Mg Sheet: The Effect of Process Parameters and Alloy Composition on Texture and Mechanical Properties

Kerstin Hantzsche, Joachim Wendt, Karl Ulrich Kainer, Jan Bohlen, and Dietmar Letzig

The paper addresses the relationship between microstructure, texture, and mechanical properties of rolled magnesium sheets. The effect of rolling temperature and alloying elements on texture development and mechanical properties is demonstrated. Special focus is paid to the potential of rare earth elements to modify the anisotropic behavior and to weaken the strong basal texture of magnesium sheets. Alloy design that considers these possibilities together with appropriate selection of process parameters show the road to magnesium sheets with improved forming properties.

How would you…

…describe the overall significance of this paper?

The paper contributes to a fundamental understanding of the recrystallization and deformation behavior of wrought magnesium alloys aiming to develop magnesium sheets with improved formability at low temperatures. It could be shown that the design of magnesium sheets with better forming properties is possible by controlling the crystallographic texture.

…describe this work to a materials science and engineering professional with no experience in your technical specialty?

The paper addresses the relationship between microstructure, texture, and mechanical properties of rolled magnesium sheets. The effect of rolling temperature and alloying elements on texture development and mechanical properties is demonstrated. Special focus is placed on the potential of rare earth elements to modify the anisotropic behavior and to weaken the typical strong basal texture of magnesium sheets. Alloy design that considers these possibilities together with appropriate process parameters show the road to magnesium sheets with improved forming properties.

…describe this work to a layperson?

This work deals with magnesium sheets and forming behavior. Usually, it is difficult to form magnesium sheets especially at low temperatures. This is caused by the metal structure itself and a so-called texture, which develops during the production process (i.e., the rolling process). By adding special metals in a low amount to magnesium it is possible to significantly alter and weaken those textures which promises improved forming behavior of the final sheets. The present work provides basic knowledge about the influence of selected metals on the magnesium sheet properties. The objective is to be able to design magnesium sheets with required properties.

INTRODUCTION

The use of sheet material as a semi-finished product enables a wide scope for design. The development of new materials such as magnesium alloys for lightweight application requires the development of semi-finished products for an expansion of possible applications. Magnesium sheet offers a significant possibility of producing components in a large variety of shapes from its simple flat structure. However, despite their great potential wrought magnesium alloys have hitherto only played a small role in lightweight structural applications. A major technical reason is the fact that commercial magnesium sheets like AZ31 are limited in their ductility and formability, especially at room temperature. This is related to the occurrence of magnesium with a hexagonal-close-packed lattice structure. In consequence, there are a small number of deformation mechanisms that can be activated during plastic deformation at room temperature compared to metals with cubic lattice structures. Basal slip and so-called tensile twinning are the most important ones that are easily activated. The limitation in ductility is further enhanced by the occurrence of strong textures after rolling, which also lead to a distinct anisotropy in yield behavior.[1] In such textures the majority of grains are oriented such that their basal planes are parallel to the sheet plane. In this orientation, the grains are hard to deform.

To overcome these limitations, it is necessary to develop sheets with optimized microstructures and designed textures. Thus, the formability may be improved by appropriate alloy modification. In this regard magnesium alloys containing rare earth (RE) elements such as cerium and yttrium have received attention in a number of research and development programs (e.g., References 2–8). However, a general influence of alloy composition on optimum process parameters can be anticipated because the rolling textures of magnesium sheets result from deformation as well as recrystallization occurring during the rolling procedure itself. Thus, any alloy modification must be considered in the context of process parameters like degree of deformation, rolling temperature, and heat treatment. In this paper different magnesium alloys like ZM21 as a conventional alloy and ZEK100—containing cerium-mischmetal—are compared regarding their recrystallization characteristics and the corresponding textures in dependence on process temperatures. Furthermore, such textures are discussed in correlation to their mechanical properties (see, e.g., Reference 9). They especially include the yield anisotropy which is quantified by testing the material in various orientations as well as the planar anisotropy or r-value. The r-

Table I. Chemical Compositions of Alloys ZM21 and ZEK100

Content (wt.%)	Mg	Zn	Mn	RE	Zr
ZM21	97.0	2.1	0.9	—	—
ZEK100	97.6	1.4	—	0.34	0.3

value describes the tendency of a sheet to flow more from its thickness or its width if stressed in the longitudinal direction. This parameter is an important characteristic for the general flow behavior of the material in sheet metal forming processes. Moreover, the orientation dependence of this value, the in-plane anisotropy, determines its behavior during anisotropic material flow (i.e., local thinning or earing).[10] In this regard tests were carried out to determine the influence of various parameter settings and the addition of RE elements on the development of microstructure, texture, and the resulting mechanical properties.

