

Article

Mg-Based Materials with Quasiamorphous Phase Produced by Vertical Twin-Roll Casting Process

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Abstract: Metallic materials with micron grains, submicron grains, or amorphous structures have attracted great interest in recent decades owing to their excellent mechanical properties and corrosion resistance. Compared with traditional forming processes, rapid solidification technology has shown great superiority and potential in the preparation of materials in such structures. In this study, fine-grained quasiamorphous Mg-based alloy strips fabricated by a twin-roll strip casting process were explored using simulation and experimental methods. The concept of critical casting speed was proposed to reflect the optimum casting conditions. The product of critical casting speed and strip thickness was used to evaluate the cooling capacity of the casting system. Based on simulation results, a twin-roll strip-casting experiment was performed on a Mg-rare earth alloy. A novel puddle-like microstructure of the as-cast alloy strip was obtained. Tensile testing results showed that the novel strip exhibited improved ductility.

Keywords: amorphous; magnesium alloy; twin-roll casting; simulation

1. Introduction

Ultrafine-grained, nanocrystalline, and amorphous materials are topics of significant current research interest in modern materials science [1–3]. Nanostructured and amorphous magnesium alloys are particularly attractive for hydrogen storage and the automotive, aerospace, electronics, and biomedical industries because of their excellent properties [4–6]. However, the preparation of these materials is complex and their high processing cost is a major challenge.

Results of previous work showed that the high cooling capacity (i.e., maximum cooling rate of 10^3 – 10^4 K s^{−1} [7,8]) of the vertical twin-roll strip casting process makes it possible to produce materials with ultrafine-grained, nanocrystalline, and even amorphous structures. Twin-roll strip casting (TRC) integrates casting and hot rolling into one step and has the merits of shortening the processing time and saving energy. Most existing Mg-based materials have poor glass-forming ability, so high cooling rates are required when producing Mg-based glassy materials.

Water cooling is usually adopted in TRC cooling systems. If the cooling water could be replaced by a liquid with a lower temperature (e.g., liquid nitrogen) or the cooling system worked more efficiency, it would be easier to produce Mg-based metallic glasses using the TRC process. Based on these considerations, thermal-flow simulation of TRC Mg-based alloys was carried out and the cooling rates under various conditions were calculated. Under guidance of the computed results, strip-casting

experiments were then performed, aimed at producing magnesium alloy strips with ultrafine-grained or amorphous microstructure.

2. Materials and Methods

2.1. Twin-Roll Strip Casting Simulation

In our previous studies, we calculated the cooling capacities of two pilot-scale vertical twin-roll casters and determined the effects of strip-casting parameters on microstructure transformation [7,9]. It was found that pouring temperature T_p , casing speed v_c , and strip thickness δ are the main parameters that influence temperature distribution at the casting zone and the final microstructure of the as-cast strip. Therefore, casing speed v_c and strip thickness δ were the main focus of the current simulation.

Liquid nitrogen has an ultralow temperature of 77 K, so if we could find a suitable material capable of working in a cryogenic environment, a liquid nitrogen cooling system could be used in the TRC process instead of cooling water, and caster cooling capacity could be improved. Based on these considerations, numerical simulations were performed in two modes: mode (i) used a liquid nitrogen cooling system, in which the cooling water used in a conventional TRC process was replaced by liquid nitrogen; mode (ii) used liquid nitrogen injection nozzles and a cooling tank. As shown in Figure 1a, the copper rollers were internally cooled by water and the as-cast strip was cooled by liquid nitrogen.

