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Microstructure and mechanical properties of Al−6Zn−2.5Mg−1.8Cu alloy prepared by squeeze casting and solid hot extrusion

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Abstract: Al−6Zn−2.5Mg−1.8Cu alloy ingots were prepared by squeeze casting under different specific pressures, and the fresh ingot with best mechanical properties was solid hot extruded. With the increase of the specific pressure from 0 to 250 MPa, the dendrites became round and small. Because the applied pressure increased the solid solubility of alloying elements, the number of $MgZn_2$ phases decreased. When the specific pressure increased from 250 MPa to 350 MPa, the grain size increased. After solid hot extrusion, the α(Al) grains were refined obviously and the $MgZn_2$ phases were uniformly dispersed in the microstructure. After solid hot extrusion, the ultimate tensile strength was 605.67 MPa and the elongation was 8.1%, which were improved about 32.22% and 15.71%, respectively, compared with those of the metal mold casting alloy. The fracture modes of the billet prepared by the metal mold casting and by squeeze casting were intergranular and quasi-cleavage fractures, respectively, whereas, that of the solid hot extrusion was mainly dimple fracture. The refined crystalline strengthening was the main reason to improve the strength and elongation of alloy.

Key words: AlZn alloy; squeeze casting; solid hot extrusion; dynamic recrystallization; microstructure; mechanical properties

1 Introduction

Al−Zn−Mg−Cu alloy is commonly used in aviation industry as a high-strength aluminum alloy, owing to its high specific strength, low density, excellent mechanical properties and so on [1−3]. The 7075 alloy is the typical Al−Zn−Mg−Cu alloy, which is not suitable for the conventional casting because of the serious segregation, strong dendrite growth tendency, coarse microstructure, casting defects and large internal stress [4−6]. Compared with the sand casting, squeezing casting can improve the microstructure and eliminate the casting defects, which is easy to achieve good property [7]. FAN et al [8] found that the α(Al) grain became round with the increase of test pressure, the grain size decreased and the dentrite almost disappeared in the study of effects of pressure and dual refiner on microstructure of the squeeze casting hollow Al−Zn−Mg−Cu alloy drive shaft. Nevertheless, plastic flow is limited during squeezing casting and there is no obvious plastic deformation, which impedes its application. During the solid hot extrusion, both the plastic deformation and the dynamic recrystallization will occur, which generate not only the texture hardening, but also the refined crystalline strengthening [9,10]. Grain refinement significantly improves the mechanical properties of aluminum alloy. Therefore, many researches were carried out to study the behavior of the recrystallization [11−13], LI et al [14] investigated effects of the temperature on microstructure evolution of 7075 aluminum alloy in process of hot deformation. XU et al [15] studied the effects of temperature and strain rate on mechanical properties and microstructure of 7075 aluminum alloy during hot deformation [15]. WANG et al [16] investigated high temperature rheological behavior and neural network constitutive model of as-extrude 7075 aluminum alloy. YANG et al [17,18] further improved the model of the thermal deformation on dynamic recrystallization. This means that the dynamic recrystallization has a great influence on microstructures and properties of alloy. Exactly as HUANG et al [19] found, the main softening mechanism

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during deformation of the alloy at high temperature is dynamic recovery and subgrain size decreases with the increase of Zener−Hollomon parameters [19]. YAN et al [20] found that the mechanical properties get better when the volume fraction of recrystallization grains decreases and the volume fraction of recrystallization grains has a minimum value, appropriately 45%, and the sample exhibits the highest strength and elongation at the deformation temperature of 400 °C.

In this study, two-step forming process was put forward, which was solid hot extrusion after squeeze casting of Al alloy, the aim is to refine the microstructure and improve the mechanical propertiess, especially the elongation. Al alloy ingots were prepared by squeeze casting under different specific pressure, and the effects of the specific pressure on the microstructure and the mechanical properties at room temperature were studied. The fresh ingot with best mechanical properties was solid hot extruded, the microstructure was studied and the mechanical properties were tested. The mechanism of squeeze casting and solid hot extrusion improving the microstructure and the mechanical properties was discussed and revealed.

