

COMPARISON OF TENSILE PROPERTIES AND CRYSTALLOGRAPHIC TEXTURES OF THREE MAGNESIUM ALLOY SHEETS

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Abstract

The most common commercially available rolled magnesium sheet alloy is AZ31B (typ. 3% Al, 1% Zn, 0.4% Mn, balance Mg). One of the often-cited shortcomings of this sheet is its limited formability at room temperature, which is attributed in part to a strong crystallographic texture in which the basal planes of the hexagonal unit cell are parallel to the plane of the sheet. Attempts have been made to avoid this rolling-induced texture by changing either (a) the alloy composition or (b) the rolling process. Specifically, sheet has been made using the conventional rolling practice, but changing the alloy to ZEK100 (typ. 1% Zn, 0.2% Nd, 0.2% Zr, balance Mg), or by keeping the AZ31B composition but rolling at a much higher temperature. In this report, both types of sheet are evaluated and compared with conventionally rolled AZ31B sheet. Both show reduced texture and attractive tensile properties, and therefore both are expected to show greater room-temperature formability than conventionally rolled AZ31B.

Introduction

Magnesium is the lightest (lowest density) of the common structural metals and therefore is potentially attractive for use in automotive applications to achieve mass reduction and the associated improvements in fuel efficiency and handling. Currently, automotive usage of magnesium is primarily in the form of die castings, and includes steering wheels, transmission housings, sunroof frames [1], and inner panels for doors and liftgates [2,3]. Numerous potential automotive applications of wrought magnesium alloys, including seat frames and IP beams, have been prototyped [4-7], but production implementation of sheet has been limited to a very low volume center console cover [8]. The issues which preclude greater implementation of the wrought alloys include the high cost of sheet, limited formability at room temperature, difficulty joining to other materials, and corrosion. Potential solutions to all of these issues are being studied by various groups around the world.

Mg sheet is formable at hot temperatures [9,10] and warm temperatures [11,12]. Ultimately, however, sheet formers would prefer to stamp magnesium alloy sheet at room temperature, similar to what is done with steel. This would reduce the costs associated with heaters, hot tools, and high-temperature-capacity forming lubricants, and might enable beneficial strain hardening of the part being formed. Such stamping might be done using blanks which benefit from having either a different chemical composition of the alloy, or which may have been thermomechanically processed differently so as to enhance formability.

There are many papers in the literature about the common commercial AZ31 wrought magnesium alloy, and several will be discussed here. Beer and Barnett [13] investigated the effects of temperature, strain rate, and annealing temperature after hot deformation, on the microstructure of AZ31. Both the deformation and annealing conditions influence the time required for stable recrystallization. Higher temperature and strain rate are good for obtaining high rate of recrystallization and coarse grains. Perez-Prado and Ruano [14] found that different recrystallizations occurred in the mid layer and outer surface of an as-extruded thick Mg sheet AZ31B during annealing, and that the $\langle 112\bar{0} \rangle$ prismatic component fiber texture predominates throughout the thickness. During the annealing of AZ31B extruded rod at 400°C for 30 minutes, Yi et al. [15] found that the texture changed from $\langle 101\bar{0} \rangle$ into the $\langle 112\bar{0} \rangle$ component.

In the aspect of formability, Takuda et al. [16] gave a flow stress model expressed in a simple form with the work hardening exponent, the strain rate sensitivity exponent and the strength coefficient, and it has a good fit in the investigated strain rate and temperature ranges. Ulacia et al. [17] proposed a new constitutive model based on modifying Johnson-Cook and accounting for the variation of strain hardening with strain rate over a wide range of strain rate and temperatures. Kim et al. [18] investigated the formability of AZ31B at elevated temperatures, and developed two failure criteria based on the effective strain or damage parameters. Both were formulated as a function of Zener-Holloman parameter, and deep drawing tests of AZ31B sheet at elevated temperatures were performed to validate the failure criteria.

Wu et al. tensile tested 0.5mm AZ31B-H24 rolled sheet at 250 °C and 370 °C, and found that bars pulled at 45° to the RD exhibited lower flow stress and greater elongation than those pulled at either 0° or 90° [19]. Fatemi-Varzaneh [20] also studied the mechanical properties of AZ31B after rolling between 300 and 450 °C. They found that a low rolling temperature and a high rolling strain led to increased strength and ductility in subsequent room temperature tensile testing. They attributed those increases to the uniform and fine (5 micron) grain size which forms by dynamic recrystallization under those rolling conditions. The normal plastic anisotropy (r value) of AZ31B was studied by Agnew and Duygulu [21]. At room temperature, the r value for rolled sheet in the H24 temper increases from ~1 in the RD to ~3 in the TD. It decreases with increasing temperature up to ~200 °C, especially in the transverse direction.

