

THE EFFECT OF RARE EARTH ELEMENTS ON THE TEXTURE AND FORMABILITY OF SHEAR ROLLED MAGNESIUM SHEET

D Randman¹, B Davis¹, ML Alderman¹, G Muralidharan², TR Muth², WH Peter², TR Watkins², OB Cavin³

¹Magnesium Elektron North America; 1001 College St.; P.O. Box 258; Madison; Illinois 62060; USA

²Materials Science and Technology Division; Oak Ridge National Laboratory; 1 Bethel Valley Rd; Oak Ridge; Tennessee 37831; USA

³Center for Materials Processing; University of Tennessee, Knoxville; Tennessee 37996; USA

Keywords: Magnesium, Asymmetric Rolling, Texture, Formability

Abstract

The lower relative formability of magnesium alloy sheet can be a restrictive factor when designing light weight engineering structures. Standard symmetric rolling introduces a strong basal texture that decreases the formability; however, asymmetric rolling has been put forward as a possible route to produce sheet with weaker basal texture and greater ductility. It has also been shown in recent work that weaker textures can be produced through the addition of rare earth elements to magnesium alloys. Therefore, this study has been carried out to investigate the effect of rare earth additions on the texture changes during asymmetric rolling. Two alloys have been studied (AZ31B and ZEK100), in which a significant difference is the presence of rare earths in the ZEK100 but not in AZ31B. The differences in texture, microstructure and mechanical properties will be discussed.

Introduction

Magnesium alloys are the lowest density engineering materials that are commonly available and have received significant interest from the automotive and aerospace industries in recent times. Aluminum alloys are often used for sheet forming applications where a light weight solution is required, such as car door panels or aircraft seat backs. Although magnesium is approximately 2/3 the density of aluminum, a barrier to the use of magnesium in these types of applications is the reduced formability at lower temperatures. In general magnesium sheet only attains acceptable levels of formability at temperatures above 200°C due to the activation of additional slip planes to accommodate deformation. This creates problems in industry because these temperatures require the use of solid lubricants, such as boron nitride, that must be removed after forming. This removal process is much harder and more energy intensive than the removal of oils that can be used below 200°C. Therefore, if the temperature of formability could be reduced, the use of magnesium sheet for forming applications would become much more attractive.

The lack of formability in magnesium sheet originates from the rolling texture present in the material. During rolling, magnesium is well known to develop a 'basal texture' in which the c-axes of the grains align strongly with the normal direction of rolling. This places the basal planes, the most common slip plane in magnesium, into a poor orientation for further deformation. Another common deformation mechanism that would allow strain accommodation during forming is the twinning system. In magnesium, this acts as a c-axis extension twin and thus is not likely to be active during forming of the textured material, when thickness reduction is required. Therefore, grains are in a poor orientation for both slip and twinning, which leads to a reduced ductility. By either weakening the basal texture or by tilting it

away from the normal direction it should be possible to produce magnesium sheet with greater formability.

Several methods have been put forward that change the basal texture of magnesium alloys and thus improve the formability: examples are Equal Channel Angular Processing (ECAP)[1, 2], Equal Channel Angular Rolling (ECAR)[3, 4], 'cross-roll rolling'[5], and asymmetric or shear rolling[6-8]. An important target for the magnesium industry is to improve the formability of magnesium sheet. Not all of these methods are suitable for commercial scale sheet production. ECAP is an extrusion process that on its own cannot be used to produce sheet. ECAR combines rolling with ECAP to produce a shear deformation immediately after rolling. This produces suitably textured sheet but requires a radical change to standard rolling practices and thus is unlikely to be easily scaled up to commercial production. Cross-roll rolling was described by Chino *et al.*[5] as a rolling process whereby the rolls are twisted horizontally from each other. Some improvements in texture and formability were achieved, but this method could not be used to produce useful sheet as the crossed rolls would produce a thickness variation across the width of the sheet. Asymmetric rolling uses work rolls travelling at different speeds to introduce shear into the material during the rolling process. This has been found to reduce the strength of the basal texture in magnesium and to cause a tilt towards the rolling direction. Although this reduction in texture is less than by using some of the other techniques, and despite a small curvature introduced in the sheet, shear rolling is thought to be the only process suitable for scaling up to larger commercial production of rolled sheet.

