

## Low temperature mechanical property of AZ91D magnesium alloy fabricated by solid recycling process from recycled scraps

LI Dong-hua<sup>1</sup>, HU Mao-liang<sup>2,3</sup>, WANG Hai-bo<sup>1</sup>, ZHAO Wang-an<sup>1</sup>

1. School of Civil Engineering and Architecture, Harbin University of Science and Technology, Harbin 150080, China;

2. School of Materials Science and Engineering, Harbin University of Science and Technology, Harbin 150040, China;

3. National Engineering Research Center for Light Alloy Net Forming, Shanghai Jiao Tong University, Shanghai 200240, China

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**Abstract:** Low temperature mechanical properties of AZ91D magnesium alloy fabricated by solid recycling process from recycled scraps were studied. Various microstructural analyses were performed using optical microscopy (OM) and scanning electron microscopy (SEM). The recycled specimens consist of fine grains due to dynamic recrystallization and the interfaces of original individual scraps are not identified. Tensile tests were performed at a strain rate of  $5 \times 10^{-3} \text{ s}^{-1}$  at room temperature (27 °C), -70, -100 and -130 °C, respectively. Ultimate tensile strength of the specimens increases slightly with decreasing the tensile temperature, and elongation to failure decreases with decreasing the tensile temperature. The tensile specimens at -130 °C show the highest ultimate tensile strength of 360.65 MPa and the lowest elongation to failure of 5.46%. Impact tests were performed at room temperature (27 °C), -70 and -130 °C, respectively. Impact toughness decreases with decreasing the impact temperature. The impact specimens at -130 °C show the lowest impact toughness of 3.06 J/cm<sup>2</sup>.

**Key words:** AZ91D magnesium alloy; solid recycling process; mechanical property; impact toughness

### 1 Introduction

In the last two decades, magnesium alloys have been widely used in electronic, automotive and aviation industries [1], where mass reduction is necessary requirement, due to their high specific stiffness, high specific strength, excellent castability and good recycling ability [2]. With an increase in demand, a large amount of waste in the form of scraps and chips is produced in the manufacturing process, such as die casting and plastic forming process [3]. At present, common recycling treatments are remelting of scraps and chips. However, these methods are costly because magnesium alloys are susceptible to oxidation. MAMORU et al [4] proposed that solid recycling process is an effective processing method for efficiently reclaiming magnesium scraps and chips. The advantage of the recycling process is that the treatment is conducted in the solid state, which does not need a special protective environment or extra caution [5–6]. Solid recycled materials show excellent

mechanical properties. In the solid recycling process, scraps and chips are fabricated by consolidation using plastic deformation and by microstructural control such as grain refinement and dispersion of the oxide precipitates [7–8].

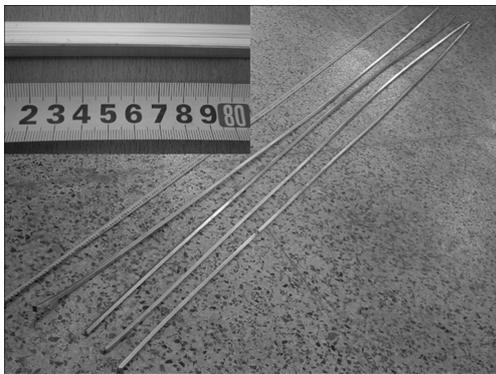
Experimental evidence showed that magnesium alloys such as AZ31 [9–11], AZ31B [12], ZK60 [13], AZ80 [14] and AZ91 [4] fabricated by solid recycling process with severe deformation exhibited high tensile strength and high elongation to failure at room temperature. However, low temperature microstructures and mechanical properties of magnesium alloys fabricated by solid recycling process have not been investigated. Many magnesium alloys are used in the condition of low temperature, and it will be valuable to investigate their low temperature mechanical properties. In this work, hot press and hot extrusion are employed for fabricating the specimens from AZ91D magnesium alloy scraps. Low temperature mechanical properties of AZ91D magnesium alloy fabricated by solid recycling process from the recycled scraps were investigated. Low

temperature fracture mechanism is also discussed.