Magnesium alloys with rare earth (RE) additions have attracted many scholars in recent years because such additions weaken the texture and increase the formability. Liu et al. [22] studied the

mechanical properties, bore expanding performance, and limiting drawing ratio of ZE10 sheet in a temperature range from room temperature to 300°C. They showed that the ZE10 has better formability at 150-300 °C than at lower temperatures.

Zhang et al. [23] investigated the effects of Er additions on the properties of a semi-continuously cast Mg-1.5Zn-0.6Zr alloy. They found those additions increased the elongation and resulted in a refined grain size and higher yield strength. A yield point occurred when the Er content was greater than 2%, but the mechanism responsible for the yield point was not identified.

Stanford et al. [24] found the addition of small amounts of Gd decreased the grain size, and due to the Gd additions, the solute strengthening of the prismatic slip system was above 100 MPa. The Gd addition also reduced the texture and this resulted in greater ductility. The upper and lower yield point were observed when the Gd content was greater than 2.75%. They suggested that it was due to the Gd solute locking dislocations during the recrystallization annealing process which was done before tensile testing.

Mishra et al. found that the addition of 0.2% Ce to Mg reduced the tensile yield strength and greatly increased the ductility of extruded rounds. They attributed these changes in tensile properties to Ce-induced changes in the recrystallization [25]. Bohlen et al. have studied the texture and anisotropy of several as-hot-rolled Mg-Zn-RE alloy sheets [26]. They reported that the overall texture strength and the basal pole intensity aligned with the sheet normal direction was lower for RE-containing alloys than for conventional alloys. The anisotropy of the yield and flow strengths was reversed and the planar anisotropy was reduced to ~1 in comparison to conventional alloy, which was related to the fact that the dominant texture components in the Mg-Zn-RE alloys placed more grains in favorable orientations for basal slip and tensile twinning. A very recent study on sheet texture modification in Mg-based alloys by Gd, Nd, Ce, La and mischmetal alloying was performed by Al-Samman and Li [27]. They found that the different rare earths have different solid solubilities, and give rise to distinct microstructures, average grain sizes, and second phase formations. The Mg-based sheets with La, Nd, Gd, and mischmetal additions have less anisotropy of UTS and ductility than the one with Ce. The *r*-values of all the Mg-Zn-RE sheets are around 1.0 except the Mg-Zn-Ce sheet whose *r* values ranged from 1.2 in the RD to 1.7 in the TD (compared to 2.2 – 4.0 for AZ31).

Another approach to making a more formable magnesium alloy sheet is to change the rolling process rather than the alloy composition. For example, Hitachi Metals, has developed a rolling process in which a commercial magnesium alloy sheet is rolled at about 100 °C higher temperature than that in the conventional rolling process (lower than 400 °C) [28]. The resulting sheet is reported to exhibit a weak crystal orientation, resulting in excellent room-temperature formability which is comparable to those of aluminum alloy sheets.

Other non-conventional rolling processes have been applied to, or proposed for application to, magnesium sheet alloys on a lab scale, and show some attractive benefits to either the production of sheet or the properties of the resulting sheets, or both. These processes include high-speed heavy rolling [29], asymmetric shear rolling and asymmetric cross rolling [30].

The purpose of this work is compare the tensile properties and crystallographic texture of conventionally-rolled AZ31B magnesium alloy sheet with two alternative magnesium alloy sheet products.

Experimental Method

Roller sheets were obtained from two manufacturers in different thicknesses and tempers. Specifically, the conventionally rolled (CR) AZ31B sheets were 1.6 mm thick, and in the H24 temper (“strain hardened and partially annealed”). The CR ZEK100 sheets were 1.5 mm thick, and in the F temper (“as-fabricated”). The specially rolled (SPR) AZ31B sheets were 0.7 mm thick, and in an unspecified temper.