2 Experimental

The nominal composition of the alloy is listed in Table 1, it is simplified as Al−6Zn−2.5Mg−1.8Cu. The melting of the alloy is as follows: Firstly, the graphite or magnesia crucible was heated to 500 °C, and then the pure aluminum ingot preheated to 180 °C was put in it; Secondly, the melting temperature was increased to 730 °C, the master alloys and pure alloys were put into the crucible in turn after the aluminum ingots were melted completely, where the master alloys are Al−50Cu, Al−10Mn, Al−5Ti, Al−10Cr and the pure alloys are zinc and magnesium alloy; Thirdly, the powder of C_2Cl_6 with the mass fraction of 0.3% was mixed into the melt when all alloys were melted completely in order to remove the impurities; Fourthly, the powder of K_2TiF_6 with the mass fraction of 1% was put into the melt for refining.

Table 1 Nominal composition of Al−Zn−Mg−Cu alloy (mass fraction, %)

Al	Zn	Mg	Cu	Mn	Cr	Ti	Others
88.7	6	2.5	1.8	0.3	0.2	0.2	0.3

The cylindrical billets with the size of *d*128 mm × 100 mm were formed by squeeze casting in vertical extruder (YH61−500G). Specific pressures used in the experiment were 0, 170, 250, 300 and 350 MPa, respectively, where the casting procedure can be regarded as metal mould casting when the specific pressure is 0 MPa. Pouring temperature was 720 °C and dwell time was 50 s. Preheating temperature of the mould was 300 °C. Before solid hot extrusion, the machining was used to remove the surface oxide skin of the billets. The billets were heated to 480 °C and held for 2 h. The temperature of the extrusion chamber and the gasket were preheated to 450 °C and the mould was preheated to 480 °C. Extruding ratio of solid hot extrusion was 42.25:1 and the extruding speed was 0.1 mm/s. Graphite lubricant oil was used during solid hot extrusion. New bars were formed by solid hot extrusion and cooled in air.

Samples with the size of *d*20 mm × 5 mm were cut from the bars, and then ground, polished and etched. The microstructure was observed by scanning electron microscope (Hitachi TM3030 and S3400). Hardness was tested with hardness test machine (HB3000B), where the pressure head was a quenching steel ball with the diameter 2.5 mm and the load was 62.5 kg. Tensile samples were cut from the bar by wire-electrode cutting. The thickness of the tensile samples was 2.5 mm and the detailed size is shown in Fig. 1. Tensile properties test was conducted at room temperature. After tensile test, the fracture samples were put in the alcohol and cleaned by ultrasonic, and the fracture morphology was observed by scanning electron microscope.

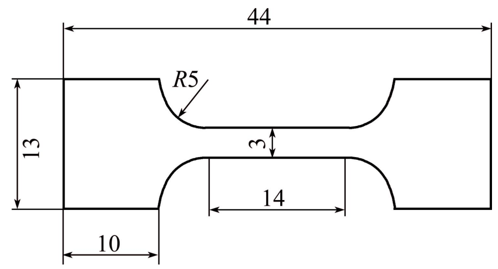

Fig. 1 Size of tensile specimen (unit: mm)

3 Results and discussion

3.1 Microstructure of Al−6Zn−2.5Mg−1.8Cu alloy

Microstructures of the Al−6Zn−2.5Mg−1.8Cu alloy prepared by squeezing casting under different specific pressures and solid hot extrusion are shown in Fig. 2. There are obvious thick dendrites in Fig. 2(a). With the increase of the specific pressure from 0 to 250 MPa, the secondary dendrite of *α*(Al) dendrite disappears and the grains become more near round and smaller; when the specific pressure is 250 MPa, the grain size is the smallest. Whereas, when the specific pressure is further increased, the grain size increases a little and the variation degree is very slight. From Figs. 2(a)−(e), it can be found that the grain size is refined obviously by squeeze casting. From Fig. 2(f), the grain is refined greatly, which indicates that solid hot extrusion is an effective method to refine the grains. According to the