ROLLING AND SHEET PREPARATION

The slab materials used in this study were continuously cast ZM21 (2.1 wt.% zinc, 0.8 wt.% manganese) and ZEK100 (1 wt.% zinc, 0.5 wt.% Cer-mischmetal, 0.5 wt.% zirconium) alloys. The chemical compositions are provided in Table I.

Rolling slabs with an initial thickness of 7.4 mm were prepared and homogenized at a temperature of 350°C for 15 h prior to rolling. The rolling experiments were performed with a speed of approximately 16 m/min. and without use of lubrication. A constant degree of deformation of about $\varphi = 0.3$ per pass is applied, where:

$$\varphi = -\ln(h_{n+1} / h_h) \qquad (1)$$

n is the consecutive number of the pass and h_n the sample thickness after pass n. Two different rolling temperatures, 300°C and 450°C, were applied. After each pass the sheet was re-heated for 15 min. to keep the rolling temperature constant. After the complete rolling procedure the sheet received a final heat treatment at the respective rolling temperature for 20 min. The final gauge of all sheets was ca. 1.35 mm. During rolling of ZEK100 at 300°C small cracks started to be generated throughout processing which then propagated with each following pass. The other sheets, however, were crack-free. On the surfaces of all sheets some flakes were present due to the tendency of magnesium to adhere on the rolls since no lubrication was used in the experiments. See the sidebar for experimental procedures.

CORRELATIONS BETWEEN MICROSTRUCTURE AND TEXTURE

Sample micrographs from the longitudinal sections of the sheets are shown in Figure 1 after rolling at the two different temperatures, 300°C and 450°C. In Figure 1a and b completely recrystallized microstructures are observed for both cases of ZM21. However, the average grain size is 8 µm after rolling at 300°C and 24 µm after rolling at 450°C (i.e. a significant increase of the average grain size with rolling temperature).

Figure 1c and d shows the same for sheet alloy ZEK100. It is easily seen that the material behavior is distinctively different. In the case of the sheet rolled at 300°C a strongly deformed microstructure is found which does not show any tendency to form small and new grains as a result of material recrystallization. In contrast, the higher temperature of 450°C allows a complete recrystallization of the sheet microstructure and reveals an average grain size of 13 µm.

In Figure 2 the recalculated pole figures obtained from the texture measurements are shown. Typically, the basal planes of Mg sheets are orientated parallel to the sheet plane but show different angular distributions from the normal direction to either the rolling or transverse direction. In the case of the ZM21 sheets presented in this work (Figure 2a-b) the angular distribution of basal planes is broader toward the rolling direction (RD) rather than toward the transverse direction (TD). This is considered to be a typical finding for a magnesium sheet texture like that of the broadly used alloy AZ31 (see also Figure 5 for comparison) (e.g., Reference 12). It is noteworthy that the maximum intensity of the basal pole figure decreases with increasing rolling temperature resulting in a weaker texture at higher temperature.

Figure 2c-d shows the same pole figures for the ZEK100-sheets. In the basal (00.2) pole figure of the ZEK100 sheet rolled at 300°C two interesting features are found (Figure 2c). First, a strong component puts the basal planes not exactly parallel to the sheet plane but tilted toward RD and in a degenerate double peak. A broad angular distribution of the basal planes toward the RD is also found. Second, an even broader angular distribution towards TD is found.