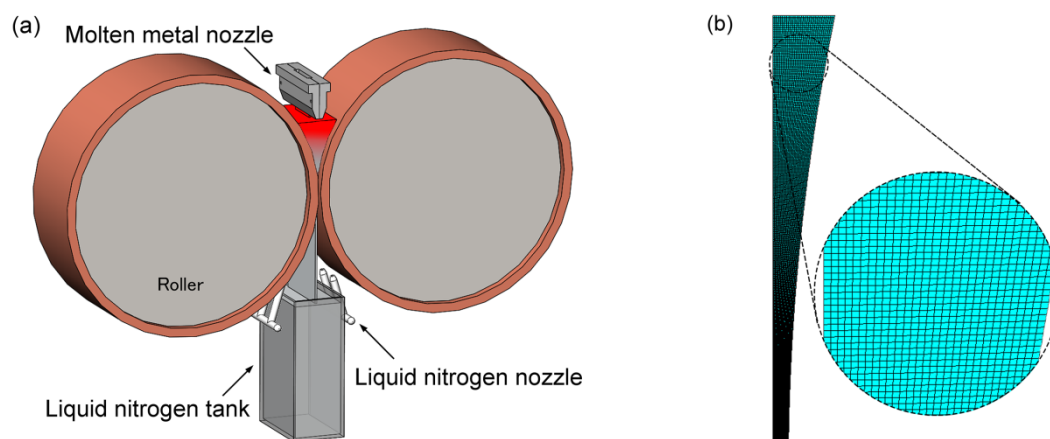


Figure 1. (a) Schematic diagram of twin-roll strip casting process; (b) corresponding two-dimensional finite-element model.

A two-dimensional finite-element model was adopted. The following assumptions were made for steady-state simulation: casting rollers were regarded as rigid bodies and there was no relative slip between the rollers and strip; the convection heat-transfer coefficient between liquid nitrogen and the as-cast strip was $500\text{--}5000\text{ W m}^{-2}\text{ K}^{-1}$ [10,11], surface temperature of the copper rollers were 323 K (water cooling) and 173 K (liquid nitrogen cooling, considering the Leidenfrost effect), free surface of the melt was steady, and the flow phenomenon in the molten pool was characterized as turbulent. Considering the latent heat, the equivalent specific heat method was adopted. A one-half domain of the TRC process was modeled due to its symmetrical geometry, as shown in Figure 1b. Physical properties of the material being cast were referenced to the AZ31 alloy. Other parameters used in the simulation are listed in Table 1.

Table 1. Input parameters for simulation.

Parameter	Value
Pouring (T_p)	923~973 K
Casting speed (v_c)	0.1~0.5 m s ⁻¹
Strip thickness (δ)	1 mm, 1.5 mm, 2 mm
Thermal conductivity [12–14]	60~120 W m ⁻¹ K ⁻¹

2.2. Experimental Details

The TRC process can be considered as a cooling system. A given apparatus has a certain cooling capacity: the less the input heat energy, the shorter is the cooling time required. In other words, a higher cooling rate could be obtained if we produced a thinner and narrower strip. In the current study, based on a vertical pilot twin roll caster with roll radius of 150 mm, roll width of 100 mm and maximum roll speed of 0.5 m·s⁻¹, magnesium alloy casting experiments in different conditions were carried out. Figure 2a shows an AZ31 alloy bar with a cross-section of 6 mm × 1.5 mm produced by the conventional TRC method. It had good toughness and could be bent through more than 180°. A cross-sectional microstructure of the bar is presented in Figure 2c. The grain size was smaller than that of the conventional as-cast strip with a larger strip width, as shown in Figure 2b,d.

