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MECHANICAL PROPERTIES AND MICROSTRUCTURES OF TWIN ROLL CAST Mg-2.4Zn-0.1Ag-0.1Ca-0.16Zr ALLOY

C.L. Mendis[1] J. H. Bae[2] N.J. Kim[2] and K. Hono[1]

[1]National Institute for Materials Science, Tsukuba, Japan

[2]Pohang University of Science and Technology (POSTECH), Pohang, Korea

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Abstract

In our previous study, we reported that the additions of 0.1 at.% Ag and 0.1 at.% Ca to Mg-2.4Zn-0.16Zr (at.%) alloy enhanced the age hardening response, and extruded alloy showed tensile yield strength of 325 MPa with the T6 heat treatment. Considering its excellent age hardenability, we attempted to develop high strength sheets from the alloy by twin-roll casting (TRC). TRC sheet of 2 mm in thickness were hot rolled to ~1.2 mm. The TRC Mg-2.4Zn-0.1Ag-0.1Ca-0.16Zr alloy sheet showed tensile yield strength of ~ 320MPa and an elongation to failure of 17% after T6 heat treatment. EBSD study indicated the average grain size is ~18±2.5μm and the grains have a weak basal texture. TEM, showed a uniform distribution of ~5 nm diameter $MgZn_2$ phase. The high yield strength was attributed to the dispersion of rod-like precipitates.

Introduction

Magnesium alloys receive continued attention as a structural material from the transport and personal electronics sectors as weight reduction becomes more important [1]. Magnesium cast products have been recently used in automotive settings as part of engine blocks and other components in the power train. However, use of wrought magnesium alloys in structural components such as space frames or body panels has not been commercially realized at present. This is mainly due to the lack of formability and lower final strength of magnesium alloys as compared with aluminium alloys. Hot rolled magnesium alloys have shown appreciable strength but they have a strong basal texture which severely affects the deformability [2-3]. Twin roll casting (TRC) has been considered to be a viable option to developing magnesium sheets but with significantly lower basal texture [4-5]. It has also been reported that the microstructure features such as grain size is significantly refined due to the relatively fast solidification rates associated with the TRC process [5].

Recently, we reported that the trace addition of 0.1at% Ag and 0.1at% Ca to Mg-2.4Zn-0.2Zr (at%) alloy significantly enhanced the age hardening response of the base alloy by the refinement of rod like $MgZn_2$ precipitates [6]. The extruded and heat treated Mg-2.4Zn-0.1Ag-0.1Ca-0.16Zr (at%) alloy showed a yield strength of 325 MPa, ultimate tensile strength of 360 MPa after a T6 heat treatment [7]. The high yield strength was attributed to the dispersion of fine rod-like $MgZn_2$ precipitates that formed during ageing at 160°C, and the high ultimate tensile strength was attributed to the fine grain structure of about ~500 nm that formed by the dynamic crystallization during the extrusion process. The number of applications that require sheet products is expected to be larger than that of the extruded products. The extruded and heat treated Mg-2.4Zn-0.1Ag-0.1Ca-0.2Zr alloy has a tensile yield strength of 325 MPa, ultimate tensile strength of 360MPa and ductility of ~14%. This is comparable to that of many automotive aluminium alloys. Therefore, the development of a high strength magnesium alloy with appreciable ductility sheet while retaining the yield strength will expand the number of possible applications. In this contribution we report the mechanical properties and microstructure of twin roll cast and rolled Mg-2.4Zn-0.1Ag-0.1Ca-0.2Zr sheet.

Experimental procedure

An alloy with a nominal composition of Mg-2.4Zn-0.1Ag-0.1Ca-0.1Zr (at%) (Mg-6.3Zn-0.16Ca-0.5Ag-0.05Zr (wt%)) was prepared by induction melting in a steel crucible in an argon atmosphere. The alloy was then re-melted under a CO_2 and SF_6 mixture and transferred into a pre heated tundish held at 680-700°C. The roll gap in the twin roll caster was set at 2 mm and a roll speed of 4 m/min was used to cast sheets of ~2 mm in thickness. The cast alloy was homogenized at 350°C for 48 h and then hot rolled at 350°C with a total reduction of ~50% to the final thickness of 1.2 mm. The rolled samples were solution heat treated for 0.5 h at 400°C quenched into cold water and aged at 160°C. The hardness response of the alloys was measured using Vickers hardness testing with a load of 0.5 kg with an average of 10 measurements. Tensile properties of TRC and rolled and heat treated samples were measured using flat tensile specimens with gauge length of 12.5 mm, gauge thickness of 1 mm and gauge width of 5 mm at a strain rate of 6.4×10^{-4} s^{-1}.

