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Twin-roll Casting after Intensive Melt Shearing and Subsequent Rolling of an AM30 Magnesium Alloy with Addition of CaO and SiC

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Keywords: AM30, CaO, SiC, intensive melt shearing, twin-roll casting, mechanical properties, hardness.

Abstract. Intensive melt shearing is a process that can be used for mixing ceramic particles into magnesium melt. It applies shear stress to the melt and can de-agglomerate nanoparticle additions to magnesium melts without the use of electromagnetic fields or ultrasound. A wrought magnesium alloy AM30 was selected for processing with intensive melt shearing and subsequent twin-roll casting. AM30 with additions of CaO and SiC were also processed by this route and the hardness and microstructure were investigated. Sheets were rolled and their tensile strength was determined. The work was done as part of the European Union research project ExoMet. Its target includes the production of high-performance magnesium-based materials by exploring novel grain refinement and nanoparticle addition in conjunction with melt treatment by means of external fields.

Introduction

Funded by the ExoMet Project, part of the 7th Framework Programme of the European Commission, the “physical processing of molten light alloys under the influence of external fields” is investigated [1, 2]. Some of these light alloys are magnesium based and one of the external fields selected is liquid shearing. The aim of strengthening magnesium alloys by the introduction of small particles or additions is not new, but with µm or even nm-sized reinforcement raises hopes that relatively small amounts such as 1-2 wt.-%, of particles will improve the mechanical properties to the same extent as 10–20 wt.-% of larger particles. The theory is they are the perfect size for Orowan strengthening by hard nanoscale reinforcement with an ideal distribution in the magnesium matrix [3]. In this paper, the magnesium alloy AM30 was chosen to be reinforced with 1.5 wt.-% CaO, and a combination of 1.5 wt.-% CaO with the same amount of SiC particles. Use of the twin-roll casting process for the production of flat strips, which can be further hot rolled in order to produce sheets, follows the great interest in further development of magnesium-based wrought materials, especially for automotive applications. Comparable work has been done by Wang et al. [4]. Strength was determined in rolling direction; the values of yield and tensile strength are comparable. Dissolution of CaO in pure Mg accompanied by formation of Mg_2Ca has been shown in [5].

Experimental Procedures

Schematic diagrams of the complete process of melt shearing [6-8] and twin-roll casting [9] is shown in Fig. 1. AM30 commercial alloy weighing 6 kg was melted in a steel crucible at 710°C under a protective atmosphere of N_2+1%SF_6. Reinforcement particles wrapped in an aluminium foil were added with the help of a steel plunger, followed by manual stirring. Three different

compositions were used for the present investigation: i) AM30, ii) AM30 + 1.5 wt.% CaO (average particle agglomerate size 10 µm), and iii) AM30 + 1.5 wt.% CaO + 1.5 wt.% SiC (average particle size 2 µm). The particles used are commercially available from Carl Roth (CaO) and Alfa Aesar (α-SiC). The melt was next transferred to a twin-screw melt-conditioning (MC) unit. A screw rotation speed of 500 rpm was used for intensive shearing of the melt at 645°C for 90 seconds. The conditioned melt was then fed into a horizontal twin-roll casting machine from a pre-heated tundish. The twin-roll caster has a pair of steel rolls with equal diameters of 319 mm. Water is circulated within the rolls to avoid heating up during the casting process. A protective environment (gas or flux) was maintained during the TRC process to prevent spontaneous ignition. A constant casting speed of 1 m/min was employed. The process thus far was performed at BCAST.
The strips had a thickness of 5.5 mm after twin-roll casting. These strips were hot rolled at MagIC, Helmholtz-Zentrum Geesthacht, to sheets with a thickness of 2.25 mm. The hot rolling was carried out after preheating the strips to 400°C. Four subsequent rolling passes resulting in a 20% reduction in thickness were performed using a warm-rolling mill.

Six micro-tensile specimens (gauge length: 9mm, 2x2 mm cross section) were extracted from these sheets using electro-discharge machining along the rolling direction and perpendicular to the rolling direction. A 5 kN universal testing machine was used and the displacement measured with a laser extensometer.