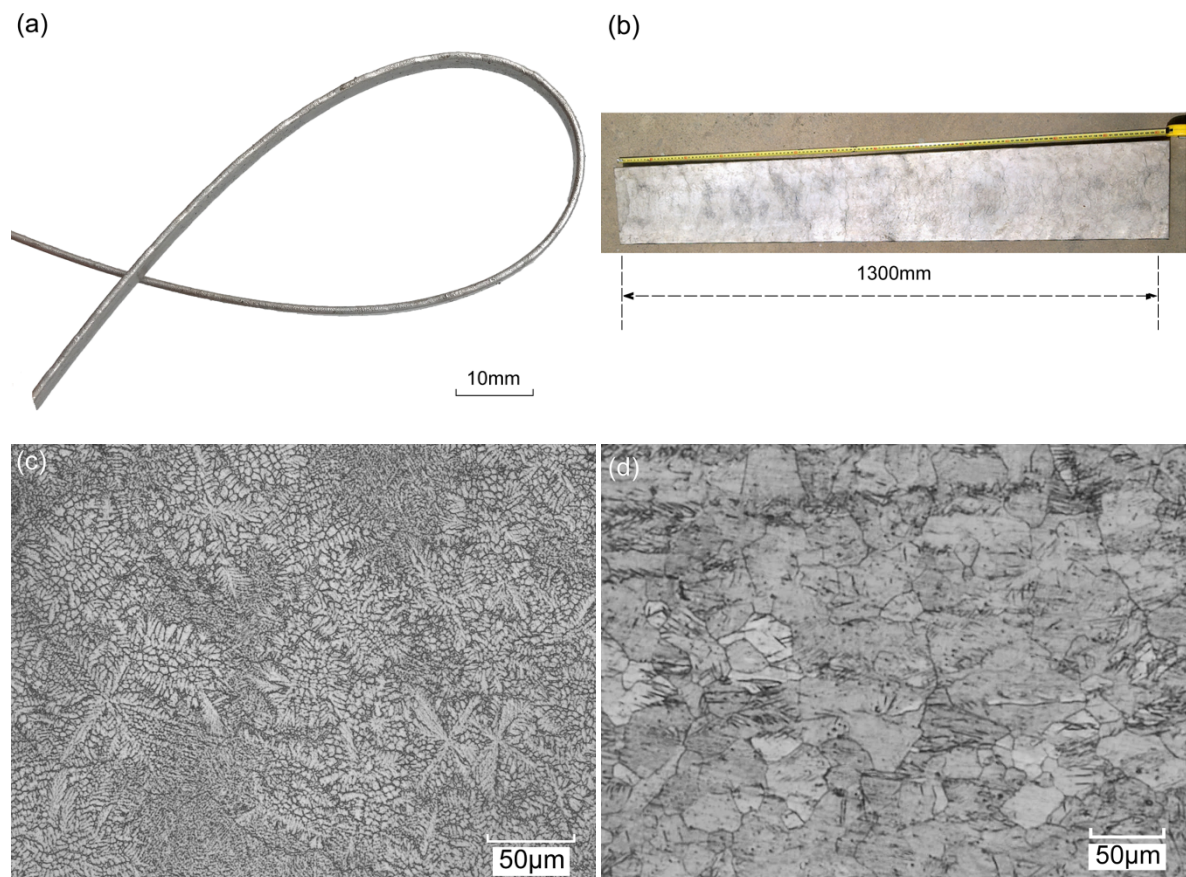


Figure 2. As-cast AZ31 alloy products and their microstructures: (a) bar with cross-section of 6 mm × 1.5 mm produced by less melt input, (b) strip with cross-section of 200 mm × 2.5 mm produced by more melt input, (c) cross-sectional microstructure of bar, and (d) cross-sectional microstructure of strip.

From the above considerations, we improved the pilot twin-roll caster to obtain a higher cooling capacity. As illustrated in Figure 1, a pair of liquid nitrogen nozzles was fixed near the strip outlet and a liquid nitrogen tank was placed right under the casting rollers. A large amount of liquid nitrogen

would have been needed if we replaced the cooling water with liquid nitrogen and the cooling circuit also needs to be well designed, so we kept the original water cooling system.

In the casting experiment, the initial roll gap was set to zero and an assisting device was adopted to counteract the large roll separating force on the movable roller side. The pouring temperature was 953 K and the casting speed was 0.3 m s^{-1} . Selection of the process parameters was based on the simulation results. As-cast Mg-rare earth (RE) alloy strips with 1.1 mm thickness and 50–60 mm width were obtained (Figure 3). Their chemical composition is listed in Table 2; the rare earth elements were considered to be helpful in the formation of glassy phases due to their negative value of heat of mixing [15].

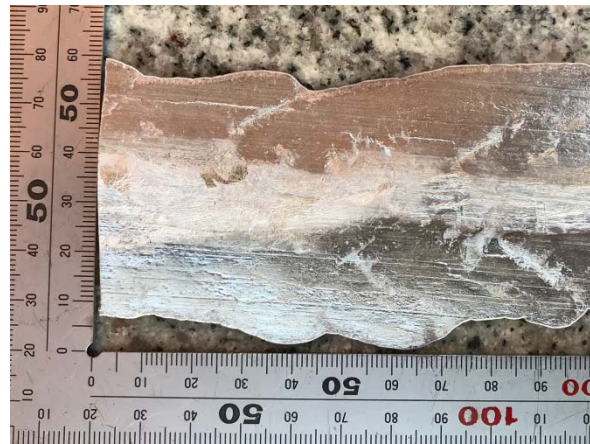


Figure 3. As-cast strip produced by the improved twin-roll casting method.

