

## Microstructural refinement and mechanical properties of direct extruded ZM21 magnesium alloys

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**Abstract:** Cast ZM21 magnesium alloys were subjected to symmetric extrusion at four different temperatures (200, 250, 300 and 350 °C) with three extrusion ratios of 4:1, 9:1 and 16:1, respectively. The effects of extrusion parameters such as temperature and extrusion ratio were studied by optical microscopy, X-ray diffraction (XRD) and tensile test. The optical micrographs exhibited various stages of recrystallization, i.e., partial to full recrystallization influencing mechanical properties to good extent. Higher extrusion temperature resulted in coarse grains, whereas finer grains were obtained at higher extrusion ratios. Ultimate tensile strength of this alloy was increased from 160 MPa to 316 MPa after extrusion at 250 °C with an extrusion ratio of 9:1.

**Key words:** ZM21 magnesium alloy; grain refinement; recrystallization

### 1 Introduction

Light-weight metallic alloys are preferred in aerospace and automotive sectors because of their high specific strength, energy saving, etc. Though magnesium is the lightest among all, its alloys are used in limited structural applications due to their poor mechanical properties and corrosion resistance [1]. The other limitations of cast magnesium alloys are the formation of coarser grain structure and micro porosity. Mg-Zn system is a medium strength alloy with good forgeability and damping capacity. The Mg-Zn alloy is one of the most promising alloys which have good corrosion resistance due to the formation of metastable phases such as MgZn, MgZn<sub>2</sub> and Mg<sub>2</sub>Zn<sub>3</sub> [2]. In Mg-Zn system, cast ZM21 magnesium is a low-cost alloy with good formability/extrudability [3]. Most of Formula-1 racing car rims are made up of ZM21 Mg alloy [4]. In ZM21 Mg alloy, the formation of the zinc-rich  $\beta$  phase is suppressed by the addition of manganese and the resultant increase in solidus temperature [5]. MAENG et al [2] studied the mechanical properties of the extruded Mg-Zn alloys developed through rapid solidification route and found the increase in hardness due to the extended solid solubility of Zn in Mg matrix. STYCZYNSKI et al [6] discussed the limited room

temperature formability of magnesium alloys attributed to the common anisotropic plastic behavior making the processing steps such as rolling, extrusion. Hot extrusion and thermo-mechanical processing (TMP) improve the formability of magnesium alloys. Extrusion processing is used for obtaining fine-grained materials in which the extrusion parameters like extrusion rate and extrusion ratio are extremely important. Forming of magnesium alloys at a temperature below 200 °C is considered unproductive due to the insufficient number of activated gliding systems and should be avoided in conventional technical forming processes [7–8]. MANN et al [9] found the little asymmetry of Hall-Petch grain-size strengthening parameter when tensile and compression test is performed on the cast Mg-2 Zn alloy. EL MEHTEDI et al [10] explained the high temperature workability of AZ31 and ZM21 alloys compared with their mechanical properties and optical structures. EL-MORSY et al [11] identified the fine grain microstructures and better tensile properties of commercial magnesium alloy AZ61 processed by a combination of hot extrusion and thermo-mechanical processing (TMP). BARNETT et al [12] measured the influence of the grain size on the extruded Mg-3Al-1Zn during compression test between ambient temperature and 200 °C with different initial grain sizes varying between 3 and 23  $\mu\text{m}$  by altering the extrusion

conditions. In their observation, twinning dominated the deformation at lower temperatures but this gave way to slip dominated flow when the temperature was raised. Even at intermediate temperatures, similar transition was observed when the grain size was reduced. AKHTAR and TEGHTSOONIAN [13] described that a range of texture and texture sharpness can arise during uniaxial extrusion, depending upon the choice of alloys and processing conditions.

This work deals with the structural evolution during extrusion of ZM21 Mg alloy at different temperatures with different extrusion ratios (ERs) and their influence on mechanical properties.

## 2 Experimental

Cast ZM21 magnesium alloy was chosen as the base material in the present investigation and its chemical composition is given in Table 1.

**Table 1** Chemical composition of ZM21 Mg alloy (mass fraction)

Zn	Mn	Fe	Ni	Mg
1.7	0.8	0.04	0.08	Bal.