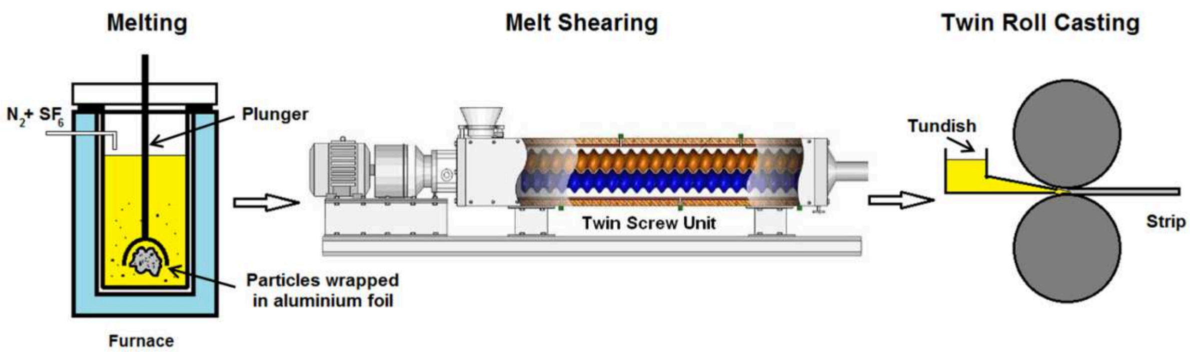
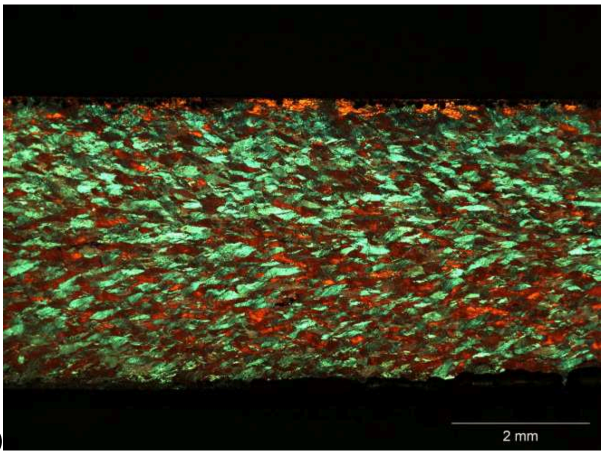


Fig. 1. Schematic diagram of the process.

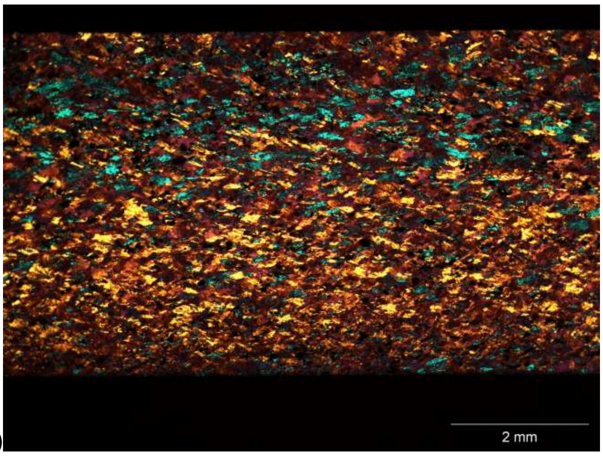
For metallography and hardness testing, samples were cut from each material, perpendicular to each direction, with x being the rolling direction (RD), y the direction perpendicular to the rolling process (TD) and z the dimension normal to the top surface of the rolled material (ND). Each sample was embedded in DEMOTEC 30, ground (500, 800, 1200, 2500 mesh) and used for hardness testing. Hardness tests were performed using the 5kg load with an EMCOTEST M1C 010 machine and 6 indents per sample were measured. After the hardness tests, the samples were ground with 2500 mesh paper, polished (1 µm diamond paste in ethanol) and etched (9% picric acid solution) for optical microscopy. Scanning Electron Microscopy (SEM) was performed on cross sections of the hot rolled samples with a VEGA3 Tescan microscope, operating at 20 kV. Samples for SEM were prepared with metallographic techniques described above but without etching.