The microstructures of the alloy were characterized with optical microscopy, scanning electron microscopy and transmission electron microscopy. TEM observations were conducted using a FEI Tecnai F30 microscope operating at 300kV. TEM specimens were prepared using twin-jet electo-polishing at -50°C, with a polishing voltage of 90V and a current of ~0.8mA in a solution consisting of 15.9 g of LiCl 33.48 g $Mg(ClO_3)_2$ 300 ml of 2-butoxy-ethanol in 1500 ml methanol. The electron back scattered diffraction (EBSD) was used to evaluate the micro-texture observed for TRC and rolled alloys.

Results and discussions

Microstructures of TRC processed alloy

Optical micrographs of twin roll cast (TRC) and SEM and TEM micrographs of TRC and rolled sheets are shown in Figure 1. The TRC sheet consisted of cellular microstructure with cell size of approximately ~ 20±5 μm, Fig 1 (a and b). The microstructure parallel to the rolling plane showed cellular structure that is typical of cast microstructures, Fig 1 (a) while the microstructure perpendicular to the rolling plane, Fig 1 (b) showed that cellular structure was elongated along the rolling plane illustrating that some of the microstructure is deformed during the twin roll casting. The TRC microstructures also showed a semi-continuous network of intermetallic particles. Following hot rolling at 350°C, the amount of intermetallic particles observed in the