## 2 Experimental

The materials used in this work were AZ91D magnesium alloy scraps directly coming from the die casting process in the industrial manufacture [15]. These scraps included water gap, casting gap and flow path agglomeration, etc.

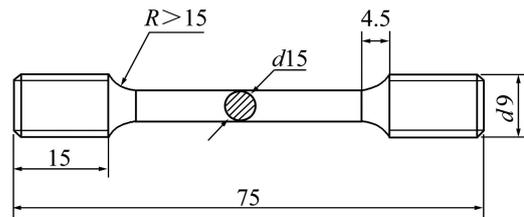
Firstly, AZ91D magnesium alloy scraps were loaded into a 95 mm cylindrical container in diameter and hot-pressed. The pressure, holding time and temperature were 400 MPa, 300 s and 400 °C, respectively. Following the hot-press process, the container with the formed billet was held for 300 s at 450 °C. At last, hot extrusion was carried out at an extrusion ratio of 29.3:1 and an extrusion rate of 0.4 mm/s to produce a square bar with a 11 mm edge, as shown in Fig. 1.



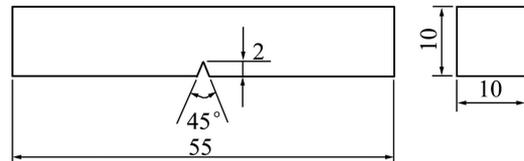
**Fig. 1** Square bar of AZ91D magnesium alloy fabricated by solid recycling process from recycled scraps

Standard tensile specimens with oriented plane parallel to the extrusion direction were prepared to test the tensile strength and elongation to failure. The extruded samples were machined into tensile specimens of 5 mm gauge in diameter and 36 mm gauge in length, as shown in Fig. 2. Tensile tests were performed at a strain rate of  $5 \times 10^{-3} \text{ s}^{-1}$  on WDW-3100 electron universal strength testing machine at room temperature ( $27 \pm 2$ ), ( $-70 \pm 5$ ), ( $-100 \pm 5$ ) and ( $-130 \pm 5$ ) °C, respectively. The tensile axis was parallel to the extrusion direction. The ultimate tensile stress and elongation to failure were recorded. Each datum was the average of results from more than six samples. Impact specimens were machined into 55 mm in length, as shown in Fig. 3. Impact tests were performed on JB30A impact testing machine tester at room temperature ( $27 \pm 2$ ), ( $-70 \pm 5$ ) and ( $-130 \pm 5$ ) °C, respectively. Each datum was also the average of results from more than six samples.

Microstructures were observed by using an OLYMPUS-GX71-6230A optical microscope (OM).



**Fig. 2** Schematic drawing of standard tensile specimens of AZ91D magnesium alloy (unit: mm)



**Fig. 3** Schematic drawing of impact specimens of AZ91D magnesium alloy (unit: mm)

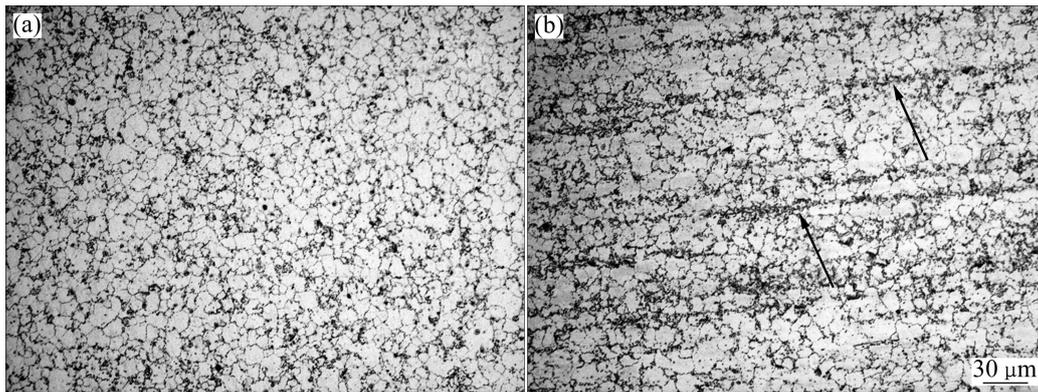
Specimens were polished with a  $\text{Cr}_2\text{O}_3$  polishing medium and etched in a 4% aqueous nitric acid solution. Fractures were characterized on a FEI-SIRION scanning electron microscope (SEM). Grain size was determined using a linear intercept method from a large number of nonoverlapping measurements [6].