Tensile bars were waterjet cut at 0°, 45°, and 90° to the rolling direction (RD). The dimensions of the reduced section are 50 mm x 6 mm x sheet thickness. Annealing was done for 15 min in an electric furnace which was filled with argon gas to protect the tensile bars from oxidization. The AZ31B bars were annealed at 350 °C, and the ZEK100 bars were annealed at 500°C. These temperatures were chosen based on unpublished annealing studies in which we observed that AZ31B-H24 rolled sheet softened much at 350 °C, but that the ZEK100-F rolled sheet required 450-500 °C to soften. Rockwell F hardness was measured on the sheet surface with a 1/16” diameter indenter and 60 kg major load. Because the SPR AZ31B sheet is relatively thin and soft, its hardness was measured on the Rockwell superficial HR15T scale with 1/16” diameter indenter and 15 kg load, then converted to Rockwell F. Five measurements were repeated for each datum.

Crystallographic texture of the sheets was studied using an x-ray diffraction (XRD) technique which is often used to study powders, but was applied to intact sheets. Specifically, the x-ray diffraction data was collected with a D8 ADVANCE-DaVinci system using Cu K-alpha radiation (40 kV and 40 mA). The scan was made using Bragg Brentano mode, which is also known as ‘coupled theta/two-theta’ mode. The relative peak intensities were measured and compared to a standard Mg powder (random texture) provided by the International Crystallography Diffraction Data (ICDD) database, PDF 00-035-0821. This technique samples only those crystallographic planes which are parallel to the surface of the sheet.

The line intensities of sheet samples were normalized by those of the random sample to give a texture intensity factor TIF, with the help of the following equation. (The “o” subscript indicates the powder standard PDF 00-035-0821.)

$$TIF = \frac{(I_{(hkl)} / \sum I_{(hkl)})}{(I_{o(hkl)} / \sum I_{o(hkl)})} \quad (1)$$

An Instron tensile machine and an extensometer with a gauge length of 25 mm were employed. The crosshead speed was set at 10 mm/min for all tests. When performing tests for each type of sheet material, three bars in each orientation were pulled to failure, and one bar in each orientation was pulled to 10% elongation to determine *r* value.

Samples for metallography were carefully cut, cold mounted, ground, and polished in order to avoid introducing twins or other artifacts. They were etched with fresh mixtures of 4.2 g picric

acid, 10 ml acetic acid, 70 ml ethanol, and 10 ml distilled water in order to reveal the grains and grain boundaries .

Results and Discussion

Compositions

Elemental compositions of the sheet materials are as follows: CR AZ31B contains 3.0 %Al, 1.1 %Zn and 0.48 %Mn. SPR AZ31B contains 2.9 %Al, 1.1 %Zn and 0.44 %Mn. CR ZEK100 contains 1.3 %Zn, 0.2 %Nd, 0.25 %Zr and 0.01 %Mn. Al and Zn are added for strength, and Zn and Mn are added for corrosion resistance. Nd is for reduced crystallographic texture, and Zr is for grain refinement.

Hardness

The CR AZ31B was received in H24 temper, and therefore softened from 69 to 51 HRF as expected during 350 °C annealing to the O temper. The CR ZEK100 was received in F temper, and softened from 55 to 44 HRF during 500 °C annealing. In contrast, the SPR AZ31B was received in an unspecified temper, and did not soften (61 vs. 60 HRF) during 350 °C annealing.

Compared to the CR AZ31B, the lower hardnesses of the CR ZEK100 in both as-received and tempered conditions are likely due to the lower alloy content, lower amount of residual cold work in as-received sheets, and larger grain sizes (as will be discussed later in this report). The hardness of SPR AZ31B is less than that of the CR AZ31B-H24, likely due to a lower level of residual cold work, and is greater than that of CR AZ31B-O, likely because of a finer grain size.

Tensile Data

These data are summarized in Figures 1-3, and are discussed below.

Effect of Alloy and Rolling Process. Generally speaking, the CR AZ31B sheet showed the greatest strength, the ZEK100 sheet showed lowest strength and the greatest ductility, and the SPR AZ31B showed intermediate strength and lowest ductility. The CR AZ31B exhibits r values (normal plastic strain anisotropy) in the range 1.2 – 4.0, compared to 0.5 – 1.2 for the other materials, as shown in Figure 4. R value is often called “the resistance to sheet thinning”, and therefore in some discussions is casually equated with good formability, at least for certain types of metals and certain types of forming processes. Whether or not the high values of r measured for CR AZ31B translate into improved formability remains to be determined.