Table 2. Chemical composition of Mg-rare earth alloy.

Elements	Al	Si	Mn	Zn	La	Ce	Mg
wt. %	4.1779	0.1169	0.2550	0.1106	2.5007	3.5963	Bal.

In order to check out the mechanical properties of the as-cast Mg-RE alloy, tensile tests were conducted. The dimension of specimen is shown in Figure 4. The specimens were cut from the as-cast strip by a wire cutting machine. After that, the specimens were grinded and polished. During the tensile experiments, a SHIMADZU IS-5000 tester (Shimadzu Corporation, Kyoto, Japan) with tensile speed of $0.027 \text{ mm} \cdot \text{s}^{-1}$ at room temperature was adopted.

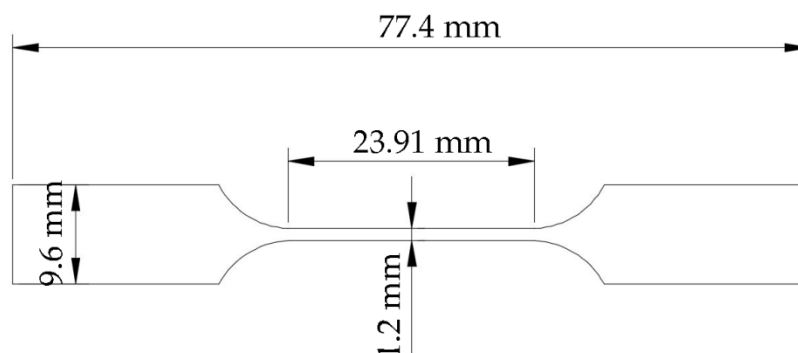


Figure 4. Specimen dimension for tension test.

3. Results and Discussion

3.1. Temperature and Flow Fields

According to the simulation results, the temperature distributions under different casting conditions could broadly be divided into two states. As shown in Figure 5, the shapes of isotherms were like a flat bow under low casting speed but resembled the letter V in appearance at high casting speed. This might be explained as follows. During the TRC process, thermal energy is mainly conducted along the roll radial direction under low casting speed due to the relatively long metal-roll contact time, so metal at the pool center region is sufficiently cooled before it flows down to the roll nip. The temperature distribution and flow state of the molten metal near the pool inlet under this condition are presented in Figure 5a. Flow direction of the metal in the casting pool is indicated by arrows, and the arrow length represents the relative velocity. It can be seen from the flow field that the melt flowed very slowly. In fact, the TRC process could become jammed during casting due to the limited rolling force of the caster. Contrarily, the metal flow state played a dominant role with increased casting speed. As shown in Figure 5b, the flow velocity was larger and oriented downward, so the temperature of the whole casting pool was elevated.

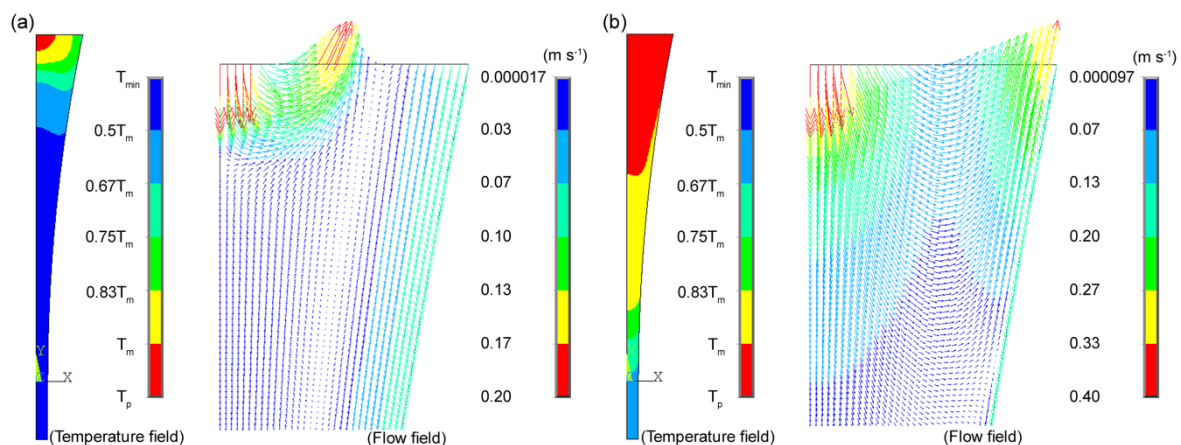


Figure 5. Simulated results of temperature fields and flow fields: (a) low casting speed and (b) high casting speed.