## 3 Results and discussion

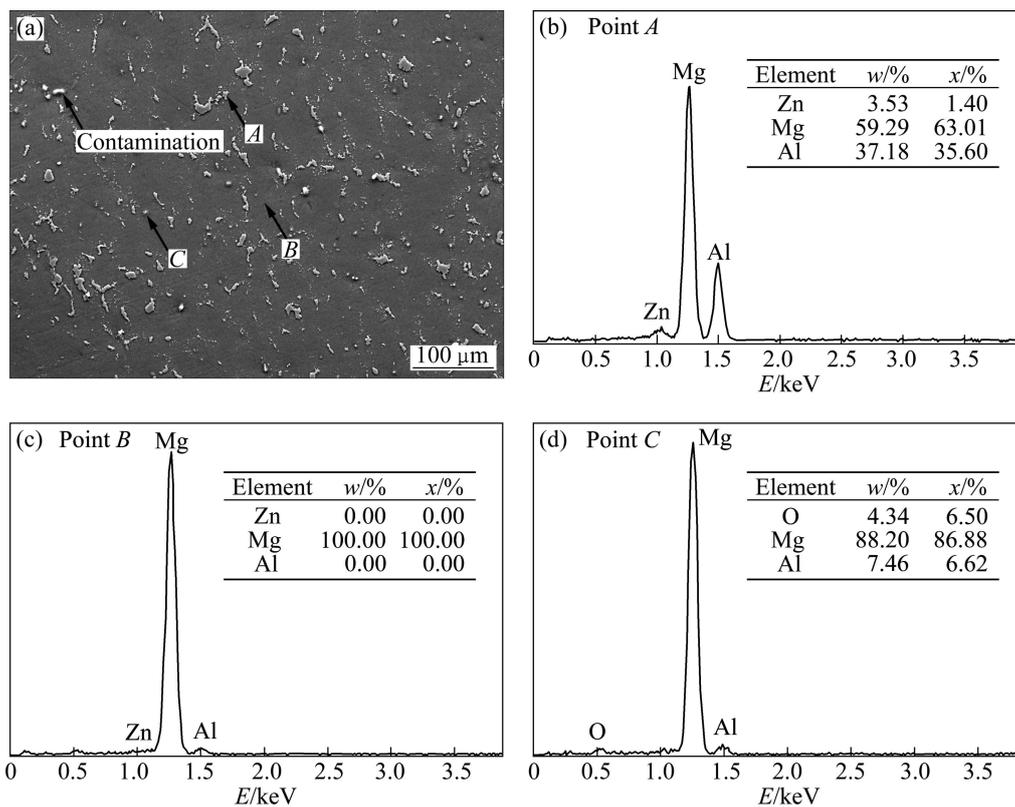
### 3.1 Metallurgical microstructure

At room temperature, magnesium alloys deform essentially by basal slip and twinning, which limits their formability [16]. Hot press and hot extrusion produce additional prismatic and pyramidal slip systems contributing significantly to deformation. Metallurgical microstructures of AZ91D magnesium alloy fabricated by solid recycling process from recycled scraps are shown in Fig. 4. The recycled AZ91D magnesium alloy consists of fine grains due to dynamic recrystallization. None of the specimens exhibit voids or cracks. Individual scraps are not clearly observed and the features of the deformation processing can be seen, as shown in the black arrow (Fig. 4(b)). Some black particles can be seen in Fig. 4(a). These particles are contaminations or oxide precipitates from the scrap surfaces, as shown in Fig. 5.