Results

Metallography. The materials after twin-roll casting are shown in Fig. 2. AM30 is in Fig. 2a, AM30+CaO in Fig. 2b and AM30+CaO+SiC in Fig. 2c. The movement direction of the material through the rolls is from left to right. Grain size is largest in unreinforced AM30 and smallest in AM30+CaO+SiC. Due to larger cooling rate close to the rolls, the grain size at the surface is smaller in all cases. Centre line segregation is the flow of highly alloyed liquid phase during the last stage of solidification [10], it is visible in AM30, but not in the CaO containing alloys. After hot rolling the slabs in four passes to a thickness of 2.25 mm, micrographs of the surfaces of the resulting sheets are shown in Fig. 3. AM30 in Fig. 3a shows the smallest grains, AM30+CaO in Fig. 3b largest.



a)



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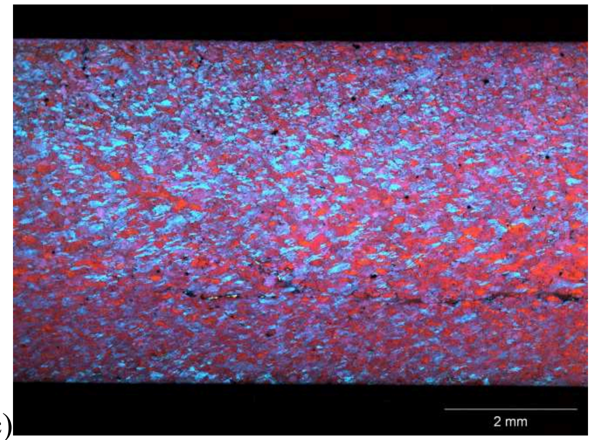
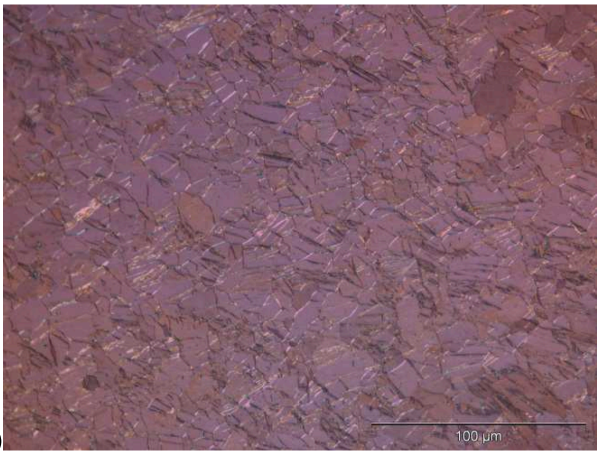


c)

Fig. 2. Micrographs of a) twin-roll cast AM30, b) twin-roll cast AM30+CaO and c) twin-roll cast AM30+CaO+SiC having average grain size of 184.0 μm, 176.9 μm and 161.4 μm, respectively. Casting direction is leftwards.



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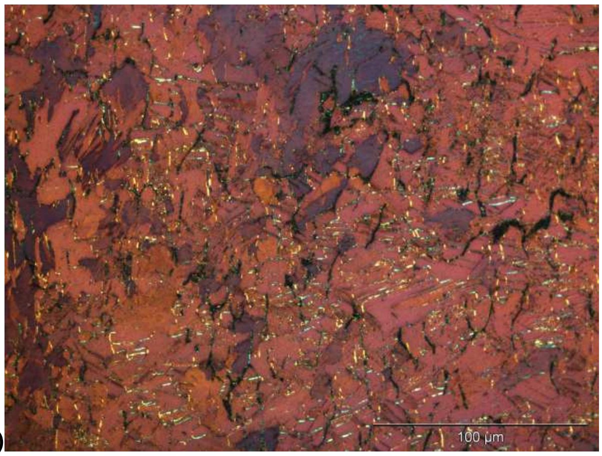


b)

c)

Fig. 3. Micrographs of a) twin-roll cast and rolled AM30, b) twin-roll cast and rolled AM30+CaO and c) twin-roll cast and rolled AM30+CaO+SiC having average grain size of 11.6 μm, 18.1 μm and 12.3 μm, respectively. Graphs show the sheet surface.