Specimens with size of 30 mm in diameter and 30 mm in height (aspect ratio of 1:1) were prepared from the cast block for extrusion. The specimen was inserted into the specially designed die after applying the lubricant to the die. Oil based graphite was used as the lubricant for extrusion. Both die and specimen were heated in a die heating furnace at a specified temperature followed by 180 min of soaking and then pressure was applied with the help of 100 t hydraulic press to extrude the same. The extrusion speed was maintained at a constant of 0.3 mm/s for all samples. Die was made up of high carbon-high chromium steel. Four different temperatures, 200, 250, 300 and 350 °C and three extrusion ratios, 4:1, 9:1 and 16:1 were chosen to study the effect of temperature and extrusion ratio on microstructure and mechanical properties of ZM21 Mg alloy. The extruded samples were characterized by optical microscopy and also subjected to mechanical testing to know tensile strength and ductility. Oxalic acid based etchant was used for revealing microstructure. Tensile test was carried out in Hounsfield tensometer and hardness measurement was carried out in Zwick 3212001 Vickers hardness tester. Fracture mode of tensile tested samples was evaluated by scanning electron microscopy (JEOL JSM 840). To understand the crystallographic orientation of cast and extruded alloys, X-ray diffraction study was conducted by using Shimadzu diffractometer.

## 3 Results and discussion

The variation in breakthrough pressure with different extrusion temperatures for extrusion ratio of 4:1 is listed in Table 2. The breakthrough pressure decreases with increasing extrusion temperature. As temperature increases, the flow stress of the material decreases. The specimen can be extruded at a lower breakthrough pressure when the extrusion temperature is raised by 50 °C, i.e. from 200 to 250 °C, and the breakthrough pressure is reduced by 40%. However, when the extrusion temperature is increased by another 50 °C, i.e. from 250 to 300 °C, the breakthrough pressure is reduced by only 16% and the similar trend is observed for raising the extrusion temperature from 300 to 350 °C. This indicates that at a low temperature the formability of magnesium is poor due to strong crystallographic basal textures.

**Table 2** Variation in breakthrough pressure with extrusion ratio of 4:1 at different temperatures

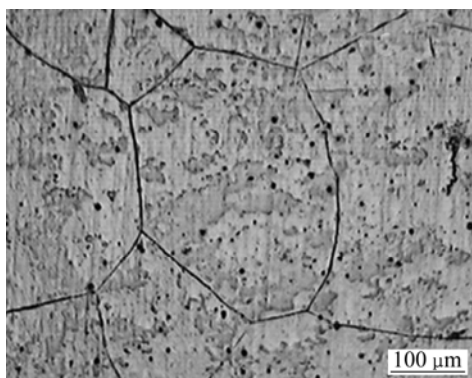
No.	Extrusion temperature/°C	Extrusion ratio	Breakthrough pressure/MPa
1	200	4:1	370
2	250	4:1	220
3	300	4:1	185
4	350	4:1	160

The variation in breakthrough pressure with different ERs at an extrusion temperature of 350 °C is listed in Table 3. The breakthrough pressure increases with increasing extrusion ratio. When the specimen is extruded with a higher extrusion ratio, it undergoes more reduction in area of cross section, which leads to increase in the pressure. The raise in breakthrough pressure is around 16% to 17% when the extrusion ratio is increased from 4:1 to 9:1. Therefore, temperature and extrusion ratio have an influence on materials flow during direct extrusion.

**Table 3** Variation in breakthrough pressure with different extrusion ratios at extrusion temperature of 350 °C

No.	Extrusion ratio	Breakthrough pressure/MPa
1	4:1	160
2	9:1	185
3	16:1	240

Figure 1 shows an optical micrograph of as-cast ZM21 magnesium alloy. The micrograph exhibits single phase  $\alpha$ -Mg with coarser grain structure. The linear intercepted grain size of as-cast alloy is around 400  $\mu\text{m}$ .