Several particular values were adopted to define the temperature contours, viz., pouring temperature T_p , melting point T_m , reduced glass transition temperature T_{rg} (T_g/T_m) [16], and temperature at the nose of the cooling curve T_n [17]. A cooling rate of approximately 10^6 K s^{-1} is required to form metallic glasses if $T_{rg} = 0.5$, whereas the undercooled melt becomes very sluggish on laboratory time scales when T_{rg} increases to 0.67 [17,18]. The value of the nose temperature is reported to vary between $0.45(T_g + T_l)$ and $0.55(T_g + T_l)$ [17], so values of $0.5T_m$, $0.67T_m$, $0.75T_m$, and $0.83T_m$ were used.

3.2. Critical Casting Speed and Cooling Rate Calculation

Previous studies showed that the temperature of as-cast material at the center of the roll nip T_{nip-c} (i.e., coordinate origin of the finite-element model in the current study) must be lower than the nose temperature of the continuous-cooling transformation diagram to avoid crystallization, and the surface temperature of as-cast material at the roll nip T_{nip-s} must be higher than the glass transition temperature T_g to prevent roll jamming [19,20]. Casting speed strongly influenced the temperature field of the casting, so we computed the exit temperatures under casting speeds of $0.1\text{--}0.5 \text{ m s}^{-1}$ for strip thicknesses of 2 mm, 1.5 mm, and 1 mm. The critical casting speed v_{ccs} for each condition was determined based on the consideration above (i.e., $T_{nip-c} < T_n$, $T_{nip-s} > T_g$). The results are listed in Table 3.

Table 3. Critical casting speeds under different casting conditions.

Cooling System	Mode(i)			Mode(ii)		
δ (mm)	2	1.5	1	2	1.5	1
T_{nip-c} (K)	523.80	533.37	545.89	519.08	523.90	535.22
T_{nip-s} (K)	507.65	519.98	536.56	508.89	516.15	530.63
v_{ccs} (m s ⁻¹)	0.18	0.23	0.32	0.13	0.18	0.27
$\delta \cdot v_{ccs}$	0.36	0.35	0.32	0.26	0.27	0.27

Critical casting speed increased with a decrease in strip thickness. The decrease of strip thickness reduced the time required for heat transfer from the pool center to the roll surface, so a higher casting speed was needed to prevent the roll from jamming. Moreover, it is noteworthy that the product of critical casting speed and strip thickness (i.e., $\delta \cdot v_{ccs}$) remained nearly constant under different casting conditions for each cooling system: the values for modes (i) and (ii) were approximately 0.34 and 0.27, respectively. It can be inferred that these constant values might have some relation to system cooling capacity, which can be represented by the following equation:

$$\dot{Q} = Q_1 \cdot Q_2 \cdot v_{ccs} \cdot \delta \quad (1)$$

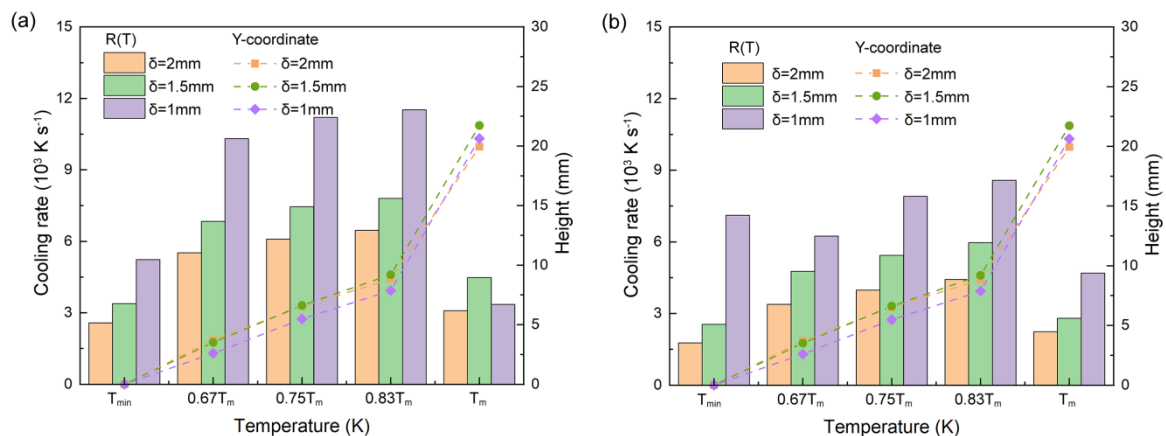
where $\dot{Q}$ is cooling capacity of the casting system, Q_1 is a physical term that refers to the thermophysical properties of the material, and Q_2 is a geometric term related to pool geometry.