Hardness. HV5 hardness is shown in Fig. 4. Fig. 4a and Fig. 4b present the hardness of twin-roll cast slabs and rolled sheets, respectively. The x, y and z plane hardness was measured for each material. The TRC material shows an improved hardness with CaO addition, but the hardness of the rolled materials is very similar.

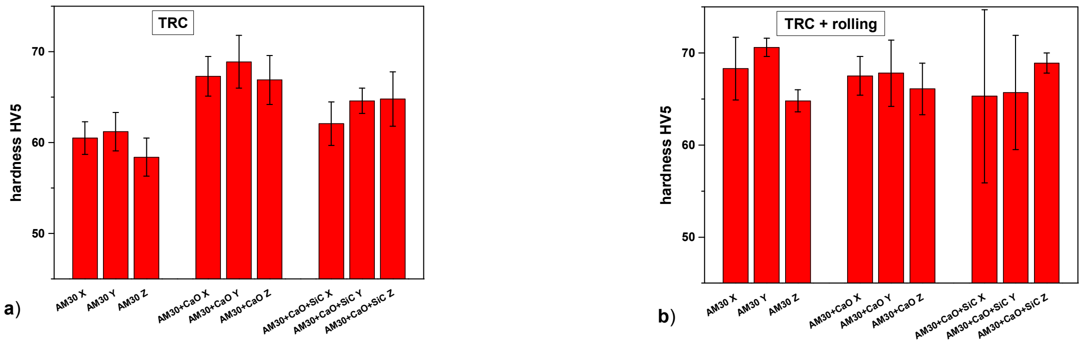


Fig. 4. HV5-hardness of AM30, AM30+CaO and AM30+CaO+SiC.

Tensile Strength. Flat micro specimens were used to test the mechanical properties parallel and perpendicular to the TRC and rolling direction. Six tests were performed for each material and experimental direction. The stress-strain curves are shown in Fig. 5. The upper row shows tests of the materials tested parallel to rolling direction (Fig. 5 a, c, e), the lower row perpendicular to it (Fig. 5 b, d, f). Tensile yield strength (TYS, $R_{p0.2}$), ultimate tensile strength (UTS, R_m) and elongation to fracture are shown in Fig. 6a and 6b, respectively. TYS is highest in unreinforced AM30 parallel to the rolling direction and lowest perpendicular to the rolling direction. In the unreinforced AM30 yield asymmetry is high. However, both of the composite materials show similar TYS parallel and perpendicular to the rolling direction and hardly any yield asymmetry can be determined. UTS and ductility are quite high for AM30 parallel to the rolling direction. It is just the opposite for the CaO reinforced AM30, which shows the highest UTS perpendicular to the rolling direction. The CaO and SiC reinforced AM30 has lower UTS and ductility.