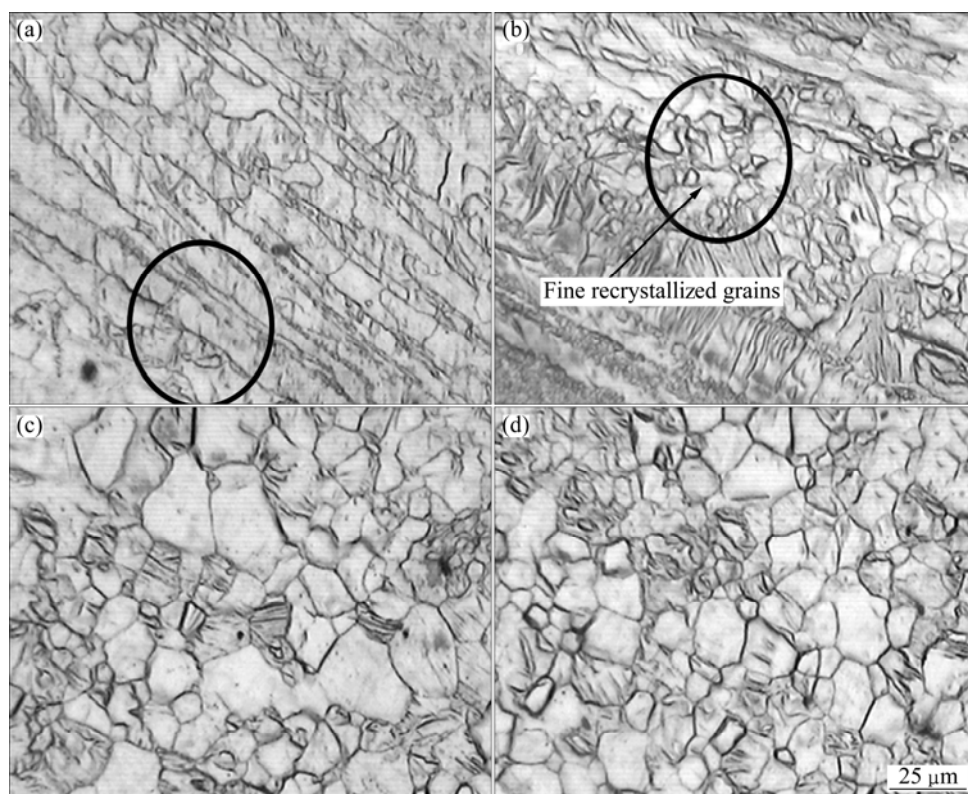
**Fig. 1** Optical micrograph of as-cast ZM21 magnesium alloy

The tensile strength of as-cast alloy is 160 MPa with an elongation of 9%.

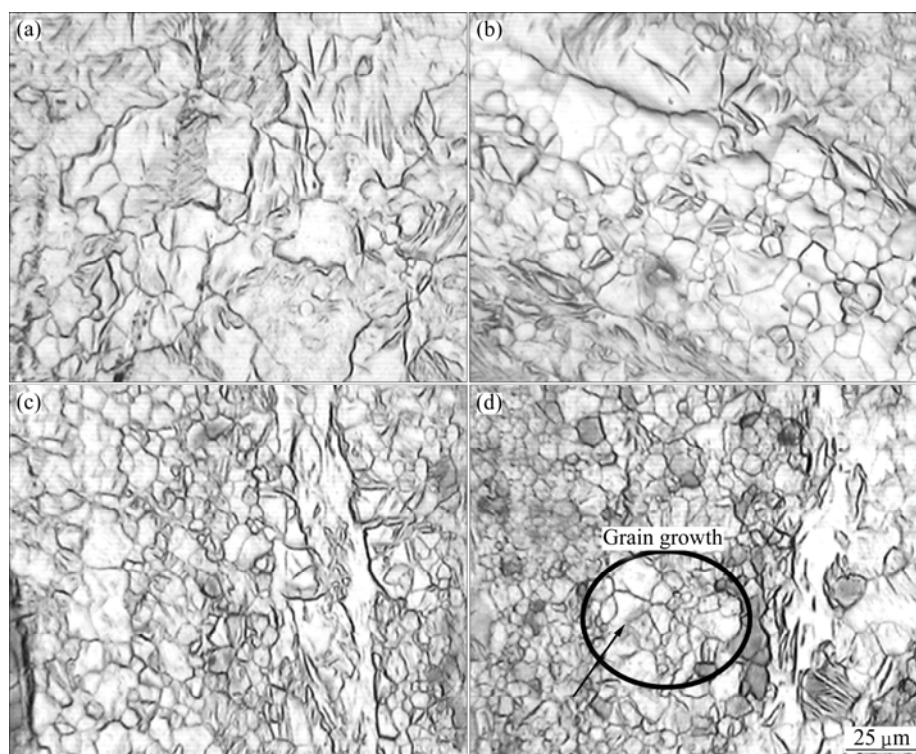
Optical micrographs of alloys extruded with an extrusion ratio of 4:1 at different temperatures are shown in Fig. 2. The alloy extruded at 200 °C exhibits very fine recrystallized grains adjacent to the large elongated grains. This microstructure is due to the partial dynamic recrystallization (DRX) during the extrusion of coarse grain ingot (Fig. 2(a)). The DRX is predominantly observed along the grain boundaries. The recrystallized grains are grown greatly through the migration of grain boundaries during hot deformation process. At this temperature, the deformation twins are also observed which are responsible for comparatively high tensile