Effect of Temper. Annealing of the CR AZ31B sheet reduced YS and UTS, but increased % elongation. This trend was also shown by the ZEK100 sheet. However, for the SPR AZ31B sheet, annealing only slightly reduced YS, didn't change UTS, and increased % elongation. (The magnitudes of all of the annealing-induced changes vary with orientation of the bar's tensile axis relative to sheet RD.) Both n value and k value of CR AZ31B and CR ZEK100 increase obviously after annealing but only slightly for SPR AZ31B. That's attributed to the as-received CR AZ31B and CR ZEK100 having initial work hardening introduced by the rolling process, but the as-received SPR AZ31B having almost none. For CR AZ31B and CR ZEK100, since the annealing process removes the initial work hardening, the O-tempered materials have greater hardening behavior.

As regards r values, as shown in Figure 4, annealing increased the already-high r values of CR AZ31B, but had little effect on those of the other two materials.

Effect of Orientation. The YS, UTS, and % elongation for CR AZ31B-H24 are all somewhat greater at both 45° and 90° than at 0°. The same pattern holds true for that sheet after annealing. In contrast, both before and after annealing, the CR ZEK100 bars lose much strength and gain much ductility as the orientation is changed from 0° to 45° and 90°. The effect of orientation on SPR AZ31B is rather small both before and after annealing. Tensile properties of this sheet are generally lower at 45° than at 0° and 90°, but only very slightly.

The cause of in-plane anisotropy of tensile properties, especially YS, is likely due to details of the crystallographic textures and, therefore, the active deformation mechanisms. For example, even though the hcp basal plane poles are typically well aligned approximately normal the surface of rolled magnesium sheet, they often are reported to be tilted +/- a few degrees in the RD. Such tilting will facilitate deformation by both the basal slip mechanism and the extension twinning mechanism when pulling is along the RD [26].

Orientation also has a strong effect on the hardening parameters, n & k , of the CR ZEK materials (Figure 2) and on the normal plastic anisotropy, r , of the CR AZ31B (Figure 4). In both cases, the values increase as the tensile bar orientation changes from 0° to 90°. Both are attributed to crystallographic textures, and particularly to the slight tilting of basal plane poles away from the sheet normals [26].

Texture

TIF data are shown in Figure 5. If the orientations of the crystallographic axes of the grains in the sheets were distributed randomly in space, the TIFs would all have a value of one. Instead, for example, the TIF for the (002) basal planes in the CR AZ31B-H24 midplane sample is over five, indicating a strong tendency for these planes to be parallel to the plane of the sheet in this sample. Correspondingly, the TIFs of most of the other planes are much less than one. This type of texture is commonly reported for both rolled and extruded magnesium alloys. This texture is slightly stronger at the sheet midplane compared to the surface, and slightly stronger for H24 temper compared to O temper.

The TIF data for CR ZEK100 and SPR AZ31B show that these materials are less textured than the CR AZ31B, as expected. Specifically, the maximum TIFs are approximately two rather than five. The through-thickness variation of texture appears to be greater for the CR ZEK100 sheet than for the (thinner) SPR AZ31B sheet. The effect of annealing on TIFs is greater for the CR ZEK100 than for the SPR AZ31B.

It is interesting to note that whereas the literature, as discussed in [26], indicates that the rolling textures are similar for many wrought Mg alloys, but the recrystallization textures differ. Figure 5 shows the rolling textures for CR AZ31B-H24 and CR ZEK100-F to be much different. (The annealed textures are even more different.) This may be due to the former having more microstructural evidence of “cold work” than the latter.