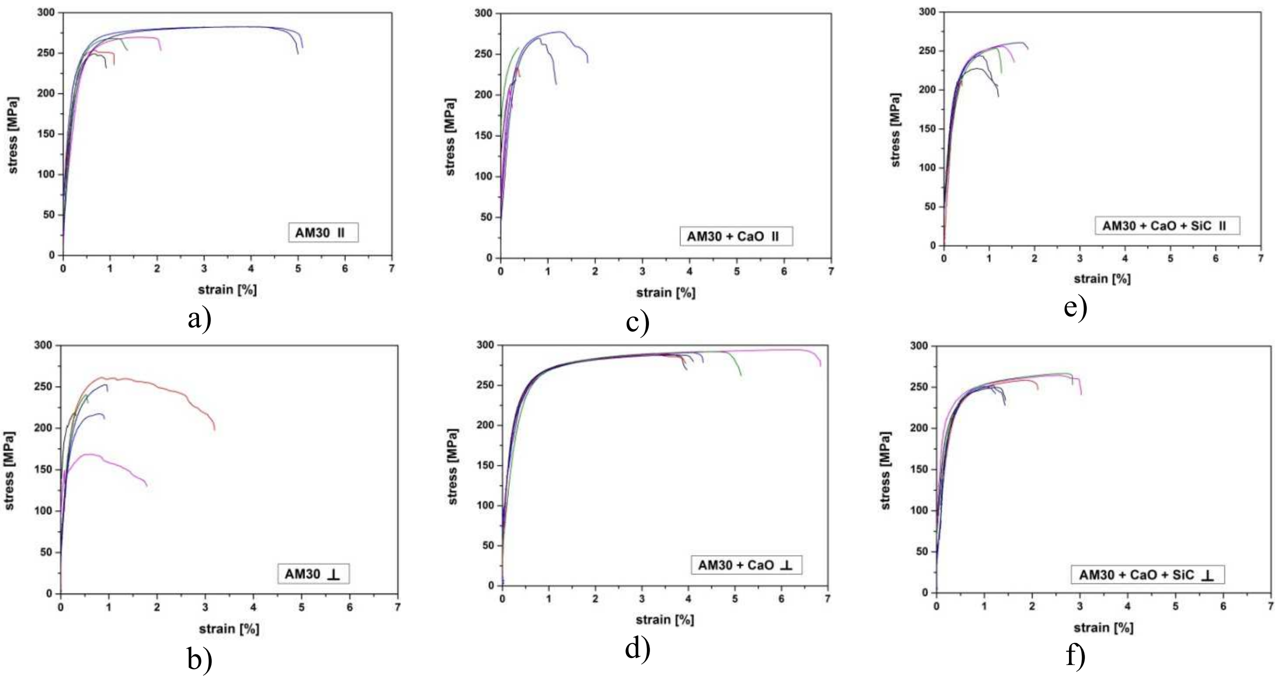
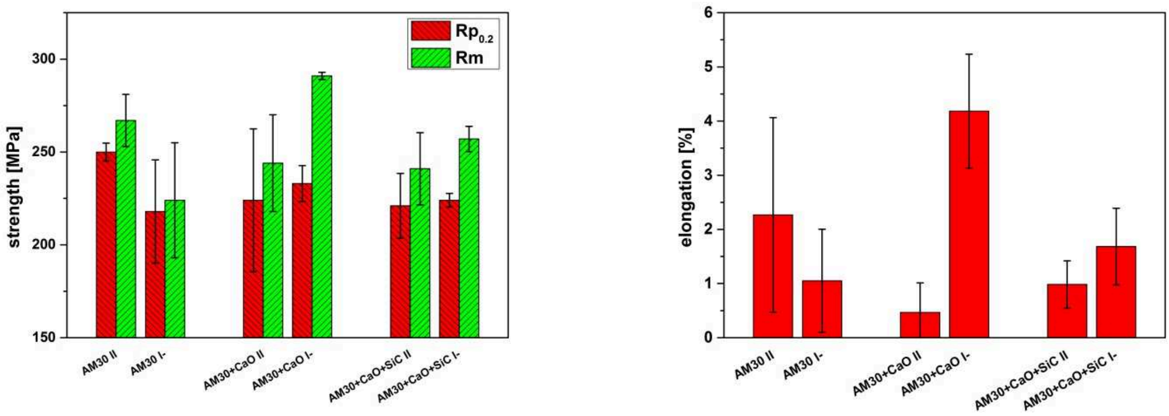


Fig. 5. Stress-strain curves of AM30 a) parallel to rolling direction and b) perpendicular to rolling direction, AM30 + CaO c) parallel to rolling direction and d) perpendicular to rolling direction, and AM30 + CaO + SiC e) parallel to rolling direction and f) perpendicular to rolling direction.



a) b)

Fig. 6. a) Tensile strength, and b) elongation of all materials tested at room temperature.