Magnesium alloy scraps are prone to form a compact oxide layer since magnesium alloys are readily oxidized. But oxide layers can prohibit the fine bonding between the scraps in the hot press and hot extrusion process. As shown in Fig. 4 and Fig. 5, none of the specimens exhibit voids or cracks and individual scraps are not clearly observed. That is to say, hot extrusion involving high compressive and shear forces at high temperature is thus used for producing fine consolidation



**Fig. 4** Microstructures of AZ91D magnesium alloy fabricated from recycled scraps: (a) Perpendicular to extrusion direction; (b) Parallel to extrusion direction



**Fig. 5** SEM image (a) and EDS analysis (b)–(d) of AZ91D magnesium alloy fabricated from recycled scraps

of the recycled scraps. An equiaxed microstructure with an average grain size of 14  $\mu\text{m}$  presents and these grains form high angle boundaries, as shown in Fig. 4(a). Few grains do not happen to recrystallize but exhibit an elongated fiber-like structure, as shown in Fig. 4(b).

### 3.2 Mechanical properties

Figures 6 and Fig. 7 show the ultimate tensile strength and elongation to failure of AZ91D magnesium alloy fabricated from recycled scraps at room temperature and low temperature. Ultimate tensile strength of the specimens increases slightly with

decreasing the tensile temperature. The tensile specimens at  $-130\text{ }^{\circ}\text{C}$  show higher ultimate tensile strength of 360.65 MPa, compared with those at room temperature. Elongation to failure of the specimens decreases with decreasing the tensile temperature. The tensile specimens at  $-130\text{ }^{\circ}\text{C}$  show lower elongation to failure of 5.46%, compared with those at room temperature.

Table 1 lists the impact toughness of AZ91D magnesium alloy specimens fabricated from recycled scraps at room temperature and low temperature. The impact toughness decreases with decreasing the impact temperature. The impact specimens at  $-130\text{ }^{\circ}\text{C}$  show

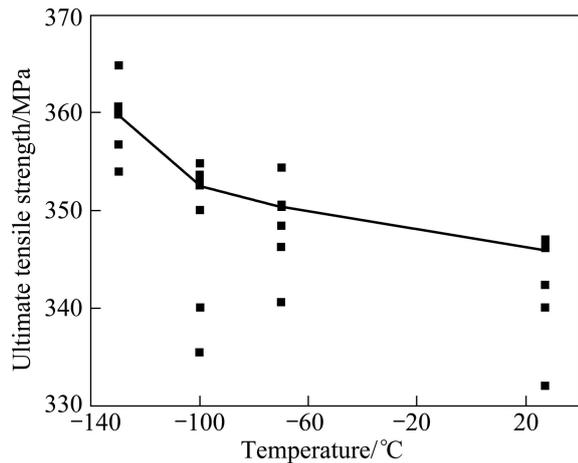


Fig. 6 Ultimate tensile strength of AZ91D magnesium alloy specimens at different temperatures

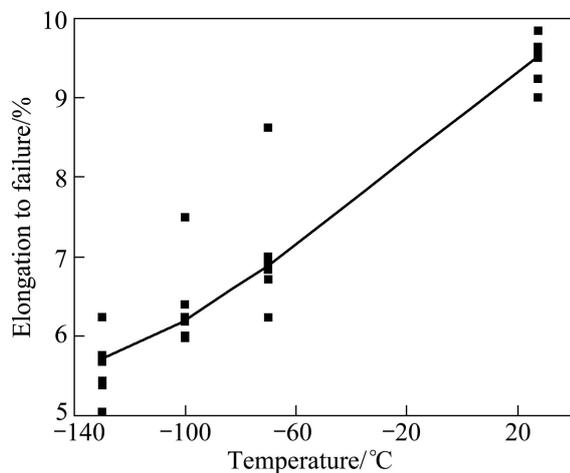


Fig. 7 Elongation to failure of AZ91D magnesium alloy specimens at different temperatures

Table 1 Impact toughness of AZ91D magnesium alloy specimens at different temperatures

Temperature/°C	Impact toughness ( $a_k$ )/(J·cm <sup>-2</sup> )
27	6.13
-70	4.29
-130	3.06

the lowest impact toughness of 3.06 J/cm<sup>2</sup>. The specimens with lower impact toughness are easy to trend the brittle fracture.

