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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 considerations in development of a new alloy. Unfortunately, there has been much lesser effort in development of 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 received in creep testing and studies of in-creep texture evolution for several wrought magnesium alloys developed for use in elevated-temperature applications. These studies were performed using E3 neutron spectrometer of the Canadian Neutron Beam Centre in Chalk River, ON, and HIPPO time-of-flight (TOF) spectrometer at Los Alamos Neutron Science Center, NM.

Introduction

Regardless of enormous efforts put in development of high-strength high-temperature magnesium alloys, the most typical applications of magnesium in automotive use is still limited to a few select applications such as the instrument panel, steering wheel, and valve cover. Limited success was reached in use of magnesium in powertrain applications such as the transmission case and engine block [1, 9]. These applications see service conditions in the temperature range of 150-200C 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 a large share of the material used in industry is in the form of castings, the use of wrought products in automotive applications is on the rise. Extruded sections provide opportunities for mass-efficient design of automobile structural and interior components.

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

Depending on manufacturing route and resulted grain/crystallographic matrix, wrought magnesium alloys can exhibit quite different behavior in creep, as 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 show data received in analysis of crystallographic texture and creep-induced residual stress – the 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. Chemical composition of the analyzed samples corresponds to the following alloy designation: AE42, AE33, AX30, AZX310, AJ32, EZ33, and ZE10. 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 a part of continuing effort to develop low-cost wrought magnesium alloys with improved castability and formability suitable for high temperature applications, as well as to add to understanding of material behavior in high temperature creep.

High-temperature Creep Testing

The received wrought material was subsequently subjected to a tensile creep test at 150°C and 175°C and then to compressive creep test at 150°C under a load of 50 MPa for the duration of 200 hrs. All the samples were creep-tested along the extrusion direction. The following charts illustrate some results of these tests.

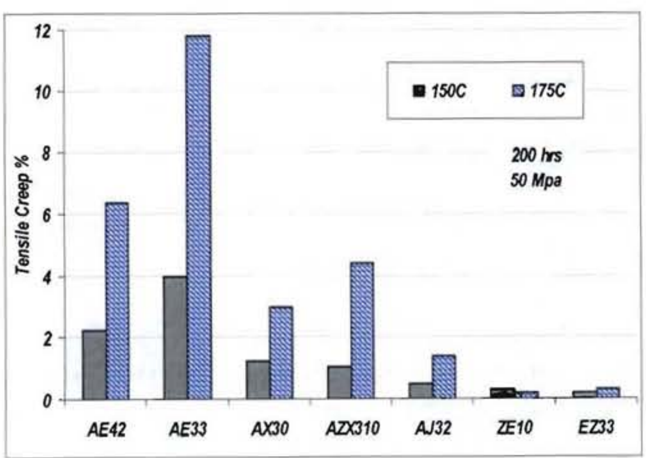


Figure 1
Resistance to creep for the selected alloys at 150 and 175°C

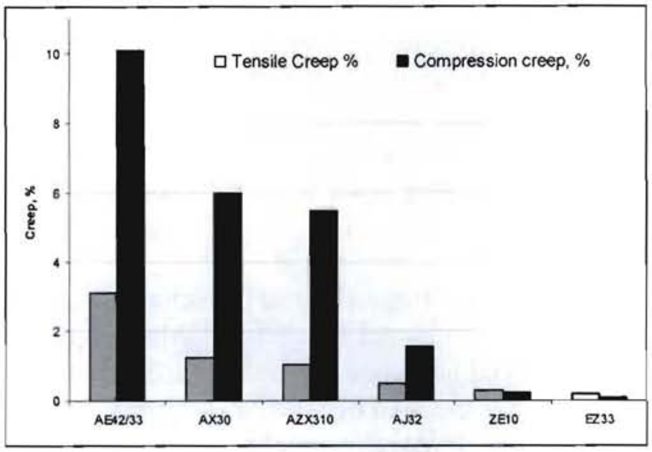


Figure 2
Tension-compression asymmetry in resistance to creep, at 150°C

Data shown in Figure 1 illustrate comparison between results received in the tensile tests at 150°C and 175°C that included both primary and secondary creep. As expected, the 25°C temperature increase lead to significantly reduced resistance to creep, typically by a factor of 3 for all alloys, except ZE10 and EZ33. Evidently, the manufacturing route applied in this study (PMC + hot extrusion) resulted in superior high temperature creep properties for the Mg-Zn-RE alloying system.