Figure 2d shows the texture of the sheets rolled at 450°C. Clearly, the intensity of this texture is the lowest compared to the others discussed in this study. Further, the shape of the basal pole figure has drastically changed. It is best characterized as a degenerate double peak tilted toward

EXPERIMENTAL DETAILS

The microstructure of the sheets was analyzed using light optical microscopy. Standard metallographic sample preparation techniques were applied employing an etchant based upon picric acid,[11] which revealed grains as well as grain boundaries. The average grain size was determined using a computer-aided linear intercept measurement.

For texture measurements an x-ray diffractometer with CuK_α radiation was used. Therefore sheet samples were ground to mid-plane and polished. The (10.00), (00.2), (10.1), (10.2), (11.0), and (10.3) pole figures were measured to a sample tilt of 70° and used for the calculation of the complete orientation distribution function (ODF) which allows the recalculation and presentation of complete pole figures.

Tensile tests were performed using a universal testing machine with a constant strain rate of $10^{-3}\,s^{-1}$ at room temperature to investigate the mechanical properties. Specimens were prepared in two sample orientations, the sheet rolling direction (RD) and transverse direction (TD). Extensometers were used to measure the strain along the length and width of each sample. Mechanical properties such as the tensile yield strength (TYS), the ultimate tensile stress (UTS), the uniform elongation (E_u) and the fracture strain (E_f) were determined as well as the r-value at room temperature at 8% strain.

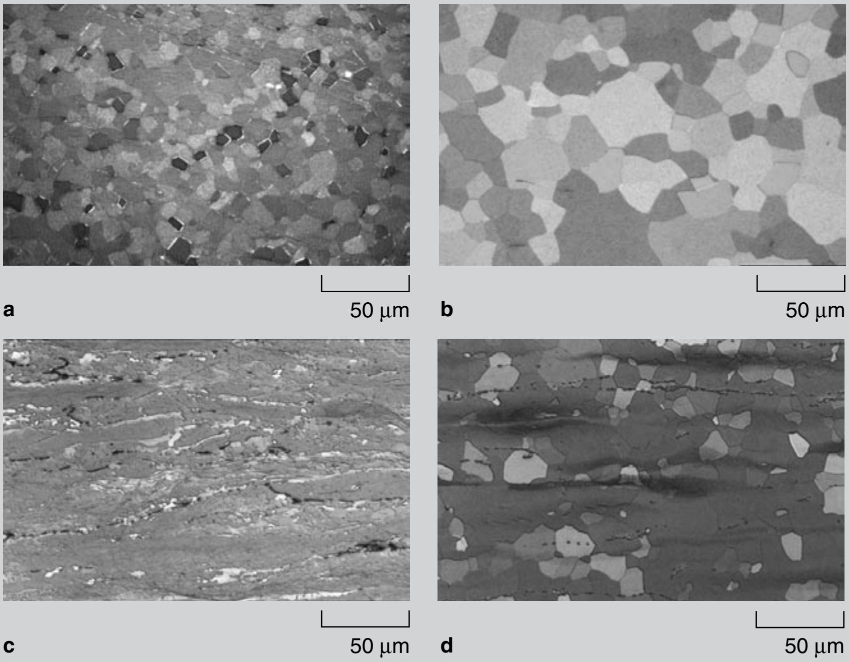

Figure 1. Microstructures of the final sheets after the post-heat treatment. (a) ZM21– 300°C, average grain size 8 μm, fully recrystallized; (b) ZM21–450°C, average grain size 24 μm. fully recrystallized; (c) ZEK100–300°C, severely deformed even after HT; high density of shear bands causing cracks; (d) ZEK100–450°C, average grain size 13 μm, fully recrystallized.

the TD and a significantly broader angular distribution of basal planes toward TD than towards RD.

MECHANICAL PROPERTIES

In Figure 3 stress-strain curves of the sheets are shown for the two sample orientations, RD and TD. Only in the case of the ZEK100 sheet rolled at 300°C no mechanical curves were obtained as a result of significant strain hardening of the sheets which led to initial fracture during testing. For better comparison the characteristic mechanical values—tensile yield strength (TYS), ultimate tensile strength (UTS), uniform elongation (E_u) and elongation at fracture (E_f)—are also plotted in Figure 4.