### 3.3 Fractography

Figure 8 shows the fractographs of AZ91D magnesium alloy specimens fabricated from recycled scraps at different tensile temperatures. All of the specimens have a similar fracture mechanism of brittle rupture. In the process of room temperature and low temperature tensile test, the specimens exhibit no tendency to “neck”. Crooked tearing edges are observed

between fracture planes, while concavities can be seen on the fracture surface.

As shown in Figs.8(a) and (b) at room temperature, the fracture surfaces are flat and plenty of small dimples present, as shown in the white arrow. This indicates that elongations to failure of the tensile specimens are high.

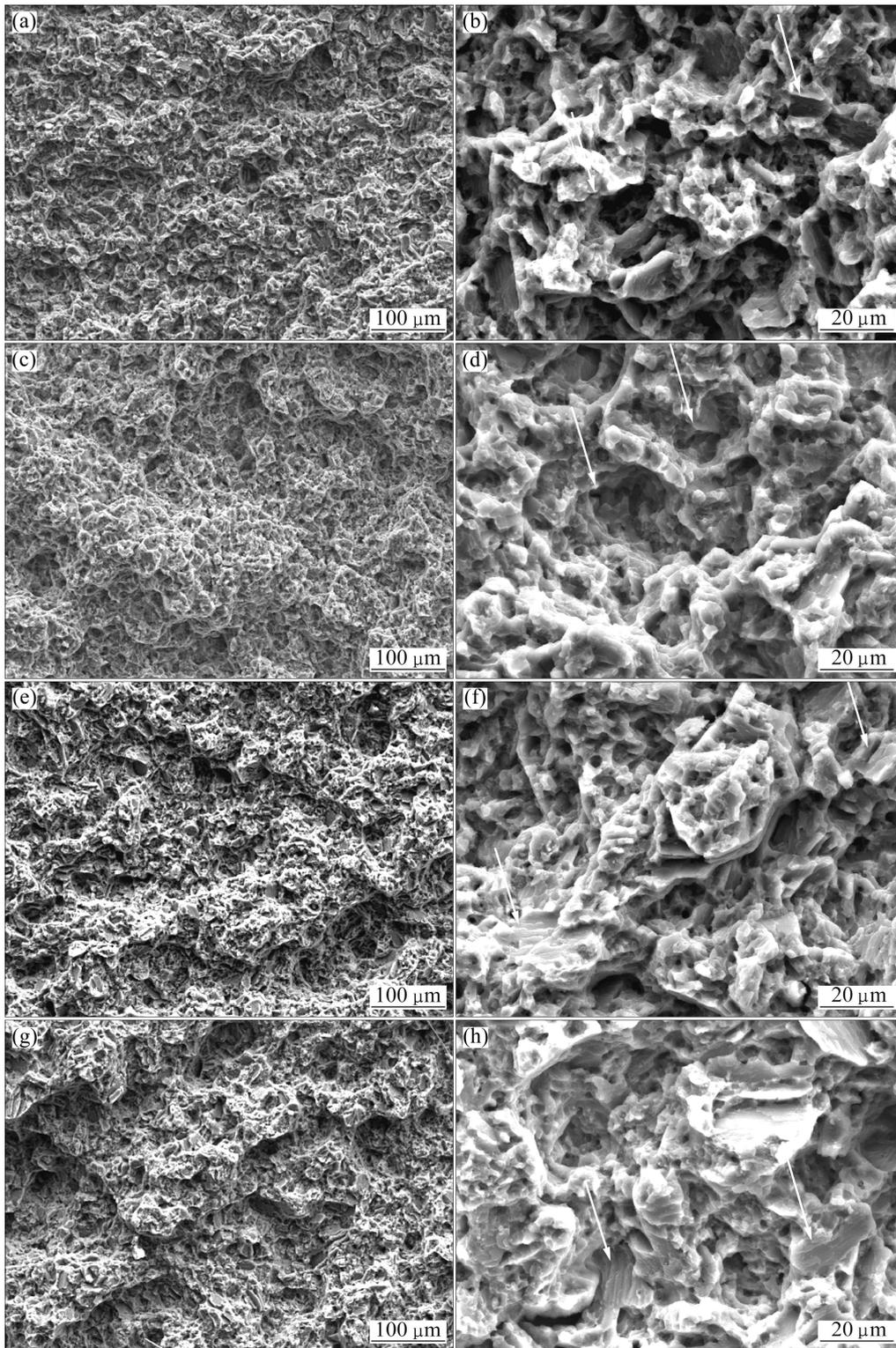
With decreasing the tensile temperature to -70 °C, the average diameters of the dimples increase, as shown in Figs.8(c) and (d). This indicates that elongation to failure of the tensile specimens decreases. With decreasing the tensile temperature to -100 °C, the failure surfaces are composed of some cleavage planes and cleavage steps, as shown in the white arrow in Figs. 8(e) and (f). The plasticity is comparatively worse, which is the typical rupture pattern of magnesium alloy with dense hexagonal crystal. With decreasing the tensile temperature to -130 °C, the failure surfaces are composed of a lot of cleavage planes and cleavage steps, as shown in Figs.8(g) and (h). Overall, the fractures exhibit the characteristics of quasi-cleavage. With increasing the cleavage planes and cleavage steps, elongation to failure decreases. This result is consistent with the above mechanical properties.