According to the material derivative in the Eulerian coordinate system [21–23], cooling rate $R(T)$ at the center of the casting pool was calculated by Equation (2)

$$R(T) = \frac{DT}{Dt} = \frac{\partial T}{\partial t} + v \cdot \frac{\partial T}{\partial y} \quad (2)$$

where T is the temperature at the pool center, y is the melt position in the pool height direction, v is the velocity of the local melt, and t is time.

As illustrated in Figure 6, the values of $R(T_{nose})$ under the critical casting speeds listed in Table 3 were approximately 10^3 – 10^4 K s⁻¹ for both cooling modes. The thinner the strip, the larger was the cooling rate. The melt at temperatures around T_g and T_{nose} appeared at nearly the same positions within the casting pool, which showed that an appropriate temperature distribution could be achieved by adjusting the casting speed.

**Figure 6.** Cooling rates and melt positions under critical casting speeds: (a) cooling mode (i); (b) cooling mode (ii).

3.3. Twin-Roll Casting of Mg–Rare Earth Alloy

A major shortcoming of Mg-based bulk metallic glasses is their lack of ductility [24]. Many efforts have been devoted to improving their plastic deformation ability [20,25–27]. Bulk metallic glasses reinforced with crystalline phases exhibit improved ductility; however, materials with an inverse microstructure (i.e., amorphous/nanocrystalline particles embedded in a crystalline matrix) have hardly been studied. Fortunately, such a structure was obtained in the current study, as illustrated in Figure 7: we named this a puddle-like microstructure. To the best of our knowledge, this type of structure has never been reported in literature prior to the doctoral dissertation submitted in 2018 [28].