Figure 3a and b shows mechanical properties for alloy ZM21 rolled at 300°C and 450°C, respectively. In both cases a higher TYS and UTS is obtained along TD than along RD. A pronounced elastic limit is found for the sheet rolled at 300°C (Figure 3a), which is more significant along TD and less pronounced along RD. It almost vanishes for the sheet rolled at 450°C (Figure 3b). In contrast, the ZEK100 sheet reveals an inversion of the yield properties (i.e., the higher TYS is found along RD rather than along TD), see Figure 3c. Further, the yield anisotropy is more distinctive in ZEK100 than in both ZM21 sheets (Figure 4a). A similar behavior is observed regarding the elongation properties (Figure 4b). The r-value is shown in Figure 4c. For both ZM21 sheets a significantly higher r-value is found along TD rather than along RD. For the sheet rolled at higher rolling temperature generally lower r-values are found compared to that rolled at 300°C. Further, the in-plane anisotropy (i.e., the orientation dependence of the r-value) is lower after rolling at 450°C. For the ZEK100, a very comparable r-value of approximately 1 is found for both the RD and TD orientations. Thus, a significant in-plane anisotropy cannot be described for this sheet.

EVALUATING THE TEXTURE OF MAGNESIUM SHEET

Study of the microstructure of the investigated sheets shows a significant

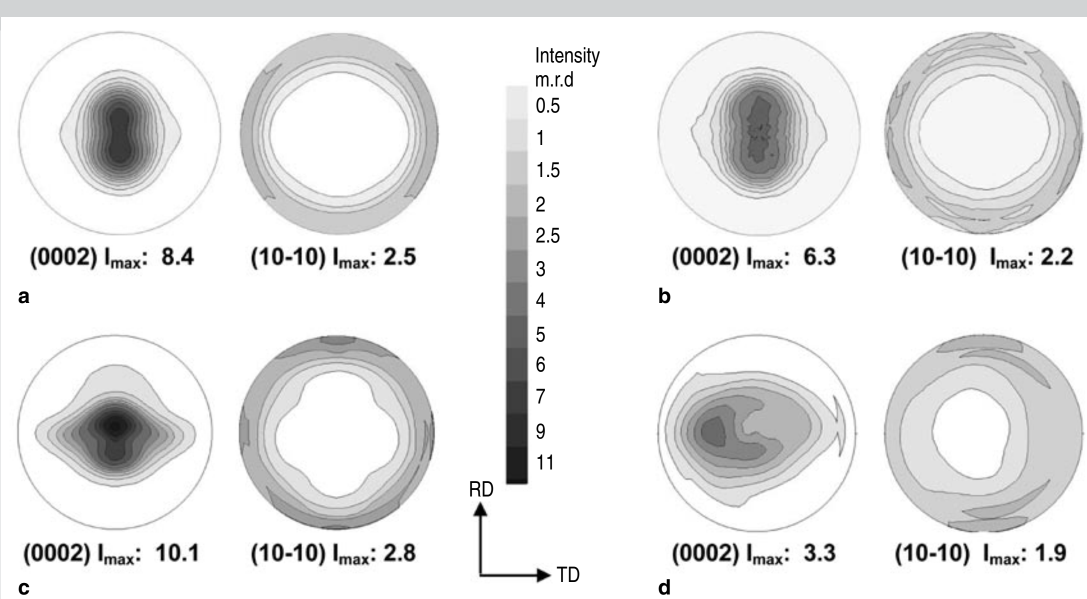

Figure 2. Recalculated pole figures of the basal (0002) and prismatic (10-10) planes of the rolled and heat-treated sheets. Maximum intensities are given in [m.r.d.]. (a) ZM21 sheet rolled at 300°C, (b) ZM21 sheet rolled at 450°C, (c) ZEK100 sheet rolled at 300°C, (d) ZEK100 sheet rolled at 450°C.