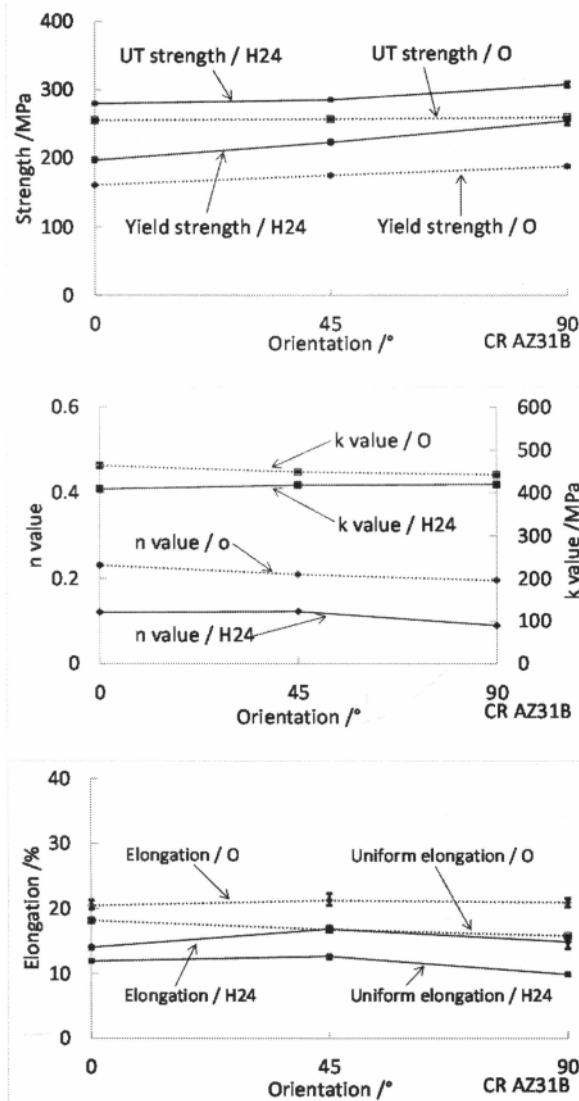


Figure 1. Orientation dependence of tensile properties of conventionally rolled AZ31B sheet in two tempers.

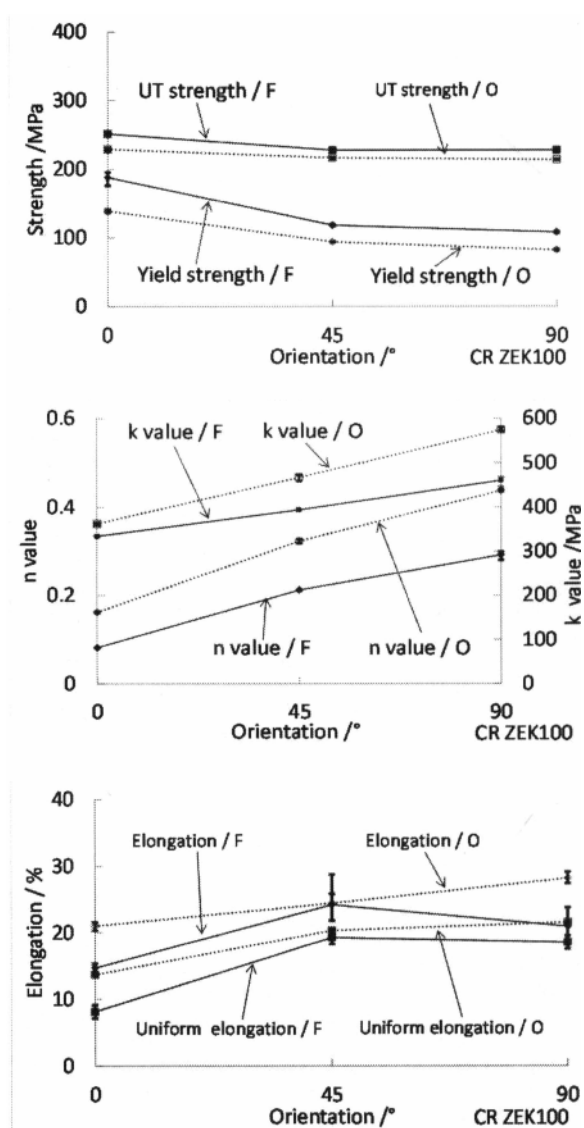


Figure 2. Orientation dependence of tensile properties of conventionally rolled ZEK100 sheet in two tempers.

Microstructures

Microstructures of the three sheet materials in each of two conditions are compared in Figure 6. There were plenty of twins in the as-received CR AZ31B and CR ZEK100, but much less in the as-received SPR AZ31B. During annealing heat treatment most of twinning was eliminated, and the grain sizes, measured in the rolling direction, increased from 4.2 μm to 8 μm for CR AZ31B, and from 15.3 to 19.3 μm for CR ZEK100. See Figure 7. However, the grain size of SPR AZ31B remained nearly constant at 5.6 μm during the annealing process. That is attributed to the work hardening in the as-received CR AZ31B and ZEK100, as it supplies more energy for nucleation and recrystallization than the SPR AZ31B in the annealing process.