strength. On further deformation, the DRX is observed within the grains. It is due to increase in the dislocation density during hot extrusion. The grain refinement causes an increase in strength of the material, necessitating high pressures for further deformation. The alloy extruded at 250 °C exhibits bimodal grains (Fig. 2(b)). The presence of smaller grains of about 5 μm is possibly due to the localized internal energy buildup during deformation at 250 °C, resulting in dynamic recrystallization. The reduction in volume fraction of deformation twins is observed at a higher temperature. When the extrusion temperature is raised to 350 °C, a higher rate of dynamic recrystallization is observed (Fig. 2(d)).

The grains are much larger and almost all fine grains and twins boundaries have been consumed by fast recrystallization and grain growth. The dynamic recrystallization process is normally classified into either continuous or discontinuous recrystallization. In general, continuous recrystallization is considered a recovery process where progressive increase in boundary mis-orientation and the conversion of low angle boundaries into the high angle boundaries may be realized. Discontinuous recrystallization involves nucleation of new grains typically at pre-existing grain boundaries. Accordingly, it has been deduced that discontinuous recrystallization is predominant at 200 °C and 250 °C in the present study. Figure 3 exhibits optical



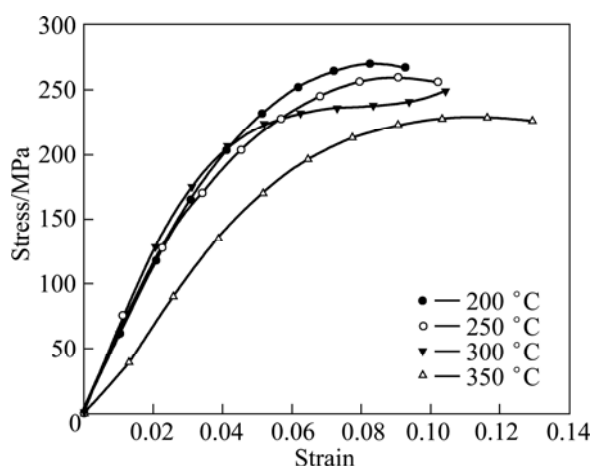
**Fig. 2** Optical micrographs of ZM21 Mg alloys extruded with extrusion ratio of 4:1 at different temperatures: (a) 200 °C; (b) 250 °C; (c) 300 °C; (d) 350 °C



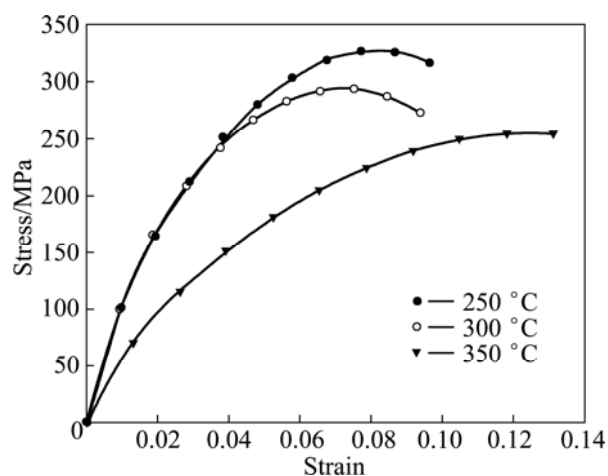
**Fig. 3** Optical micrographs of ZM21 Mg alloys extruded with different extrusion ratios at different temperatures: (a) 250 °C, 9:1; (b) 300 °C, 9:1; (c) 350 °C, 9:1; (d) 350 °C, 16:1

micrographs of ZM21 Mg alloys extruded at 350 °C with two different ERs, namely, 9:1 and 16:1. The grain size is decreased with increase in ER. For the sample extruded at 350 °C with 16:1 ER, the average grain size is reduced to ~20 μm (Fig. 3(d)).

The tensile curves of extruded ZM21 Mg alloy are presented in Figs. 4–6. The cast alloy without extrusion shows poor strength, whereas the extruded alloy exhibits improvement both in strength and ductility. The effect of extrusion temperature on particular extrusion ratio is better understood by Figs. 4 and 5. The effect of extrusion ratio on particular temperature is compared in Fig. 6.