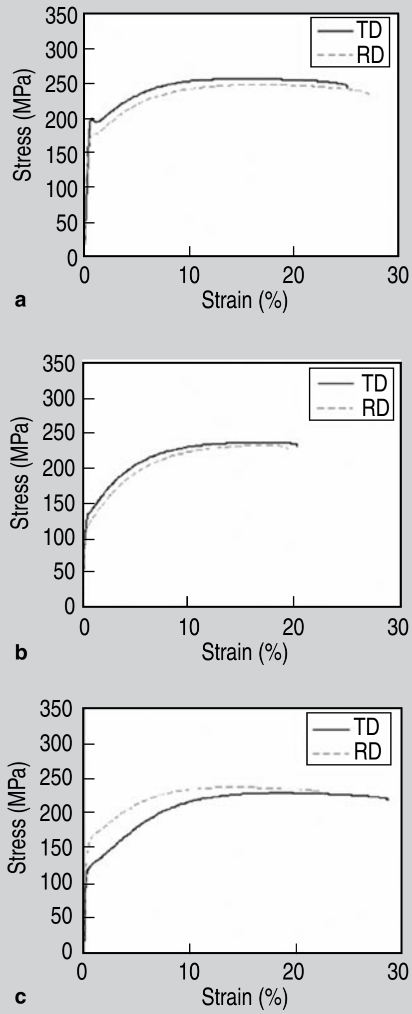

Figure 3. Stress-strain curves of the final sheets: (a) ZM21 rolled at 300°C, (b) ZM21 rolled at 450°C, (c) ZEK100 rolled at 450°.

dependence of the recrystallization on alloy composition. While the ZM21 sheet is fully recrystallized even at the lower rolling temperature the microstructure of the ZEK100 sheet rolled at 300°C remains severely deformed.

Both ZM21 sheets show essentially the basal texture which is typically found for commercial sheet AZ31, as shown in Figure 5. The remarkably strong texture of the AZ31 sheet is connected to a well-recrystallized microstructure with grain boundary bulging as the principal recrystallization mechanism.[13]

It is noteworthy that the texture of the ZM21 rolled at higher temperature is slightly weaker than after rolling at 300°C. However the texture shape does not change. In the case of the ZEK100 rolled at 450°C a well-recrystallized microstructure corresponds to a weak texture. This weak texture is best characterized by the angular distribution of the basal planes being broader from ND to TD rather than from ND to RD. This kind of texture has been described in earlier works, which is connected with alloys containing RE-elements.[6,14] After rolling at 300°C, however, a different texture was developed, which can be correlated with a microstructure that does not show any recrystallization. However, investigations using SEM techniques reveal that small new grains that result from the rolling procedure can be observed as shown in Wendt et al.[5] It can be concluded that the recrystallization that occurs during processing takes place more slowly than in ZM21.

The texture of this sheet rolled at 300°C consists of two components, one being the deformation texture as a result of the rolling process and one being the result of the formation of new grains during recrystallization. Mackenzie and Pekguleryuz[3] have shown that after the rolling of ZEK100 at an even lower temperature of 150°C a strong basal texture results, which basically is similar to the texture observed in the ZM21 sheets in this study. Additionally, the angular spread of basal planes towards the TD may be regarded as being due to the new texture component introduced by recrystallization during the rolling procedure. In this view after rolling and heat treatment at 450°C complete recrystallization of the microstructure occurs and the strong basal texture component vanishes. Instead of that a degenerate double peak at an angle of 35° is developed. The overall texture is remarkably weak. It is not a purpose of this paper to discuss the origin of this component. However, some authors attributed this to the presence of the RE-elements being in solid solution in the alloy.[4–6,14] This hypothesis assumes indeed a different kind of recrystallization mechanism compared to that which is typically active in the commercial alloy AZ31 or the ZM21 in this study.[13,15] This interpretation can be directly connected with the finding that the two different alloys in this study obey different recrystallization kinetics.