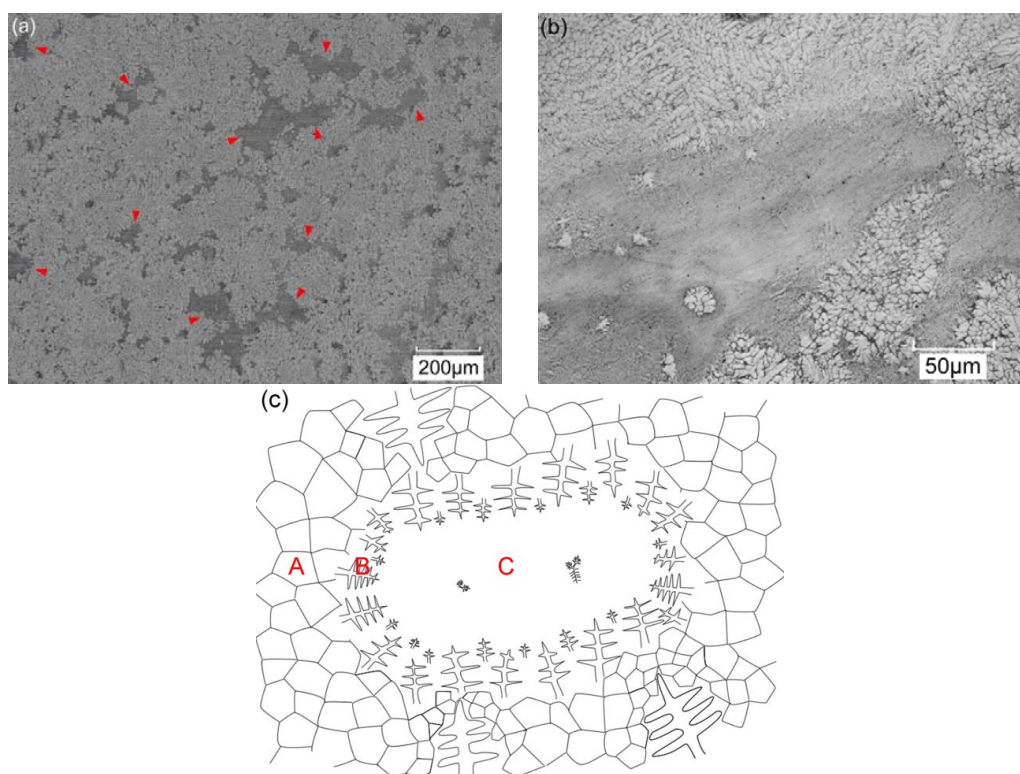


Figure 7. Puddles and puddle-like microstructure: (a) puddle-like microstructure, (b) quasiamorphous phase, and (c) schematic illustration.

As shown in Figure 7a, the microstructure of the as-cast strip was mainly characterized by fine equiaxed grains and fine dendrites with closely spaced secondary dendrite arms. There were also some special regions distributed in the crystalline matrix that did not reveal any crystalline features under optical observation (marked with red arrows). The microstructure of these special regions was a quasiamorphous phase (Figure 7b). As stated above, the crystalline matrix composite with quasiamorphous particles was named a puddle-like microstructure. Energy-dispersive X-ray (EDX) analysis of the quasiamorphous phase and crystalline matrix are listed in Table 4. Further details concerning the puddle-like microstructure will be published elsewhere.

Table 4. Energy-dispersive X-ray compositional analyses corresponding to quasiamorphous phase and crystalline matrix.

Elements	Mg	Al	Si	La	Ce
Quasiamorphous phase	87.08	4.97	0.22	3.05	4.68
Crystalline matrix	95.32	1.64	0.29	1.22	1.54

As illustrated in Figure 7c, the quasiamorphous phase and its surroundings were mainly divided into three parts. Zone A shows the crystalline matrix with fine equiaxed grains and dendrites; Zone B is characterized by fine dendrites with closely spaced secondary dendrite arms distributed around Zone C; and Zone C indicates the quasiamorphous phase, in which nuclei may exist, but their growths are inhibited. EDX analysis showed that the La and Ce contents in the quasiamorphous phase were higher than those in the crystalline matrix. This might be helpful for formation of the quasiamorphous phase. We further confirmed the nature of the quasiamorphous phase by transmission electron microscopy. It can be seen from the selected-area electron diffraction pattern that the quasiamorphous phase (Figure 8a) has no clear crystalline feature comparing to the crystalline matrix (Figure 8b).

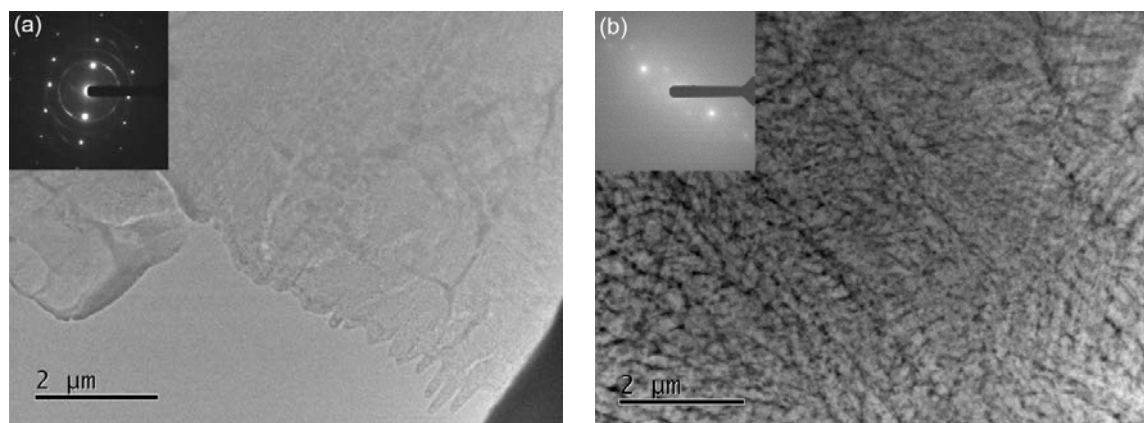


Figure 8. Bright-field electron micrograph with an inset of selective area electron diffraction pattern: (a) crystalline phase; (b) quasiamorphous phase.