This fact was also confirmed in the compressive creep test, performed at 150°C. It is known that typically HCP crystallographic systems exhibit much reduced resistance to creep in compression. This fact can be observed in the following chart, Figure 2, that demonstrates the tension/compression asymmetry in the creep resistance for the studied alloys. Figure 2 indicates that for the identical applied load and duration of the test the resistance to creep was reduced by a factor of 3.5~4 for all of the alloys, but, again, ZE10 and EZ33. It can be concluded from these observations that, compare to the other samples studied in this research, the applied manufacturing route affected favorably the ability of Mg-Zn-RE alloying system to resist the high temperature creep, both in tension and compression. In addition to that, almost no tension-compression asymmetry was observed for the ZE-group samples (in all the tests the resulted creep was within 0.25~0.3% for the ZE10 samples and 0.1~0.2 for EZ33).

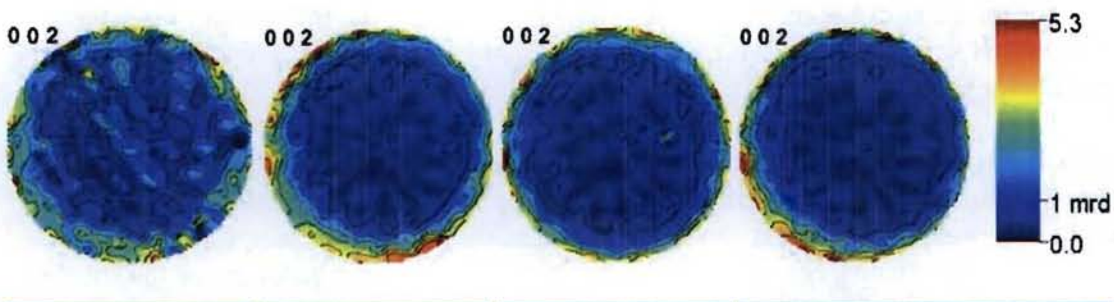
Figures 1 and 2 also show that in all tests the AE-group samples exhibited inferior creep properties, compared to the other three alloying systems. This was partially explained in [11] by presence of β-phase $Mg_{17}Al_{12}$, along the grain boundaries in the magnesium matrix that significantly reduced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

Texture Evolution During Creep Deformation

It was also 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 amount of deformation in creep (up to 10~12% strain), there could be a noticeable texture evolution/modification during the creep testing. These two assumptions were verified in texture analysis performed in 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 in Chalk River, ON, on pure magnesium and Mg-1.5wt.%Mn samples [13, 14]. It was confirmed that the tested material had gone through significant texture modification in creep testing at 150°C and under the load of 50MPa. A typical "rolling" texture could be observed on the crept specimens. Important conclusions were reached with regards to creep deformation mechanisms for the studied materials.

The following pictures (Fig. 3 and 4) show the results of texture calculation using E-WIMF algorithm of the MAUD texture-analysis software [12] based on neutron measurements for several selected alloys.



a. – AE42 b. – AJ32 c. – AX30 d. – EZ33

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

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

The Figure 4, however, shows somewhat different intensities for the prismatic $\{10\bar{1}0\}$ orientation for the initial texture and as it modifies as a result of creep deformation. All the samples in Fig. 4 were ID'ed in the following manner:

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

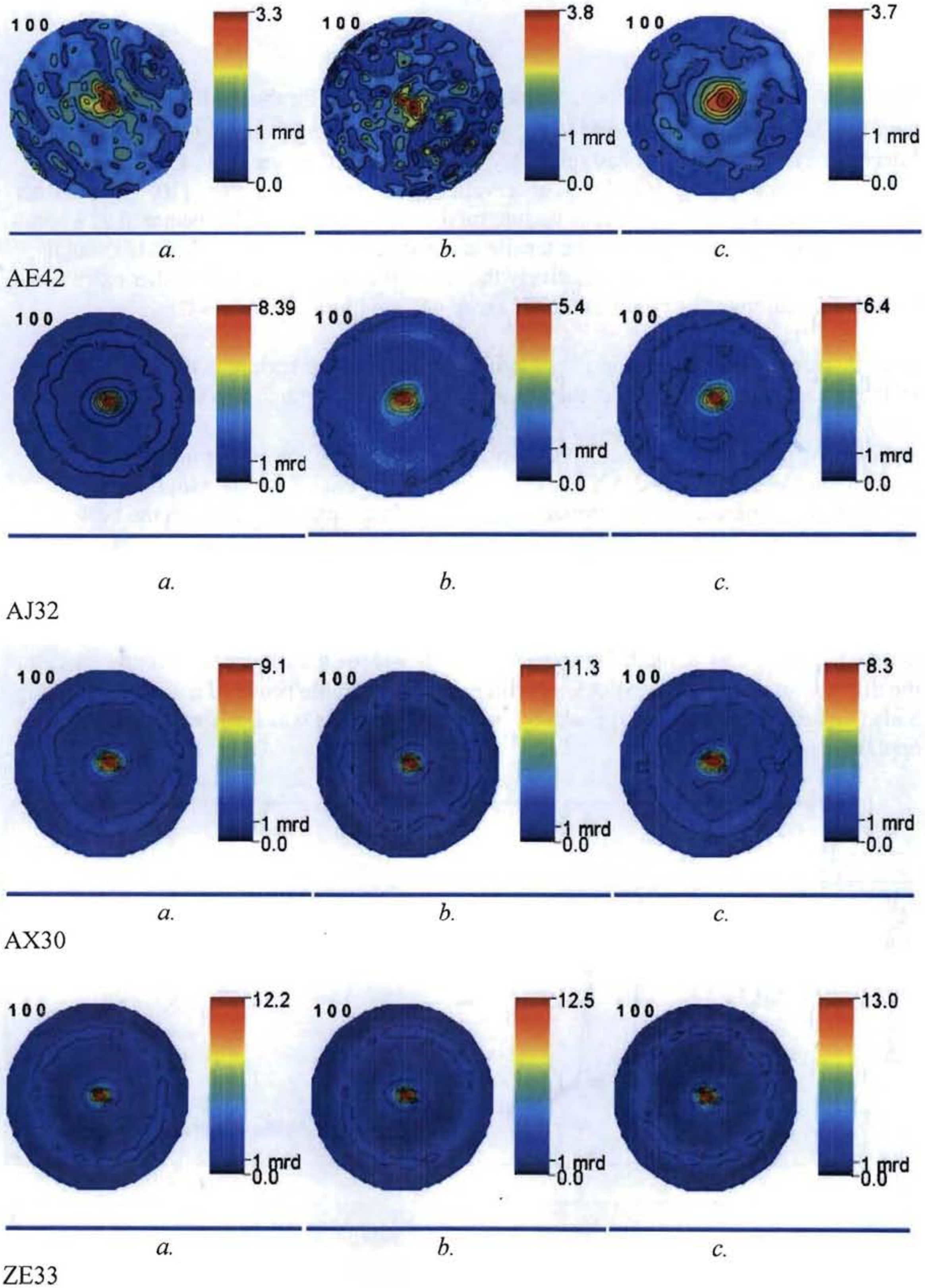

100
3.3
1 mrd
0.0
100
3.8
1 mrd
0.0
100
3.7
1 mrd
0.0
a.
b.
c.
AE42
100
8.39
1 mrd
0.0
100
5.4
1 mrd
0.0
100
6.4
1 mrd
0.0
a.
b.
c.
AJ32
100
9.1
1 mrd
0.0
100
11.3
1 mrd
0.0
100
8.3
1 mrd
0.0
a.
b.
c.
AX30
100
12.2
1 mrd
0.0
100
12.5
1 mrd
0.0
100
13.0
1 mrd
0.0
a.
b.
c.
ZE33

Fig. 4

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

Fig. 4 shows that 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 ~3 to ~12 m.r.d. Interestingly, the alloys that had relatively weak extrusion texture typically did not fare well in the creep testing (Fig. 1), an observation made earlier in studies [10, 11]. Another observation from Fig. 4 is obvious texture modification on the AE42 specimen as a result of creep testing, particularly for the tensile creep specimen (Fig. 4, AE42, *c.*). Though strength of the texture remains relatively the same, the grains received further extrusion-type reorientation as the result of tensile creep deformation.

The other three alloys shown in Fig. 4 exhibited much lesser texture evolution in creep, which is consistent with the fact that these alloys deformed much less than AE42.

Apparently, presence of the $Mg_{17}Al_{12}$ β-phase is only partial reason of inferior performance of AE, AJ, and AX alloys as 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 presence of a second phase in the diffraction pattern (Fig. 5). Along with easily identifiable peaks of α-magnesium, Fig. 5 also depicts several [circled] peaks of another phase. This was further analyzed with application of SEM.