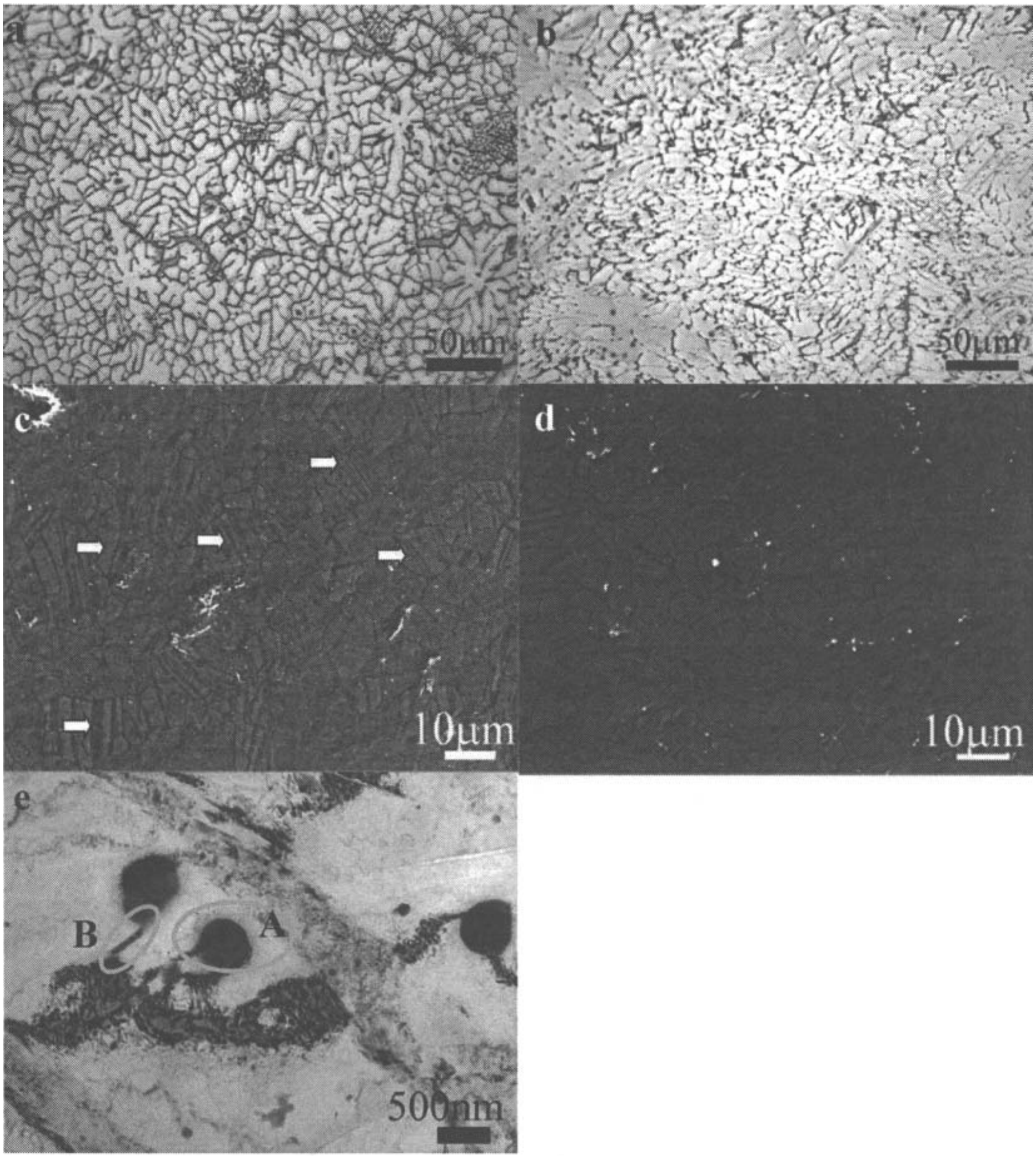

Figure 1 The Mg-2.4Zn-0.1Ag-0.1Ca-0.1Zr alloy (a, b) optical microstructure of TRC alloy; (c, d) SEM microstructure of TRC and rolled alloy and (e) TEM image of the TRC and rolled alloy showing precipitates observed, where A and B represent the two types of precipitates observed in the microstructure . (a and c) are parallel to the rolling plane while (b and d) are perpendicular to the rolling plane. The arrows in (c) indicate twinning observed in the microstructure.

microstructure decreased significantly, Fig 1 (c and d). The grain size remained approximately 18 ± 5 μm, Fig 1 (c,d) following rolling. There is a large number of twins in the rolled samples and this can be clearly observed on the microstructure parallel to the rolling plane, Fig 1 (c). This showed that the rolling reductions did not result in extensive recrystallization and grain growth during hot rolling. This can be contrasted with the hot extrusion results reported previously where a fine recrystallized grain size without significantly twining activity [7].

The TRC and hot rolled samples were examined with TEM and showed that there is multiple twinning within grains. The TEM results for the TRC and rolled alloy, Fig 1 (e). . Some coarse precipitates were also observed in the rolled microstructure, marked A and B. The electron diffraction patterns recorded from the coarse precipitate particles will be reported elsewhere. The spherical shaped particles, marked as A, were characterized as Mg_7Zn_3 orthorhombic phase (space group Immm a=1.403 nm, b=1.048 nm and c=1.449 nm) [8]. The lath-like particles designated B are $MgZn_2$ phase, (hexagonal with space group

P63/mmc a=0.521 nm and c=8.78 nm [9]). However, unlike with the as-extruded Mg-2.4Zn-0.1Ag-0.1Ca-0.16Zr alloy [7, 11], there are no fine rod-like precipitates of Mg(Zn,Zr)observed in the microstructure of the TRC and rolled alloy.

The EBSD orientation map recorded from the TRC and rolled alloy in the rolling plane is illustrated in Figure 2. The orientation maps collected from the rolling plane showed that majority of the grains were oriented with basal plane parallel to the rolling plane, Fig 2 (a). However there are some grains with their orientation away from the basal orientation observed with the orientation map recorded parallel to the rolling plane. There are some grains with defined twins within the grains and these can be clearly seen with the orientation map. The inverse pole figure, Fig 2 (b) recorded for the TRC and rolled alloy showed that grains are not oriented with basal plane parallel to the rolling plane but ~ 10° from the rolling plane.