Figure 9 shows the impact fractographs of AZ91D magnesium alloy specimens fabricated from recycled scraps at room temperature and -130 °C. As shown in Figs.9(a) and (c), some short crooked-cracks can be seen on the fracture surface (marked by white arrows). These cracks are the bonding interface of the original scraps. During impact test, inconsistent deformation of matrix and oxide precipitates causes stress concentration in the matrix adjacent to the oxide precipitates. And micro-voids or microcracks are prone to form in this area, which could result in premature fracture. As shown in Fig. 9(d) with the white arrow, the failure surfaces are composed of many cleavage planes and cleavage steps. This indicates that impact toughness decreases, compared with those at room temperature. This result is consistent with the above impact toughness.

## 4 Conclusions

1) AZ91D magnesium alloy fabricated by solid recycling process from recycled scraps consists of fine grains due to dynamic recrystallization and the interfaces of original individual scraps are not identified.

2) The recycled specimens of AZ91D magnesium alloy present better mechanical properties. Ultimate tensile strength of the specimens increases slightly with decreasing the tensile temperature. Elongation to failure of the specimens decreases with decreasing the tensile temperature. The tensile specimens at -130 °C show the

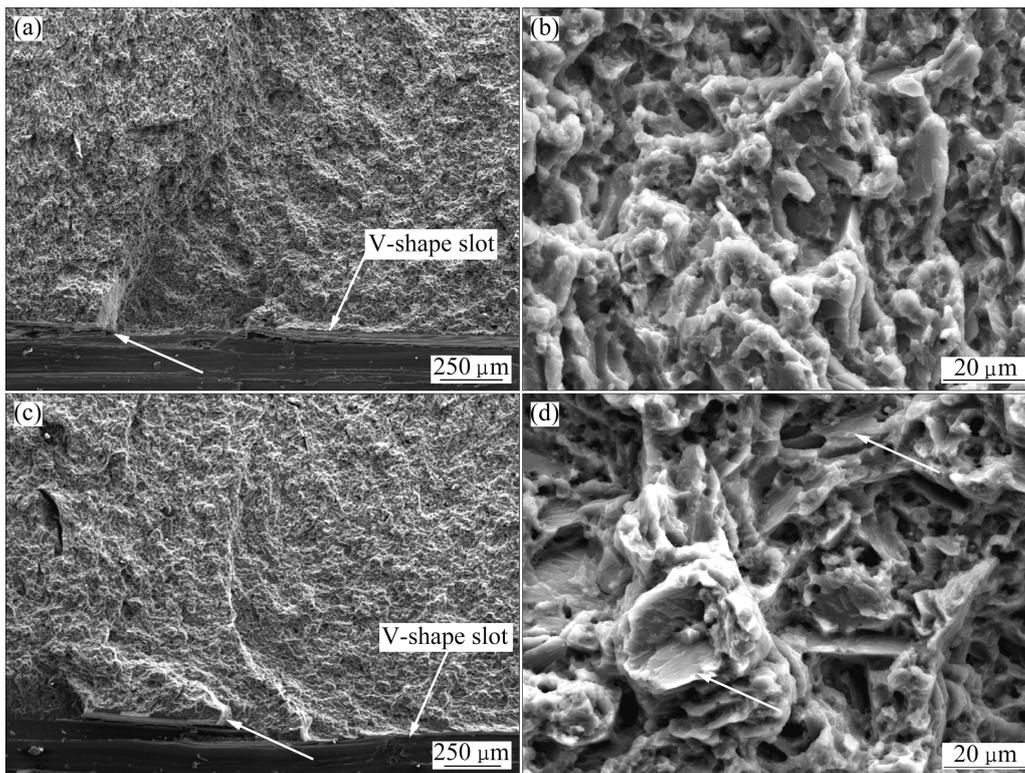


**Fig. 8** Fractographs of AZ91D magnesium alloy specimens at different tensile temperatures: (a) Macro fractograph at room temperature; (b) Micro fractograph at room temperature; (c) Macro fractograph at  $-70\text{ }^{\circ}\text{C}$ ; (d) Micro fractograph