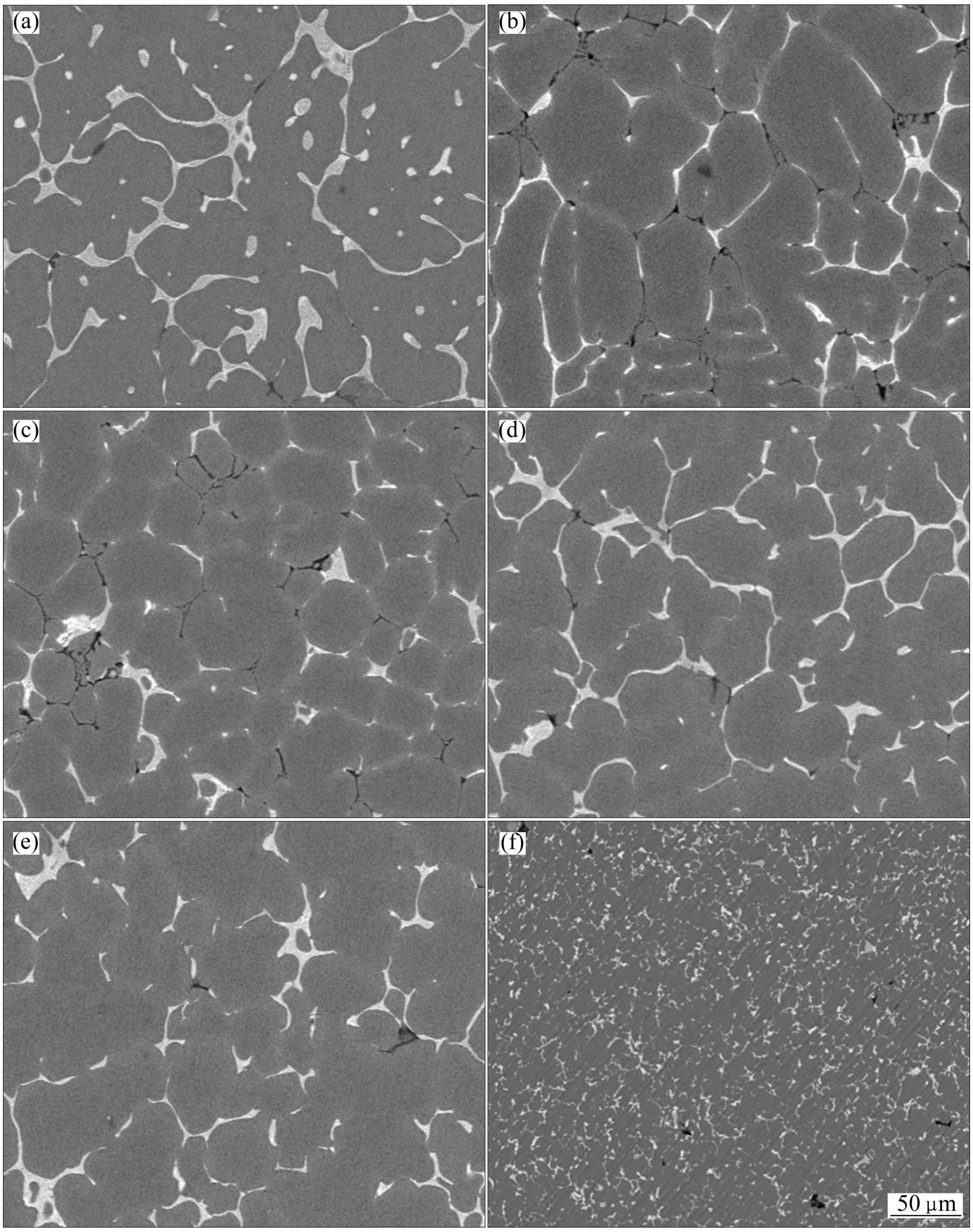

Fig. 2 Microstructures of Al−6Zn−2.5Mg−1.8Cu prepared by squeezing casting with different specific pressures and solid hot extrusion: (a) 0 MPa; (b) 150 MPa; (c) 250 MPa; (d) 300 MPa; (e) 350 MPa; (f) Solid hot extrusion

quantitative metallographic analysis, the equivalent diameters of the grain of these billets are calculated and shown in Fig. 3.