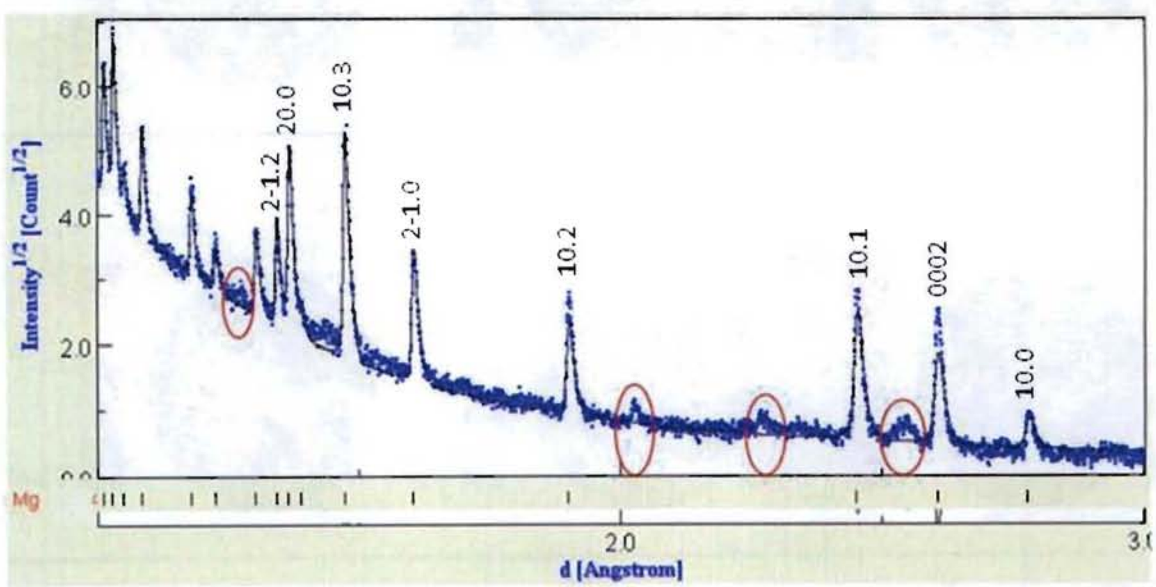
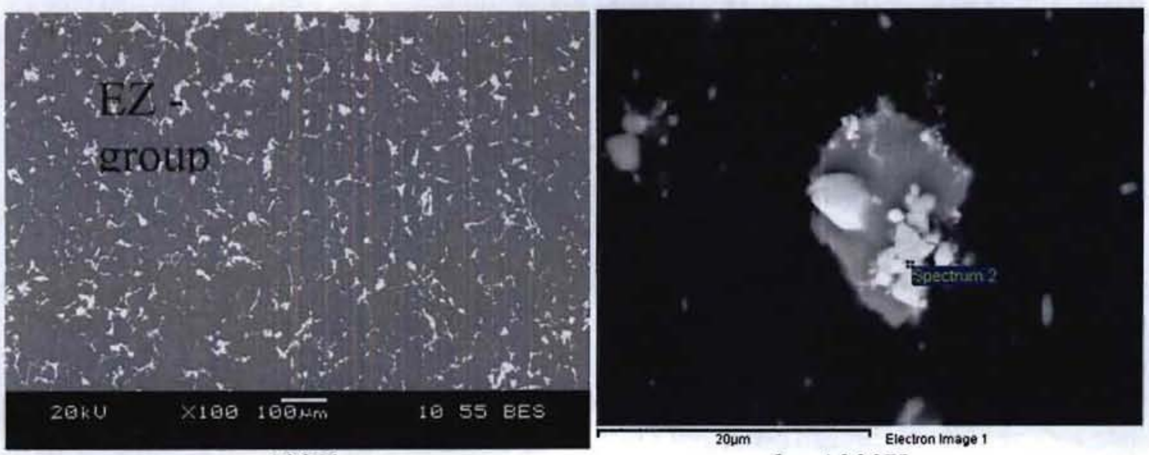


Fig. 5
Neutron Diffraction pattern received for EZ33 specimen

Fig. 6*a* shows that second phase is formed along the grain boundaries. Higher magnification (Fig.6*b*) revealed that it mostly consists of two different intermetallics, with most of the phase volume taken by magnesium-rich (up to ~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 ZrMn2 crystallographic unit cell are different than the ones present in Fig. 5 and are likely hidden in the background "noise", which could be expected considering much limited volume fraction of this phase.

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



a. 100X *b.* 1000X

Fig. 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 performed at the Canadian Neutron Beam Centre and Los Alamos Neutron Science Center confirmed that there is a notable difference in the strength of extrusion-type texture for the extruded samples of studied alloys, that represent four alloying systems; namely, Mg-Al-RE, Mg-Al-Ca, Mg-Al-Sr, and Mg-Zn-RE. Two alloys representing the later 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 in the high-temperature creep testing at the temperatures of 150 and 175°C, both under compressive and tensile load. Opposite to that, 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 in the creep testing.

2. The neutron diffraction spectrum for all the selected alloys, but ZE-group, reveals the typical one-phase (α-magnesium) spectrum. On the other hand, diffraction pattern for the ZE alloys exhibit second-phase peaks arising from presence of complex Mg-Zn-Zr-RE intermetallic. There is also another minority phase in the material, namely $ZrMn_2$, that was forming inside the larger second-phase intermetallic. Due to its limited volume fraction $ZrMn_2$ phase doesn't get detected in the diffraction patters.

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

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