The formability and deformation behavior of the final sheet is strongly dependent on the microstructure and texture that are developed during the shear rolling process. These vary according to the parameters used during rolling, i.e. temperature, rolling speed and reduction per pass (strain rate). Temperature is one of the most important conditions involved as the deformation mechanisms are strongly affected by this. It is well known that twinning is more prevalent at lower temperatures, while a greater number of slip systems are active at higher temperatures in magnesium alloys[9]. However, it is as yet unknown how these different mechanisms will respond to the different stress states experienced in shear rolling compared to standard symmetric rolling. Temperature also affects the recrystallization behavior of the material, both in dynamic recrystallization during rolling and static recrystallization during both interpass pre-heat cycles and post deformation annealing. Dynamic recovery and recrystallization have been observed in various studies[7, 10-12], often causing grain refinement. It has been seen that asymmetric rolling undergoes higher levels of dynamic recrystallization than symmetric rolling under the same conditions[11], which would be expected due to the higher strains experienced. Recrystallization

can also be expected to affect the texture and has previously been observed to decrease the intensity of the basal texture in shear rolled sheet[7, 13, 14].

Different roll speed ratios have been used in the literature, ranging from 1.1 to 3[10, 13, 15]. Effects on the mechanical properties of the sheet have been seen at all ratios, although stronger changes were generally observed at higher roll speed ratios. Despite the general increase in the effectiveness of shear rolling as the roll speed ratio increases, it is thought that an upper limit should be reached due to slipping of the material. This limit will be a function of the friction between the material and the work rolls. Lower than the upper limit for friction, it is thought that there will be a ratio at which the applied shear stress is adequate to produce the necessary changes in microstructure and which it is not necessary to exceed.

The reduction applied per pass affects the final microstructure and properties of the material. This is the result of the amount of strain introduced and retained work in the material, as well as the distribution of the deformation through the thickness. Xia *et al.*[13] stated that flow localization is more likely at larger reductions and that a more homogeneous microstructure, with more dynamic recrystallization, is achieved using smaller reductions in a multi-pass schedule.

Shear rolling has been carried out in the current work using a mill with a speed ratio of 3:1 on both AZ31B and ZEK100 alloys and the differences in behavior have been studied. One set of rolling conditions has been studied in detail to provide an initial analysis of these differences; a commercially viable process route was used, with symmetric rolling down to a low gauge, followed by multiple shear rolling passes to the final gauge.

Scope of the Project

This project has been set up by Oak Ridge National Laboratory in collaboration with Magnesium Elektron North America. The overall aim of the project is to develop more formable magnesium sheet for use in automotive applications, in order to increase the light weighting of vehicles and save fuel usage. The project has been split into 5 different tasks in order to achieve this final goal.

Task 1: Design, fabricate and install shear rolling mill

An important part of this project is the installation of a specially designed shear rolling mill, to advance the understanding of asymmetric rolling of magnesium sheet on a pilot scale, above that possible on laboratory scale mills. The shear mill will be capable of rolling strips up to 250mm wide with very accurate control of temperature, via heated work rolls and roller tables, and shear rolling ratios, with independently driven work rolls.

Task 2: Alloy Selection

Four different alloys were initially studied to develop an understanding of their different behaviors under shear rolling conditions. These alloys were AZ31B (Mg-3wt%Al-1wt%Zn), K1A (Mg-<1wt%Zr), ZK10 (Mg-1wt%Zn-<1wt%Zr) and ZEK100 (Mg-1wt%Zn-<1wt%Rare Earths-<1wt%Zr). It was found in preliminary trials that K1A and ZK10 did not perform as well when processed under a fixed set of parameters. These sheets were more prone to cracking than AZ31B and ZEK100. As such

the breadth of the study was limited to future processing of AZ31B and ZEK100. Results for the K1A and ZK10 will not be reported here.

Task 3: Develop Shear Rolling Process

Task 3 has been set out to find ideal processing parameters for AZ31B and ZEK100 alloys in order to gain the greatest benefit from shear rolling and impart the highest formability. The vast majority of this work will be carried out on the dedicated shear mill once it is installed, but preliminary results from initial trials are presented here. The research focuses on the microstructural and textural changes from temperature, reduction per pass and roll speed ratio.

Tasks 4 and 5: Demonstrate shear rolling in an industrial process environment and fabricate components

Once optimum processing conditions have been established for the two alloys in question the concept will be scaled up and implemented on an industrial size mill at Magnesium Elektron. The proof of concept will be carried out to form industrially relevant parts for the automotive industry through OEM partnerships.