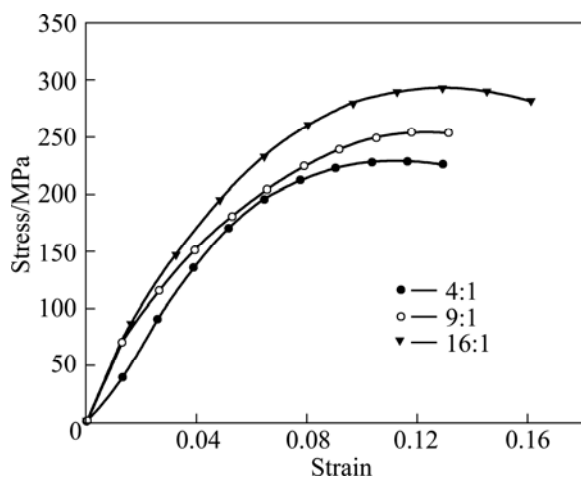


**Fig. 4** Stress—strain curves of alloys extruded at different temperatures with extrusion ratio of 4:1



**Fig. 5** Stress—strain curves of alloys extruded at different temperatures with extrusion ratio of 9:1

The ultimate tensile strength (UTS) of the alloy extruded with 9:1 ER is 316 MPa, exhibiting 98% improvement in UTS after extrusion at an extrusion temperature of 250 °C compared with the cast alloy (160 MPa). As the extrusion temperature increases irrespective of the extrusion ratio, the UTS value decreases with improvement in ductility. When the alloy is extruded with 4:1 ER at a temperature of 350 °C, the UTS decreases to 22%, but the elongation is increased considerably compared with the sample with the same ER and extrusion temperature of 200 °C. The same trend



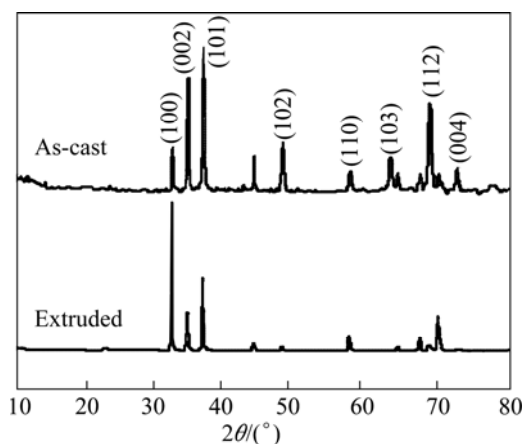
**Fig. 6** Stress—strain curves of alloys extruded with different extrusion ratios at 350 °C

is observed for the alloy with 9:1 ER. This is in line with optical micrographs which reveal increase in grain size due to fast recrystallization and its growth. Also, according to well-known Hall-Petch relation, the strength depends on the grain size. The maximum ductility of 16% is observed for this ZM21 Mg alloy with 16:1 ER at an extrusion temperature of 350 °C. Increase in UTS with concomitant loss of ductility is observed with increasing ER at a given temperature. The same sample exhibits 77% improvement in elongation, which is higher than as-cast alloy (9%). The enhanced dynamic recovery and recrystallization play a significant role in raising ductility. The combination of higher strength and ductility is also attributed to the crystallographic

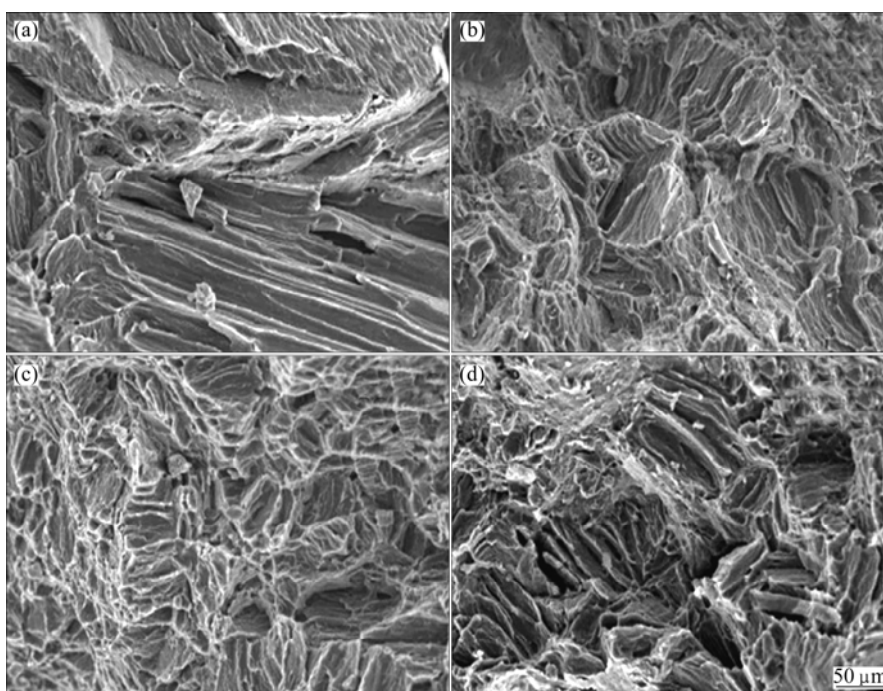
direction of the alloy. Figure 7 shows the XRD patterns of as-cast and extruded alloys (4:1 ratio at 300 °C). The grain orientation of as-cast alloy is multidirectional; however, (101) and (002) are dominant planes. After extrusion, (100) plane becomes dominant. This plane orientation may offer better strengthening to Mg alloy.