at  $-70\text{ }^{\circ}\text{C}$ ; (e) Macro fractograph at  $-100\text{ }^{\circ}\text{C}$ ; (f) Micro fractograph at  $-100\text{ }^{\circ}\text{C}$ ; (g) Macro fractograph at  $-130\text{ }^{\circ}\text{C}$ ; (h) Micro fractograph at  $-130\text{ }^{\circ}\text{C}$

highest ultimate tensile strength of 360.65 MPa and the lowest elongation to failure of 5.46%, compared with those at room temperature.

3) Impact toughness decreases with decreasing the

impact temperature. The impact specimens at  $-130\text{ }^{\circ}\text{C}$  show the lowest impact toughness of  $3.06\text{ J/cm}^2$ . Some short crooked-cracks can be seen on the impact fracture surface.



**Fig. 9** Fractographs of AZ91D magnesium alloy specimens at different impact temperatures: (a) Macro fractograph at room temperature; (b) Micro fractograph at room temperature; (c) Macro fractograph at  $-130\text{ }^{\circ}\text{C}$ ; (d) Micro fractograph at  $-130\text{ }^{\circ}\text{C}$

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## 固相再生 AZ91D 镁合金边角料的低温力学性能

李冬华<sup>1</sup>, 胡茂良<sup>2,3</sup>, 王海波<sup>1</sup>, 赵望安<sup>1</sup>

1. 哈尔滨理工大学 建筑工程学院, 哈尔滨 150080;

2. 哈尔滨理工大学 材料科学与工程学院, 哈尔滨 150040;

3. 上海交通大学 轻合金精密成型国家工程研究中心, 上海 200240

**摘 要:** 采用固相再生方法回收 AZ91D 镁合金边角料, 研究再生合金的低温力学性能、微观组织和断口形貌。在 WDW-3100 型微机控制电子万能试验机上进行低温拉伸实验, 实验温度为 27, -70, -100 和 -130 °C; 在 JB30A 型冲击试验机上进行冲击实验, 温度分别为 27, (-70±5) 和 (-130±5) °C。结果表明: 再生合金在固相再生过程中发生了动态再结晶, 块与块之间结合较好, 原始的块与块之间的界面已经不能分辨。再生合金随着温度的降低, 抗拉强度略有增加, 伸长率呈下降趋势, 即温度降低脆性倾向增加, 在 -130 °C 时拉伸, 抗拉强度和伸长率分别为 360.65 MPa 和 5.46%; 随着冲击温度的降低, 再生合金的冲击功随之降低, 在 -130 °C 时冲击功为 3.06 J/cm<sup>2</sup>。

**关键词:** AZ91D 镁合金; 边角料; 力学性能; 冲击韧性

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