Experimental Materials and Procedure

Magnesium Elektron supplied the material for these investigations in the form of commercially rolled sheet. The AZ31B and ZEK100 were both direct chill (DC) cast as slabs that were then rolled down to the final gauges of 2.36mm and 2.18mm respectively. This sheet formed the rolling feed stock for the shear rolling program.

The laboratory scale shear mill employed during these experiments had a bottom roll with a diameter of 229mm and a top roll diameter of 76mm, giving a ratio of size and circumferential speed of 3:1. To counteract the curling of the sheet as it exited the mill, a stripper plate arrangement was fitted to the mill, forming a channel through which the curled sheet travelled to remove some of the curve. Two inch wide strips were rolled at different temperatures and reductions per pass.

Table 1 – Table showing the temperature, roll ratio and thickness of the sheet with each pass. AZ31B and ZEK100 underwent 7 and 8 passes respectively.

Pass	Temp.	Roll Ratio	Thickness after pass (mm)	
			AZ31B	ZEK100
1	225°C	3:1	2.29	2.01
2	225°C	3:1	2.13	1.85
3	225°C	3:1	2.03	1.75
4	225°C	3:1	1.96	1.63
5	225°C	3:1	1.70	1.50
6	225°C	3:1	1.55	1.37
7	225°C	3:1	1.47	1.27
8	225°C	3:1		1.19

Samples for optical microscopy were cut from the rolled sheet and mounted and polished using standard metallographic techniques. An acetic-pical etch was used to reveal the grain structure in conjunction with an Olympus GX51 microscope equipped with

polarized light optics. The mean linear intercept method was used for grain size analysis.

Samples for X-ray diffraction (XRD) analysis were cut from the rolled sheet and polished down to the centreline for texture analysis. A PTS Tube unit was used with a cobalt x-ray source, and pole figures were measured for the (0002) peaks.

Longitudinal samples for tensile testing were removed from the rolled strips. Two tensile bars were kept as-rolled and two were annealed at 249°C for 2 hours. The microstructures were studied optically and the mechanical properties were compared. Tensile testing was carried out to ASTM E8 specifications.

Results

Microstructures

Figure 1 and Figure 2 show the starting material prior to shear rolling. The microstructures were fully recrystallized and homogeneous through the full thickness. It can also be seen that the grain size differs between the two materials; ZEK100 had a grain size that was approximately twice that observed in the AZ31B sheet, see Table 2.

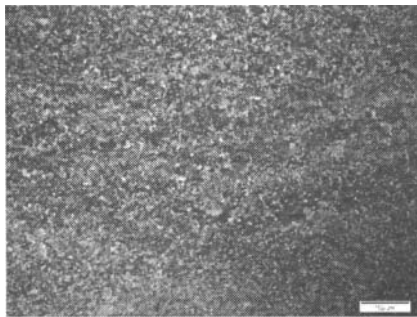


Figure 1 – As received AZ31B feed stock material that was symmetrically rolled to the starting gauge of 2.36mm for asymmetric rolling.



Figure 2 – As received ZEK100 feed stock material that was symmetrically rolled to the starting gauge of 2.18mm for asymmetric rolling.

The shear rolling conditions chosen for the current work were multiple passes at 225°C to a large total strain, as shown in Table 1. Micrographs of the material following rolling are shown in Figure 3 and Figure 4.

Table 2 shows the grain sizes measured in the starting and post-shear rolling conditions of both alloys. It can be seen that there was little change in the grain size of the AZ31B, although this was hard to measure in the deformed specimen as there was a high density of twins and slip bands present. It can be seen in Figure 3 that although there were a lot of other features present, the grain boundaries still exhibited a roughly equiaxed shape. In Figure 4, the ZEK100 sample showed a small amount of grain refinement with shear rolling, although it can be seen that a large number of the grains stayed the same size as in the starting material and showed high levels of deformation, particularly in the form of twins. The recrystallized grains were observed primarily at the grain boundaries of the deformed grains. Some of the recrystallized grains appeared to be undeformed, without evidence of a deformation structure.