Tensile properties of the as-cast Mg–RE strips at room temperature and those of other Mg-based alloys produced by different methods are listed in Table 5. Data for the AZ31 strips produced by TRC at three different casting speeds were performed by Dr. Hu, a senior of our laboratory [29]. The as-cast Mg–RE strips possessed moderate ultimate tensile strength and larger elongation compared with the other materials listed in Table 5. Contrary to the intrinsic brittleness of metallic glasses [30,31] and limited ductility of the amorphous matrix composite sheets [20], Mg–RE strips produced in the current work also exhibited improved mechanical properties. These might be attributed to the special microstructure, that is, the fine crystalline matrix enhanced ductility and the major portion of local stress was borne by the quasiamorphous phase. From the scanning electron microscopy (SEM) image of the tensile fracture surface (Figure 9), we can infer that the zigzag fracture route is helpful in absorbing energy during tension.

Table 5. Tensile properties of Mg-based alloys at room temperature.

Material	Production Method	UTS (MPa)	Elongation (%)	Ref.
AZ91	Casting	131	1~3	[32]
ZK60	Casting	275	5	
AZ91	Powder metallurgy	432	6	
ZK61	Powder metallurgy	400	7	
AZ31	TRC ($v_c = 0.22 \text{ m s}^{-1}$)	172.6	7.3	[29]
AZ31	TRC ($v_c = 0.3 \text{ m s}^{-1}$)	205.12	5.88	
AZ31	TRC ($v_c = 0.5 \text{ m s}^{-1}$)	182.35	5.14	
Mg-RE	TRC ($v_c = 0.3 \text{ m s}^{-1}$)	215.88	12.68	Current work

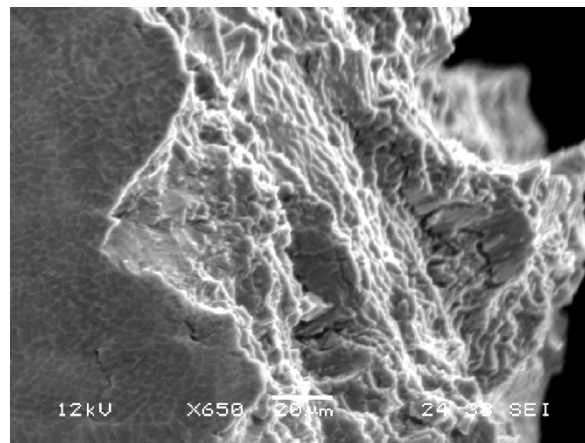


Figure 9. Tensile fracture surface of the as-cast Mg-RE alloy.

A recent study showed that the squeezing force during TRC contributes to a higher energy state of metallic glasses, which can improve plasticity of the as-cast glassy phase [33]. This might be another reason for the superior tensile properties of the as-cast strip produced in this work.

4. Conclusions

In this study, twin roll strip casting of magnesium alloys was investigated using simulation and experimental methods. Several main conclusions are reached, as follows:

- (1) As casting speed increased, the shape of isotherms at high temperatures in the casting pool transformed from a flat-bow shape to a V shape, which meant that the temperature of the entire casting pool was elevated.
- (2) Critical casting speed increased with a decrease in strip thickness. The value of the product of these two parameters ($\delta \cdot v_{ccs}$) remained nearly constant for all cooling systems and both cooling modes. The constant value may reflect the cooling capacity of the cooling system: a larger value corresponded to a higher cooling capacity.
- (3) A novel puddle-like microstructure, comprising a crystalline matrix composite containing quasiamorphous particles, was obtained by TRC of Mg-RE strips. To the best of the authors' knowledge, this type of Mg-based alloy structure has never been reported before.
- (4) Tensile test results showed that the novel as-cast Mg-RE strip exhibited superior tensile properties to those of amorphous matrix composite sheets and conventional as-cast alloys. This will be beneficial for downstream processing.

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