Scanning electron microscopy. The scanning electron micrographs for AM30 reinforced with CaO, and CaO and SiC, from the centre of the twin roll cast and hot rolled material are shown in Fig 7 (a) and (b) respectively. The rolling plane is parallel to the horizontal direction of the page. The CaO in both alloys disassociates during melting and subsequently forms $(Mg,Al)_2Ca$ phase as reported previously in cast Mg-Al alloys [11-12]. The central regions contained more intermetallic particles compared with the surface region. The region marked A in Fig 7 (a) contains CaO particles that are not incorporated into intermetallic phase observed while region B in Fig 7 (b) illustrates an example of agglomeration of SiC particles. Within the SiC particle agglomerations there are some brightly contrasting particles that were identified as CaO. In the SiC containing alloy the intermetallic particles of $(MgAl)_2Ca$ are distributed more uniformly as compared with the AM30+CaO alloy and no massive intermetallic particles, Fig 7 (a), are observed.

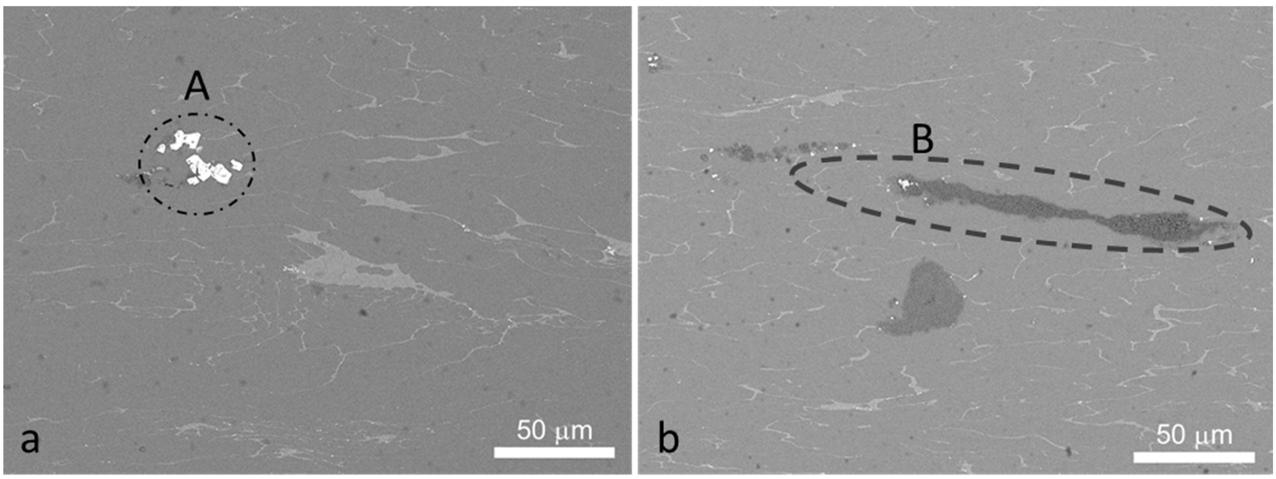


Fig. 7. Scanning electron micrographs for AM30 reinforced with CaO (a), and CaO and SiC (b), from the centre of the twin roll cast and hot rolled material.

Summary

Magnesium alloy AM30 was reinforced with 1.5 wt.-% CaO, and 1.5 wt.-% CaO + 1.5 wt.-% SiC. Particles were added and the melt was then transferred to a twin-screw melt-conditioning unit. The particles were de-agglomerated under intensive shearing of the melt at 645°C for 90 seconds. The conditioned melt was then fed into a horizontal twin-roll casting (TRC) machine from a pre-heated tundish. Slabs were subsequently rolled in four passes to sheets, which were mechanically tested. The TRC material with CaO addition shows improved hardness, but the hardness of rolled materials is very similar. High yield asymmetry of AM30 was reduced by the addition of CaO. The highest

UTS was demonstrated by CaO reinforced AM30 perpendicular to the rolling direction. Addition of both CaO and SiC resulted in poor ductility, which may be attributed to SiC agglomerates found in SEM. Dissolution of CaO and subsequent formation of $(Mg,Al)_2Ca$ phase positively influences mechanical properties and reduces grain size.

Acknowledgements

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