Table 2 – Grain sizes of the starting and rolled materials

		Ave. Grain Size (µm)
AZ31B	Starting	4.09±0.17
	After shear rolling	3.98±0.25
ZEK100	Starting	8.59±0.33
	After shear rolling	7.16±0.39

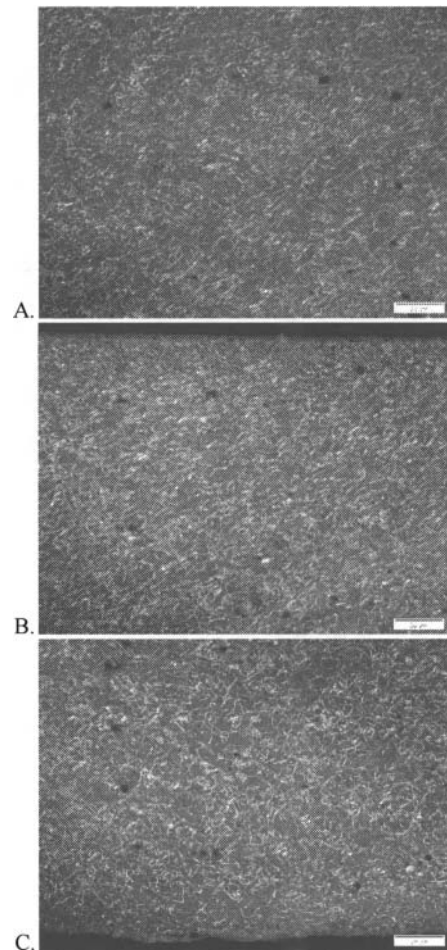


Figure 3 – Micrograph showing the grain structure at the A. centre line, B. slow roll surface and C. fast roll surface of AZ31B sheet after 7 passes of asymmetric rolling to a total strain of 48%. In A.,

the slow roll surface is towards the top of the micrograph and the fast roll surface is towards the bottom. The rolling feed direction is left to right.

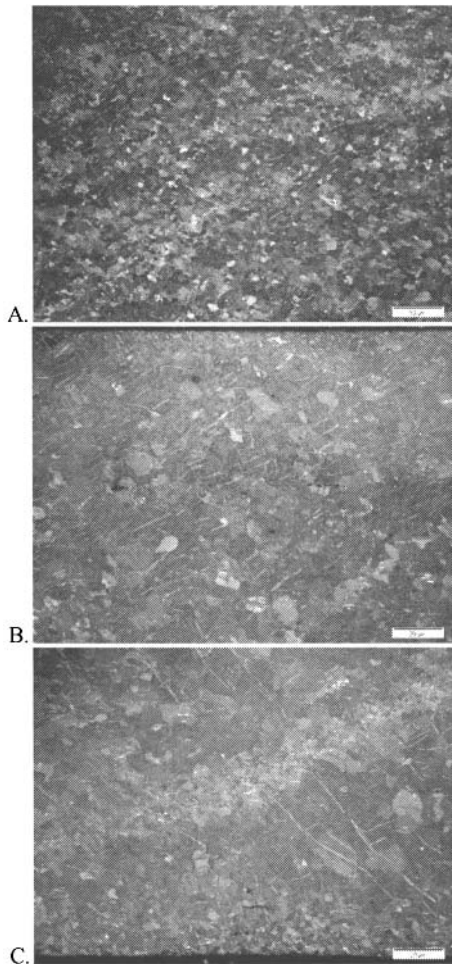


Figure 4 – Micrograph showing the grain structure at A. the centre line, B. slow roll surface, and C. fast roll surface of ZEK100 sheet after 8 passes of asymmetric rolling to a total strain of 65%. In A., the slow roll surface is towards the top of the micrograph and the fast roll is towards the bottom. The rolling feed direction is left to right.

Whilst the bulk of the samples showed homogeneous microstructures, there were significant differences in a thin layer at each surface, generally less than 20µm thick. In the AZ31B sample, finer grains were observed at the slow roll surface, while little change was seen at the fast roll surface. In the ZEK100 sample, a band of fine grains was observed at both surfaces with smaller grains seen at the slow roll surface. It is thought that these grains were formed by recrystallization of an intensely deformed region. It is thought that the bottom surface of the material did not move three times faster than the top surface. The fast work roll was assumed to be moving faster than the material and equally the slow roll slower than the material. This caused slippage during shearing that created a ‘smearing effect’ at the surfaces of the material with a very high level of strain and induced extensive recrystallization in those regions.