The equivalent diameter of dynamic recrystallization grain is 32.68 μm after solid hot extrusion. According to the model of average grain size of dynamic recrystallization, in DEFORM-3D, it can be described in the following models [21]:

$$d_{\text{rex}} = a_3 d_0^{h_3} \varepsilon^{n_3} \dot{\varepsilon}^{m_3} \exp[Q_3/(RT)] \quad (1)$$

where d_{rex} is the average grain size of dynamic recrystallization, d_0 is initial grain size, $\dot{\varepsilon}$ is the strain rate, ε is dependent variable, R is the gas constant, T is thermodynamic deformation temperature, a_3, h_3, n_3, m_3 and Q_3 are the regression coefficients.

Taking the logarithm on both sides of the formula, Eq. (1) is changed as

$$\ln d_{\text{rex}} = \ln a_3 + h_3 \ln d_0 + n_3 \ln \varepsilon + m_3 \ln \dot{\varepsilon} + Q_3/(RT) \quad (2)$$

To obtain the coefficient in the equation, the regression method was adopted. The grain size (d_0) and the compression were experimentally measured, which are 90 μm and 60% (the true strain is 0.9162), respectively. The calculated results are: $a_3 d_0^{h_3}$=178.46, n_3=0.7961, m_3=−0.06408, Q_3=−10813 J/mol.

The dynamic recrystallization grain size model can be expressed as

$$d_{\text{rex}} = 178.46 \varepsilon^{0.7961} \dot{\varepsilon}^{-0.06408} \exp[-10813/(RT)] \quad (3)$$

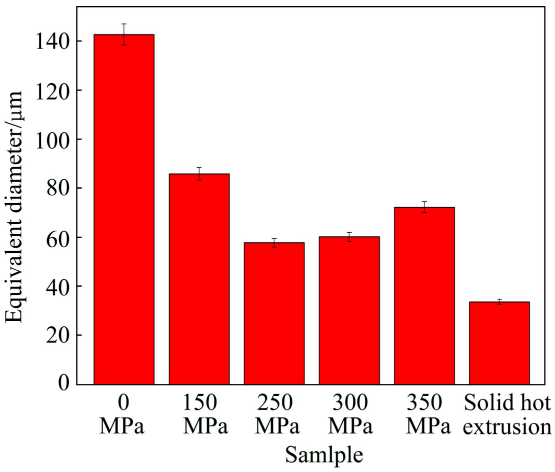

Fig. 3 Equivalent diameter of Al−6Zn−2.5Mg−1.8Cu under different conditions

In this work, the strain rate $\dot{\varepsilon}$ is 0.1 mm/s, the deformation ε is 42.25, the gas constant R is 8.3145 J/(mol·K), the deformation temperature (T) is 450 °C, the calculated dynamic recrystallization grain size (d_{rex}) is 32.08 μm. The calculated results are in well agreement with the experimental results.

The distribution of the eutectic structure and the precipitated phase is shown in Fig. 4. The eutectic structure consists of dense porous T phase and α phase. $MgZn_2$ phases and the eutectic structure distribute in the matrix and grain boundary respectively in Fig. 4(a). The number of $MgZn_2$ phases decreases and the T phase becomes round and small (Fig. 4(b)). The applied pressure will increase the solid solubility of alloying elements [22], and result in more $MgZn_2$ phases dissolved into the matrix. The microstructure of solid hot extrusion alloy is shown in Fig. 4(c). The eutectic structure distributes along the grain boundary after hot deformation and $MgZn_2$ phases appear in the matrix. Dynamic recrystallization process occurs during the solid hot extruding process.