Conclusions

Compared to conventionally rolled (CR) AZ31B magnesium alloy sheet:

1. Both a CR ZEK100 alloy sheet and a specially rolled (SPR) AZ31B alloy sheet exhibit significantly reduced crystallographic texture. Annealing the CR ZEK100 sheet further reduces the already weak basal texture, at least at the midplane of the sheet.
2. The CR ZEK100 is significantly weaker, more ductile, and shows more in-plane anisotropy of tensile properties. The pattern of in-plane anisotropy of strength is opposite that in CR AZ31B.
3. The SPR AZ31B is slightly weaker, less ductile, and shows less in-plane anisotropy of tensile properties.

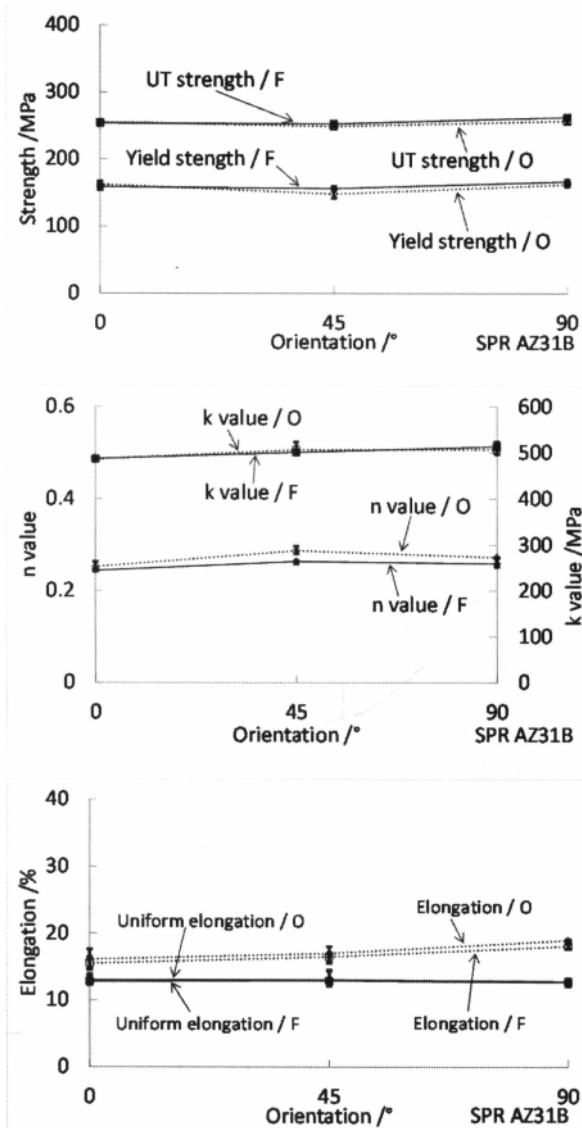


Figure 3. Orientation dependence of tensile properties of special rolled AZ31 sheet in two tempers

Also:

4. Annealing reduces the strength of both conventionally rolled alloys, but has no effect on the strength of the specially rolled alloy.
5. Annealing increases the ductility of all three alloys.

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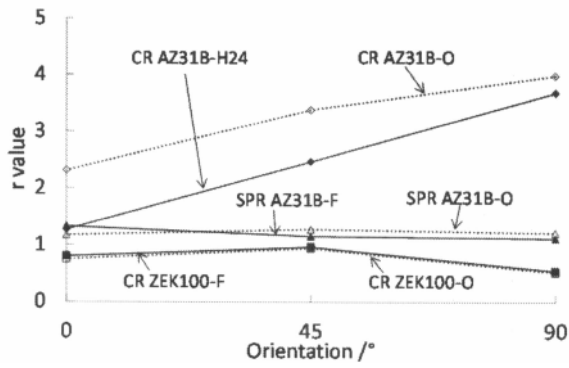


Figure 4. Orientation dependence of r values at 10% strain.