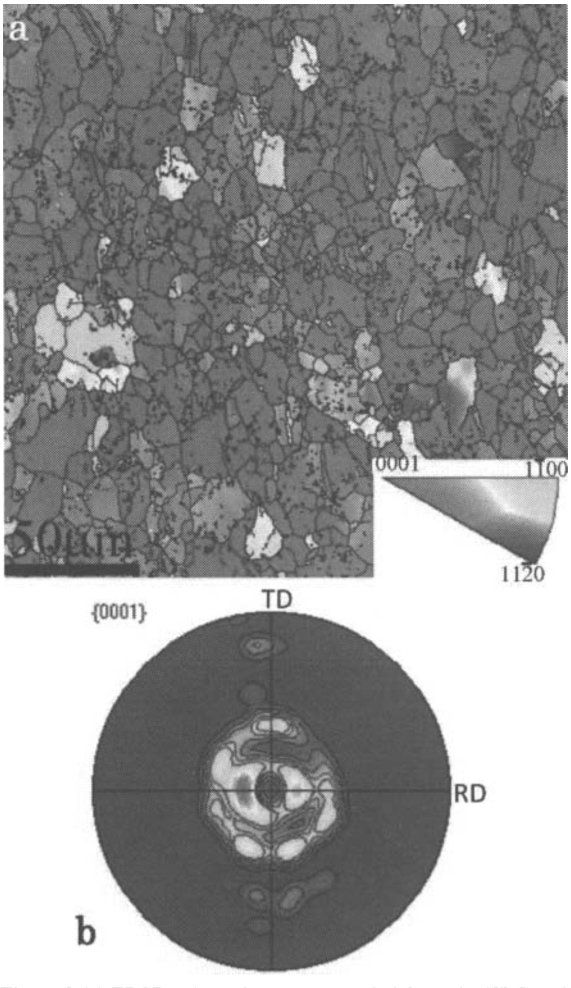

Figure 2 (a) EBSD orientation map recorded from the TRC and rolled alloy parallel to the rolling plane. And (b) the pole figure recorded from same EBSD orientation map showing the orientation of the $[0001]_{Mg}$ with respect to rolling plane.

Age hardening response and precipitate microstructures

The age hardening response of the TRC and hot rolled samples was measured following the solution heat treatment, Figure 3 (a). The hardness increases from as quenched hardness of ~62VH to a peak hardness of 96VH after ageing for 24 h at 160°C. The maximum hardness observed in the TRC alloy was similar to that observed for the cast and extruded alloy. However the time to reach peak hardness is shorter for the TRC alloy at 24h as compared with that for the extruded alloy at 48h [7].

The microstructure of the peak aged alloy samples were examined by transmission electron microscopy, Fig 3. It showed that the microstructure consisted two size of particles when observed with the electron beam parallel to the $[0001]_{Mg}$ showed that there is two different types of contrast from the precipitate particles, Fig 3 (b). The first is black in and was designated as diamond shaped precipitates and had a diameter of approximately 5±1.5 nm. The second is light grey, designated plate-like precipitates had a diameter of ~15±2.5 nm,. The microstructure observed was tilted approximately 40° from the $[0001]_{Mg}$ zone to show the microstructure perpendicular to the $[0001]_{Mg}$, Fig 3 (c). The black contrast precipitates now show elongated microstructure parallel to the $[0001]_{Mg}$ direction, thus these precipitates were deemed to be the rod-like precipitates of $MgZn_2$ phase. The diffraction patterns recorded from the black precipitates with $[0001]_{Mg}$ zone axis can be indexed according to $MgZn_2$ with the β_1' orientation relationship with magnesium as described in the previous investigations Fig 3 (d). The precipitates with grey contrast does not covert into rod-like microstructure but show more plate or lath-like structure when tilted away from the $[0001]_{Mg}$ zone axis . The electron diffraction patterns from these precipitates are indexed according to the $MgZn_2$ with the β_2' orientation relationship with magnesium, Fig 3 (e). The diameter of the rod-like precipitates observed in the TRC alloy could be compared with that for the extruded and heat treated Mg-2.4Zn-0.1Ag-0.1Ca-0.2Zr alloy [7] and the cast alloy [6, 7] and the number density of these precipitates (not reported here) is also similar regardless of the processing route.

Mechanical properties of TRC alloys

The tensile stress-strain curves of the twin roll cast and rolled alloys are shown in Fig. 4 (a). The TRC and rolled alloy showed a tensile yield strength (0.2%YS) of approximately 177 MPa with an ultimate tensile strength (UTS) of 285 MPa with a tensile strain to failure of approximately 28%. The tensile properties were lower than that for the as-extruded alloy [7]. The reasons for the lower yield strength is attributed to the significant differences observed in the microstructures; i.e., the extruded alloy contained much finer grain size compared to the TRC alloy [10] and a uniform distribution of fine scale precipitate distribution within the grains [7,10]. The fine scale precipitates found in the extruded alloy was not observed for the TRC and rolled alloy. However, the TRC and rolled alloy showed very high strength after solution treating at 400°C and ageing at 160°C for 24 h; i.e., yield strength of 320 MPa and the UTS of ~342 MPa with an elongation to failure of ~17%. The tensile properties reported for the TRC and rolled alloy is comparable with the extruded and heat treated alloy [7]. In both these alloys the major contribution to strengthening is the rod-like precipitate.