Technologically, the alloy ZEK100 requires a higher rolling temperature compared to ZM21 to produce a sheet with a fully recrystallized microstructure. The resulting weak textures are likely to improve the mechanical performance of the sheet. It is found for ZM21 that the strength properties follow the Hall-Petch relation where $\sigma \sim d^{-1/2}$, with d being the average grain size (i.e., higher yield strength results from lower grain size caused by the lower rolling temperature). The strong concentration of basal planes in the sheet plane with a wider angular distribution toward the RD than to the TD in the case of the ZM21 always causes a lower TYS along RD. This can be explained by an easier activation of the basal slip systems in RD. A similar behavior due to its texture is found for the commercial alloy AZ31 where the TYS along TD with almost

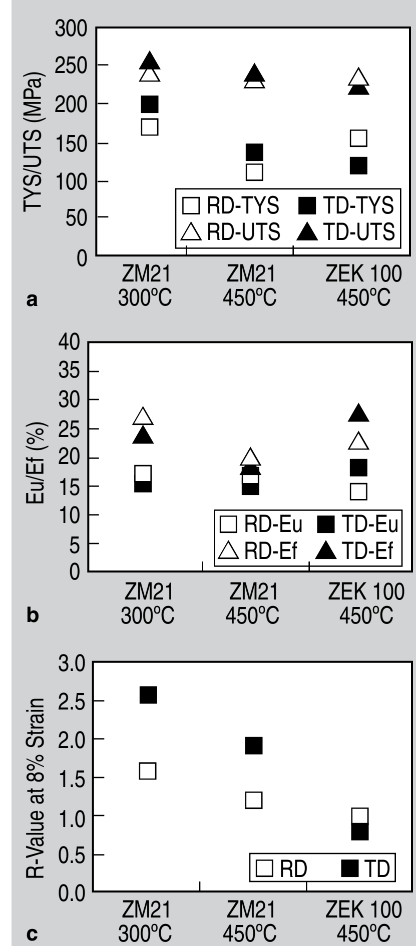

Figure 4. Comparison of the characteristic mechanical values of the sheets: (a) Tensile yield strength (TYS) and ultimate tensile strength (UTS); (b) uniform elongation (E_u) and elongation at fracture (E_f); (c) Lankford-coefficient (R-value) at 8% strain.

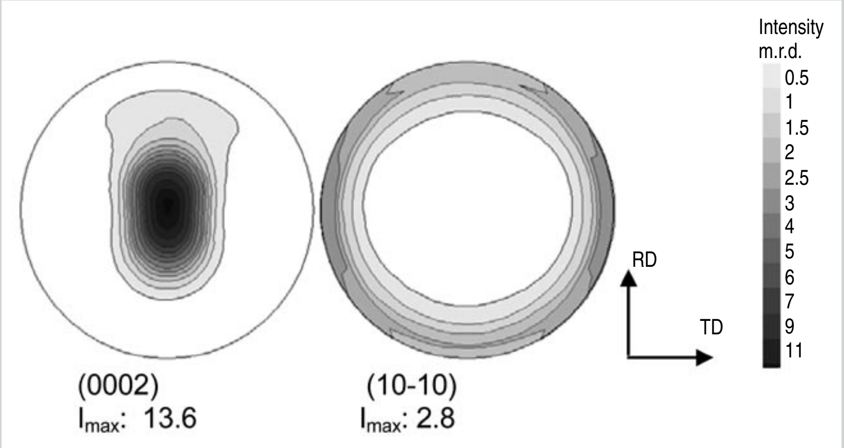

Figure 5. Pole figures of the basal (0002) and prismatic (10-10) planes of commercial AZ31 sheet. Maximum intensities are given in [m.r.d.]. (recrystallized microstructure).

190 MPa is higher than the TYS of 160 MPa measured along RD. In contrast, the ZEK100 sheet shows an inverse yield behavior. Due to the distribution of the planes the TYS is significantly lower along TD rather than along RD. The lower texture intensity of the ZM21 after rolling at 450°C compared to rolling at 300°C leads to a decrease in the r-value independent of the sheet orientation. Also the in-plane anisotropy is decreased in that case. The ZEK100 sheet after rolling at 450°C exhibits minimized in-plane anisotropy due to its extremely weak texture. Regarding the AZ31 sheet the strong basal texture causes high r-values of 1.9 at 8% strain along RD and of 2.9 along TD expressing the typical anisotropic behavior. Hence, both ZM21 sheets possess an inhomogeneous forming behavior similar to the AZ31 sheet which is characteristic for magnesium alloys, whereas the ZEK100 rolled at an appropriate temperature promises improved formability as a result of the texture formation.