Microstructure after annealing

Following an annealing treatment at 249°C for 2 hours the microstructure was again studied, see Figure 5 and Figure 6. The grain sizes of the annealed material are given in Table 3. It can be seen that the post-annealed AZ31B grain size was only slightly larger than the as shear rolled metal, although the number of twin boundaries and slip bands within the grains appeared to have decreased. The grain size increased significantly in the ZEK100 material, by approximately 50%, and it can be seen from Figure 6 that this was mostly achieved by the large growth of a small number of grains. It is thought that this was grain growth of dynamically recrystallized grains present in the microstructure just after rolling. The remaining grains still show high levels of cold work in the form of twin boundaries and slip bands. This microstructure would not be expected to give good ductility.

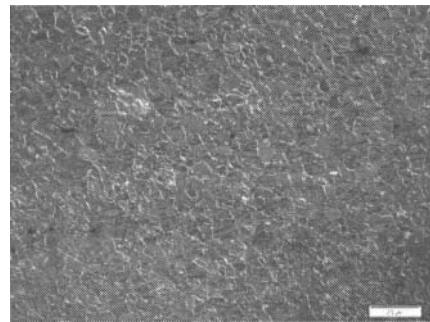


Figure 5 – Micrograph showing the grain structure at the centre line of AZ31B sheet after 7 passes of asymmetric rolling, to a total strain of 48%, then annealed at 249°C for 2 hours.

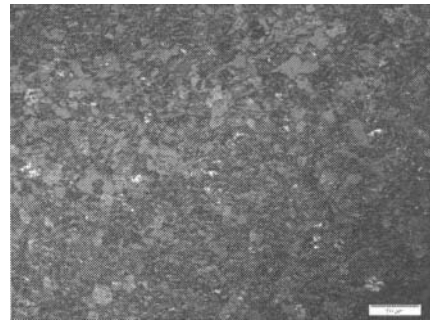


Figure 6 – Micrograph showing the grain structure at the centre line of ZEK100 material after 8 passes of asymmetric rolling, to a total strain of 65%, then annealed at 249°C for 2 hours.

Table 3 – Grain sizes of the samples following annealing

		Av. Grain Size (µm)
AZ31B	After Anneal	4.34±0.29
ZEK100	After Anneal	10.78±0.72

Texture

The bulk textures on the centerlines of the shear rolled samples were measured with XRD and the {0002} pole figures are presented in Figure 7. These pole figures show that the basal texture is tilted away from the normal direction and towards the rolling direction in each sample.

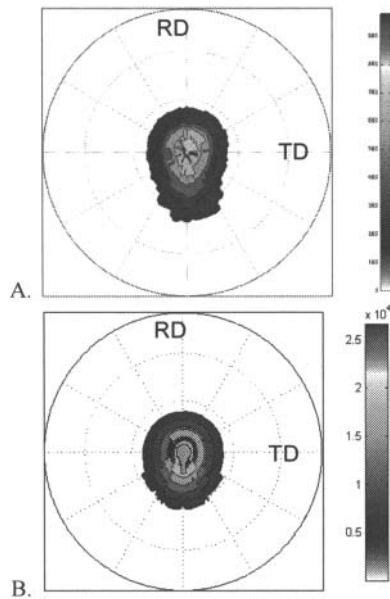


Figure 7 – {0002} pole figure measured at the centerline of A. AZ31B sheet after 7 passes of asymmetric rolling, to a total strain of 48% and B. ZEK100 sheet after 8 passes of asymmetric rolling, to a total strain of 65%.

Mechanical Properties

Table 4 – Mechanical properties of the symmetrically rolled and asymmetrically rolled sheets in the longitudinal direction

Alloy	Processing	Yield (MPa)	UTS (MPa)	Elongation (%)
AZ31B	Symmetric	210	288	10.4
	Symmetric plus anneal	174	265	17.5
	Asymmetric	236	290	6.0
	Asymmetric plus anneal	148	256	8.0
ZEK100	Symmetric plus anneal	212	263	10.4
	Asymmetric	205	249	7.0
	Asymmetric plus anneal	137	232	11.0

While the tensile ductility studied here does not give a direct link to formability, the indication to final forming properties is still useful. It can be seen that shear rolling under the conditions used here degraded the ductility of AZ31B when compared to symmetrically rolled sheet in both the as-rolled and annealed conditions. However, the ductility of the ZEK100 was improved slightly by shear rolling plus annealing.