The hardness of as-cast ZM21 Mg alloy is HV 40. It is increased with increasing ER and decreased with increasing extrusion temperature. The maximum hardness of HV 96, i.e. 148% of improvement is observed for 9:1 ER at an extrusion temperature of 250 °C.

Figure 8 exhibits SEM fractographs of tensile fractured ZM21 Mg alloy in as-cast and extruded conditions. As-cast fractograph reveals transgranular



**Fig. 7** XRD patterns of as-cast and extruded (4:1 ER at 300 °C) magnesium alloys



**Fig. 8** Fractographs of as-cast ZM21 Mg alloy (a) and extruded alloys at 350 °C with extrusion ratio of 4:1 (b), 9:1 (c) and 16:1 (d)

feature which is cleavage type of fracture. This type of fracture occurs through cleave of the crystals along crystallographic planes. These cleavage facets which are relatively large indicate that large grained structure is evident from the optical micrograph. Quasi cleavage type of fracture, which blends cleavage facets with areas of dimple rupture, is the characteristic feature of SEM fractographs of extruded magnesium alloy. Due to the fine grain structure, the size of the facets is also reduced. These fractographs also exhibit river type of pattern which is formed when the cleavage fracture is forced to reinitiate at the boundary of a grain in a different orientation. These tear ridges tend to merge in the direction of crack growth. Fine to medium ductile dimples are also seen which occur from micro void emergence in place of high local plastic deformation.

## 4 Conclusions

1) During extrusion, for the constant extrusion ratio (ER) the breakthrough pressure decreases with increasing extrusion temperature; at a given extrusion temperature, the breakthrough pressure increases with increasing ER.

2) Extrusion temperature and extrusion ratio (ER) have great influence on the microstructure. Fine recrystallized grains adjacent to coarse unrecrystallized grains are observed at an extrusion temperature 200 °C and 4:1 ER, which is due to the partial dynamic recrystallization. ZM21 alloy starts its recrystallisation during extrusion even at such low temperature (200 °C).

3) Higher extrusion temperature and ER lead to higher rate of dynamic recrystallization and grain growth, which decreases the ultimate tensile strength (UTS) of alloy and increases its ductility (elongation).

4) The highest UTS is obtained when ZM21 alloy is extruded at 250 °C with 9:1 ER, which is 316 MPa, with about 10% elongation.

5) SEM fractographs of unrecrystallized grains after extrusion show quasi cleavage fracture mode, while the recrystallized grains show cleavage fracture mode. Reduced size of cleavage facets indicates the grain refinement.

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# 直接挤压 ZM21 镁合金的组织细化和力学性能

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**摘要:** 对铸态 ZM21 镁合金在不同温度(200, 250, 300 和 350 °C)与不同挤压比(4:1, 9:1, 16:1)下进行挤压。借助光学显微镜、X 射线衍射仪(XRD)和拉伸试验来研究挤压参数(温度和挤压比)的影响。挤压钛合金的光学显微组织呈现出不同阶段的再结晶组织,从部分到完全再结晶,进而影响到合金的力学性能。较高的挤压温度会导致生成粗大的晶粒,然而,较高的挤压比导致细小的晶粒。在 250 °C、挤压比 9:1 下挤压后,合金的极限拉伸强度从 160 MPa 增加到 316 MPa。

**关键词:** ZM21 镁合金; 晶粒细化; 重结晶

(Edited by YANG Hua)