CONCLUSIONS

Due to the different recrystallization kinetics in ZEK100 compared to conventional alloys such as AZ31 or ZM21 it becomes necessary to adjust the process parameters accordingly. One of the theories currently receiving attention and supported by the present results connects nucleation and recrystallization with solute drag of the RE on the grain boundaries.[4] Another possible effect may, however, be to prevent grain boundary bulging as the common recrystallization mechanism in conventional magnesium alloys.[13] Nevertheless, the fact remains that the fully recrystallized microstructure of the ZEK100 rolled at higher temperature causes a significantly different and weaker texture.

Thus, the decreased planar and in-plane anisotropy of mechanical properties may be explained by the recrystallization mechanism in connection with the weakened and broadened texture. The correlation of texture and mechanical behavior found in this study outlines the great potential of the RE-elements to improve the formability of magnesium sheets.

The findings described in this paper settle the need for further investigations of the effects of the rare earth elements. Of special interest is a deeper understanding of the nucleation and grain growth mechanisms. Furthermore quantitative formability studies by means of several series of forming tests would give a sound basis for future alloy development.

ACKNOWLEDGEMENTS

The authors appreciate financial support by the Deutsche Forschungsgemeinschaft (Grant Numbers KA 1053/10-1 and LE 1395/3-1) and NSF (DMR-0603066) as part of the Materials World Network.

References

1. S.R. Agnew, *Magnesium Technology 2002,* ed. Howard Kaplan (Warrendale, PA: TMS, 2002), pp. 169–174.
2. R. Cotam et al., *Materials Science and Engineering A,* 485 (2008), pp. 375–382.
3. L.W.F. Mackenzie and M.O. Pekguleryuz, "The Recrystallization and Texture of Magnesium–Zinc–Cerium Alloys," *Scripta Mater.,* 59 (2008), pp. 665–668.
4. J. Hadorn et al., "Influence of Rare Earth Elements on the Microstructure and Texture Development during Rolling of Magnesium Alloy Sheets" (Paper presented at the TMS 2009 Annual Meeting & Exhibition, San Francisco, CA, 14–18 February 2009).
5. J.Wendt et al., *Magnesium Technology 2009,* ed. S. Agnew et al. (Warrendale, PA: TMS, 2009), pp. 289–294.
6. J. Bohlen et al., *Acta Materialia,* 55 (2007), pp. 2101–2112.
7. C.E. Dreyer, R.H. Wagoner, and S.R. Agnew, *J. Mater. Process. Tech.* (submitted for publication 2008).
8. M.R. Barnett, M.D. Nave, and C.J. Bettles, *Materials Science and Engineering A,* 485 (2004), pp. 205–211.
9. K. Hantzsche et al., *Proceedings of the Light Metals Technology Conference*, ed. K. Sadayappan and M. Sahoo (Saint-Sauveur, Quebec: LMT, 2007), pp. 189–200.
10. H. Mecking, *Texture and Anisotropy,* ed. U.F. Kocks, C.N. Tomé, and H.R. Wenk (New York: Cambridge University Press, 1998), pp. 2–4.
11. V. Kree et al., *Practical Metallography,* 41/5 (2004), pp. 233–246.
12. A. Styczynski et al., *Scripta Mater.,* 50 (2004), pp. 943–947.
13. S.E. Ion, F.J. Humphreys, and S.H. White, *Acta Metall.,* 30 (1982), pp. 1909–1919.
14. N. Stanford and M. Barnett, *Scripta Materialia,* 58 (2008), pp. 179–182.
15. J.W. Senn and S.R. Agnew, *Magnesium Technology 2008,* ed. M. Pekguleryuz et al. (Warrendale, PA: TMS, 2008), pp. 153–158.

Kerstin Hantzsche, Jan Bohlen, and Dietmar Letzig are with GKSS-Forschungszentrum GmbH, Max-Planck-Str. 1, D-21502 Geesthacht, Germany; Joachim Wendt and Karl Ulrich Kainer are with the Institute of Materials Science and Technology, Hamburg University of Technology, Eißendorfer Straße 42, D-21073 Hamburg, Germany. Dr. Kainer can be reached at karl.kainer@gkss.de.