The tensile properties of Mg-2.4Zn-0.1Ag-0.1Ca-0.1Zr alloy is compared to ingot cast and rolled alloys [11] and other experimental TRC alloys as shown in Figure 4 (b). The tensile properties of the ingot cast and rolled Mg-Zn base alloys (ZM21) are comparable with the TRC Mg-2.4Zn-0.1Ag-0.1Ca-0.1Zr, but

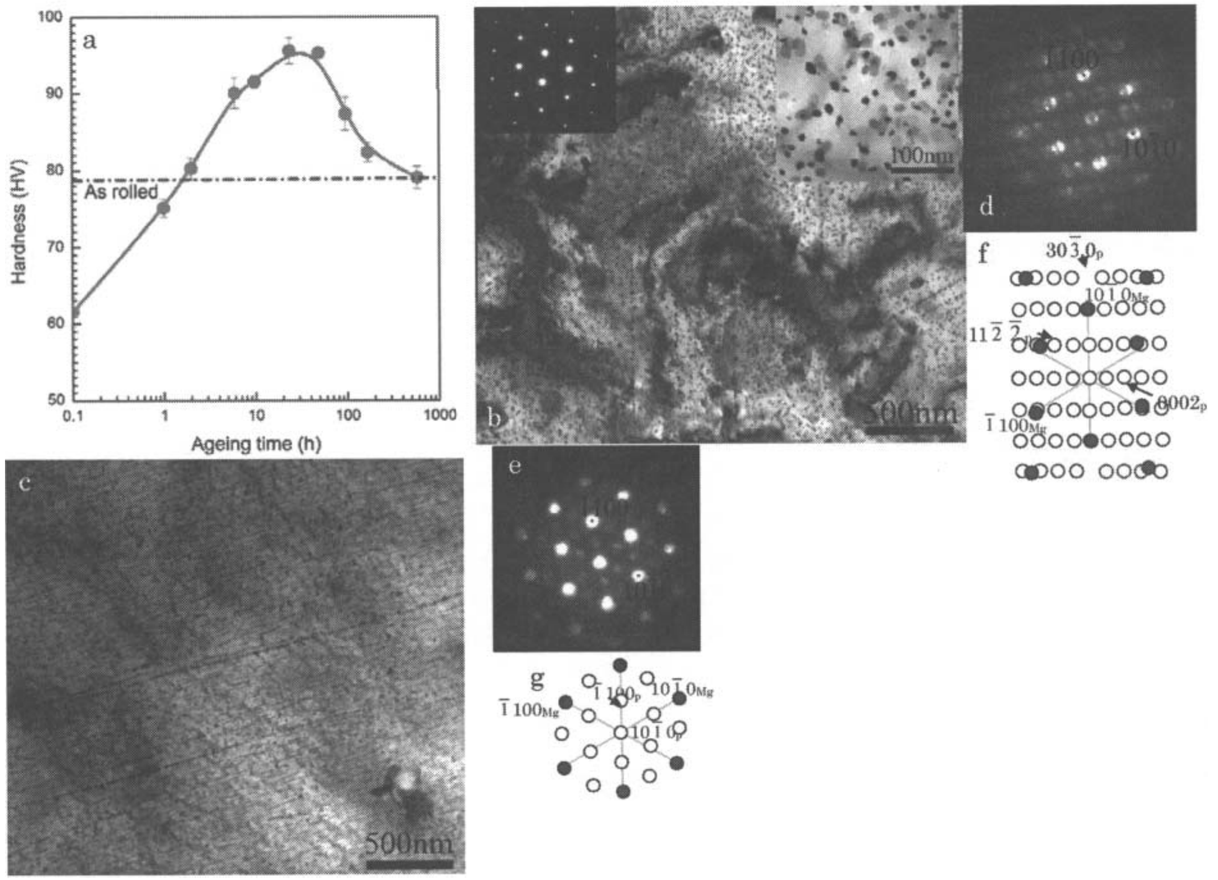

Figure 3 (a) the age hardening response of TRC Mg-2.4Zn-0.1Ag-0.1Ca-0.1Zr alloy at 160°C (b,c) TEM micrograph of the alloy peak aged at 160°C (b) electron beam is parallel to [0001]Mg (c) microstructure tilted 40° from [0001]Mg. electron micro-diffraction patterns from (d) diamond-shaped and (e) plate-like particles and (f, g) the schematic representation of diffraction patterns in (c and d) respectively.