Discussion

Figure 1 and Figure 2 show that the starting grain sizes were different in the two different materials. It is thought that this is due to the variation in process route as the AZ31B was cast into a much larger slab and therefore underwent a greater reduction to reach this gauge.

Shear rolling of AZ31B did not cause a significant change in grain size or morphology. The grains were approximately equiaxed after a total reduction of 48%, see Figure 3. Under conventional rolling conditions it would be expected that at this level of rolling reduction the grains would become elongated. The presence of equiaxed grains is a strong indication of recrystallization. It can also be seen that the majority of grains contained some evidence of work in the form of twins and/or slip bands. This indicates that deformation continued after the recrystallization event, suggesting that recrystallization was not dynamic but static during the interpass holding times.

Figure 4 showed that there were some smaller grains formed through recrystallization in the ZEK100 during shear rolling. Many of these do not show any further signs of work so it can be assumed that they formed by dynamic recrystallization near to the end of deformation. This change in recrystallization mechanism is a significant difference between the two alloys and could be expected to affect both the shear rolling behavior and the post-rolling properties.

The recovery and recrystallization behavior during post-deformation annealing was also strongly affected by the alloying elements. AZ31B underwent some recovery and recrystallization but little grain growth, whereas ZEK100 underwent significant grain growth but little further recrystallization. The recrystallization seen in AZ31B was not complete as there was still evidence of deformation within many of the grains and thus a longer heat treatment is required to maximize the ductility. This is thought to be the reason the tensile ductility shown in Table 4 was lower for the shear rolled and annealed AZ31B relative to the symmetrically produced and annealed material. It is also thought that the shear rolling process can introduce more flow localization than symmetric rolling and consequently is more likely to induce cracking in those regions, reducing ductility. There are various ways in which the flow localization can be reduced, including lower speeds, higher temperatures, or lower reductions on each pass, and these factors will be looked at in future investigations to improve the shear rolling response.

ZEK100 showed a very small improvement in ductility from symmetrically rolled material, although this could be due simply to experimental variation. The annealed sample of ZEK100 showed grain growth, thought to be primarily from the dynamically recrystallized grains.

Despite this lack of improvement in ductility, it should be remembered that formability is only indirectly related to tensile ductility. Due to the nature of tensile testing, the extension in one direction can be accommodated by contraction in either of the two remaining directions, whichever is easiest. For rolled magnesium tested in the longitudinal direction, the contraction is easiest in the transverse direction, and vice versa. However, in biaxial forming, such as deepdrawing, there is extension in both the longitudinal and transverse directions and thus the contraction occurs in the thickness direction. This is not tested with tensile tests and thus can be very different; true formability testing requires biaxial deformation and this will be analyzed in later studies by the current authors.

The pole figures in Figure 7 showed that the texture had been tilted away from pure basal by shear rolling in both alloys. It can be seen that the main intensity peak was tilted further in ZEK100

than AZ31B, which indicates that shear rolling had a greater effect in ZEK100. Weaker textures have been seen previously in the rolling of rare earth containing magnesium alloys[16] and it is thought that the rare earth content of ZEK100 has allowed a greater tilting of the texture during shear rolling.

The current work has shown that ZEK100, containing rare earths, gives a greater ductility after shear rolling than AZ31B. This is associated with the modification of the texture, as well as the amount of recrystallization that occurred, as discussed above.

Summary

- AZ31B and ZEK100 magnesium sheets have been produced by shear rolling at 225°C.
- Following shear rolling, the microstructures show high levels of cold work in the form of twins and slip bands within grains.
- The texture of the material is altered during shear rolling to give a tilt of the basal pole towards the rolling direction.
- Following annealing, shear rolled AZ31B underwent recrystallization to reduce the deformation inherent within the grains.
- Shear rolled ZEK100 underwent grain growth during annealing.

Acknowledgements

The materials processing research was carried out in collaboration with Oak Ridge National Laboratory under sub contract 4000092046 as part of the American Recovery and Reinvestment Act through the U. S. Department of Energy, Office of Energy Efficiency and Renewable Energy, Industrial Technologies Program. The materials characterization research through the Oak Ridge National Laboratory's High Temperature Materials Laboratory User Program was sponsored by the U. S. Department of Energy, Office of Energy Efficiency and Renewable Energy, Vehicle Technologies Program. Thanks goes to those staff members at Oak Ridge National Laboratory that contributed to the research program.

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