\{10\bar{1}0\}$ reflection). These alloys also exhibited the lowest resistance to creep during creep testing.

2. The neutron diffraction spectrum for all selected alloys, except for the ZE-group, revealed the typical one-phase (α-magnesium) spectrum. On the other hand, the diffraction pattern for the ZE alloys exhibited second-phase peaks arising from the presence of complex Mg-Zn-Zr-RE intermetallic. Another minority phase was revealed in the material during the SAE analysis, namely $ZrMn_2$, that was forming inside the larger second-phase intermetallic. Due to its limited volume fraction, the $ZrMn_2$ phase is not detected in the neutron diffraction patterns.

3. The effect of the presence of complex Mg-Zn-Zr-RE intermetallic on the actual mechanism of creep deformation in wrought Mg-Zn-RE(Zr) alloys remains to be studied.

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