with significantly lower elongation to failure. However the ZM21 and Mg-Th based HK31 alloys show significantly lower tensile yield strengths compared to the heat treated Mg-2.4Zn-0.1Ag-0.1Ca-0.1Zr TRC alloy. The Mg-1.7Gd-0.3Y (at%) alloy [12] has comparable yield strength but with far smaller elongations to failure. The yield strength of the as-TRC and rolled Mg-2.4Zn-0.1Ag-0.1Ca-0.1Zr alloy is comparable to that of the ZM61 alloy which has a similar composition. The ZMA611 and ZMA613 which contain a higher concentration of solute have higher yield strengths. Following ageing at 160°C, the TRC Mg-2.4Zn-0.1Ag-0.1Ca-0.1Zr alloy showed higher yield strength than the TRC processed and heat treated ZM61 and ZMA611 alloys subjected to a duplex ageing treatment. The mechanical properties of the TRC and heat treated Mg-2.4Zn-0.1Ag-0.1Ca-0.1Zr alloy is comparable to the tensile properties of heat treated 6009 series Al-Mg-Si based aluminium alloys both in yield strength and % elongation to failure [12]. In addition the formability of the TRC and rolled Mg-2.4Zn-0.1Ag-0.1Ca-0.1Zr alloy was found to be superior to any other reported Mg alloy sheets, which will be reported elsewhere.

Conclusions

Mg-2.4Zn-0.1Ag-0.1Ca-0.1Zr alloy show significant potential as a twin roll cast and rolled alloy for sheet applications. The grain size of the TRC and rolled alloy was approximately 18±5 μm. A tensile yield strength of 177MPa for the as twin roll cast and rolled sheet was substantially enhanced to 320 MPa following ageing at 160°C. Fine scale distribution of $MgZn_2$ precipitates phase with a rod-like morphology observed contributes to the increased strengthening after heat treatment.

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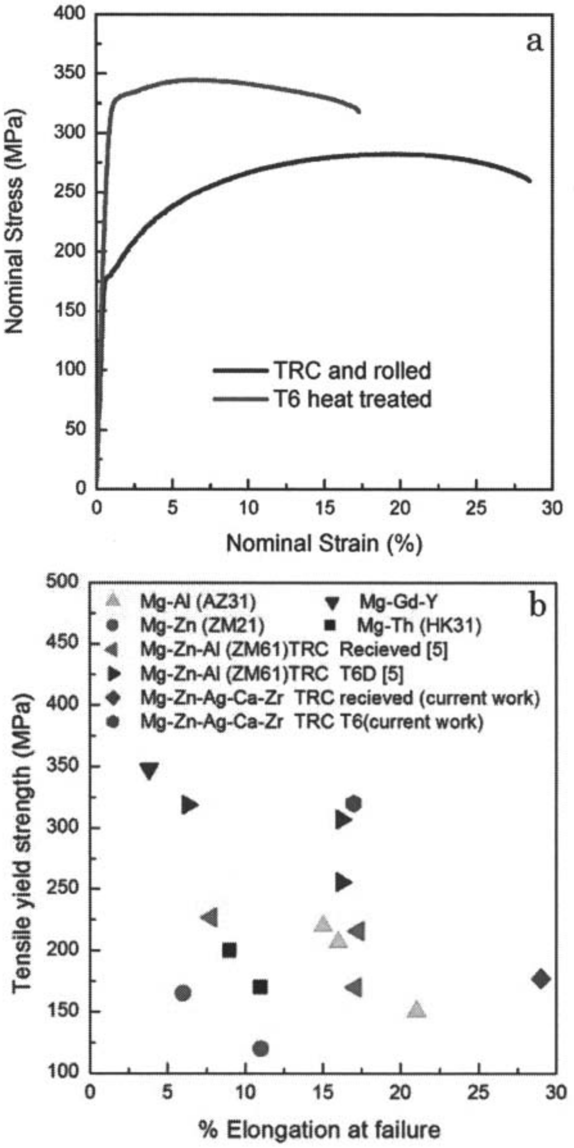

Figure 4 (a) Tensile stress strain curves for the twin roll cast (TRC) and rolled and heat treated Mg-2.4Zn-0.1Ag-0.1Ca-0.1Zr alloy and (b) comparison with the literature data for TRC and rolled or ingot cast and rolled magnesium alloys unless otherwise referred to data from [11 and 12]

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