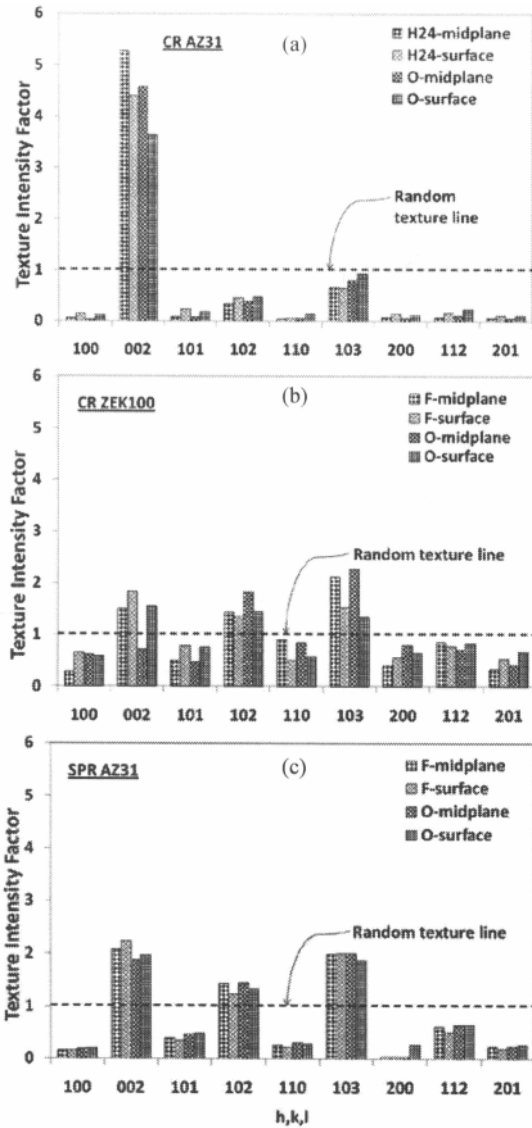
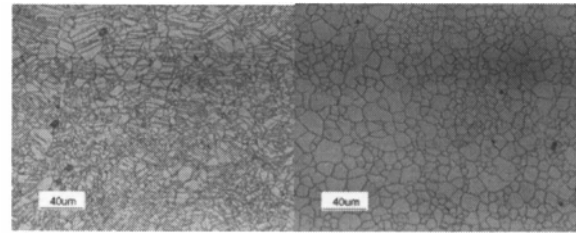
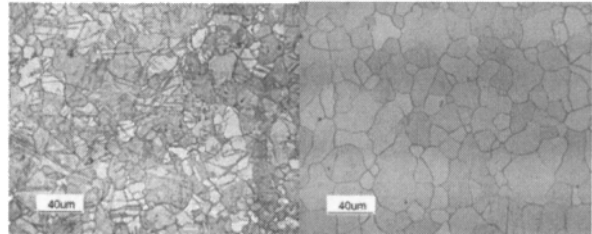


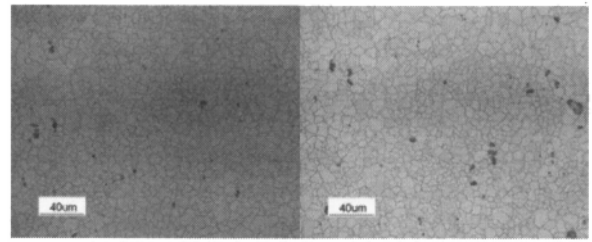
Figure 5. Charts showing texture intensity factors at sheet surface and sheet midplane for three alloys in two tempers each.



(a) CR AZ31B in H24 (left) and O (right) tempers



(b) CR ZEK100 in F (left) and O (right) tempers



(c) SPR AZ31B in F (left) and O (right) tempers

Figure 6. Photomicrographs of the three magnesium sheets before and after annealing. RD is horizontal. TD is vertical.

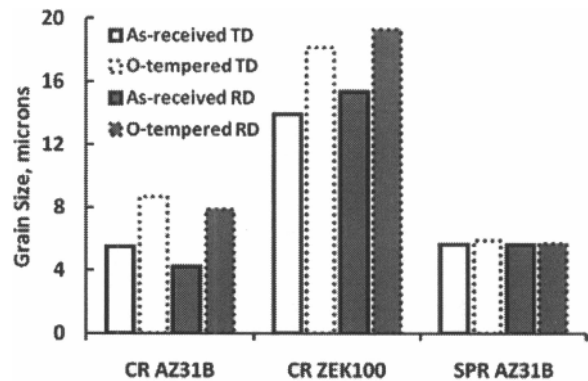


Figure 7. Mean intercept length of grains of three magnesium alloys in two tempers measured RD and TD.