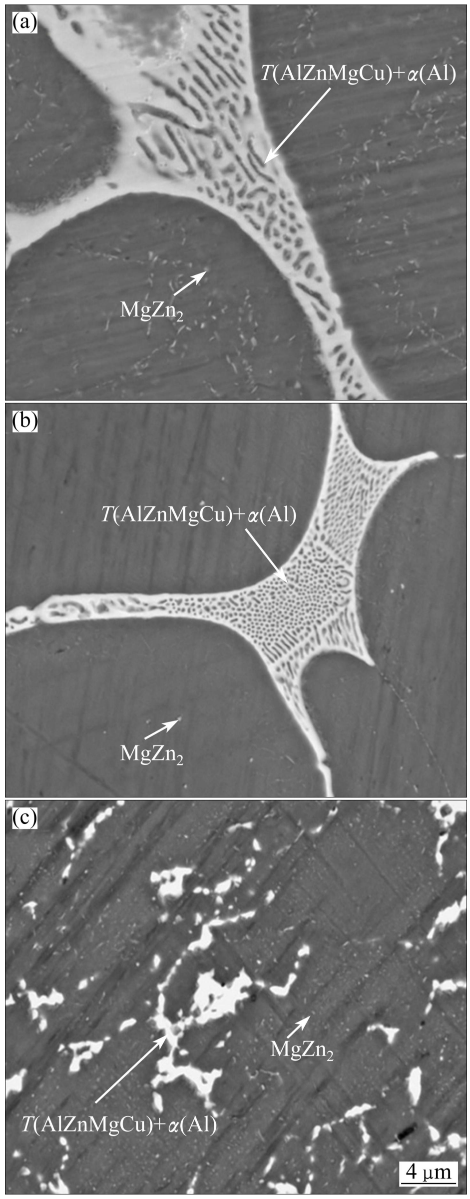

Fig. 4 Microstructures of Al−6Zn−2.5Mg−1.8Cu alloy under different forming processes: (a) Metal mold casting; (b) Squeeze casting; (c) Solid hot extrusion after squeeze casting

3.2 Mechanical properties of Al−6Zn−2.5Mg−1.8Cu alloy

The Brinell hardness of billets prepared by squeeze casting is shown in Fig. 5. From Fig. 5, it can be seen that squeeze casting can improve the Brinell hardness. With the increase of specific pressure, the Brinell hardness is increased from 98 N/mm^2 (specific pressure of 0) to 122 N/mm^2 (specific pressure of 250 MPa). When the specific pressure continues to increase, the Brinell hardness begins to decrease. For comparison, the billets prepared by squeeze casting under 150 MPa and 250 MPa were chosen for subsequent solid hot extrusion. The Brinell hardness of them is shown in Fig. 6. It can be noticed that the Brinell hardness of the billets prepared

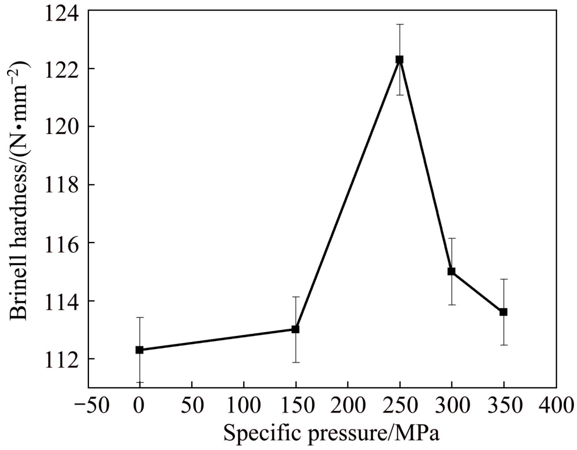

Fig. 5 Brinell hardness of billets by squeeze casting

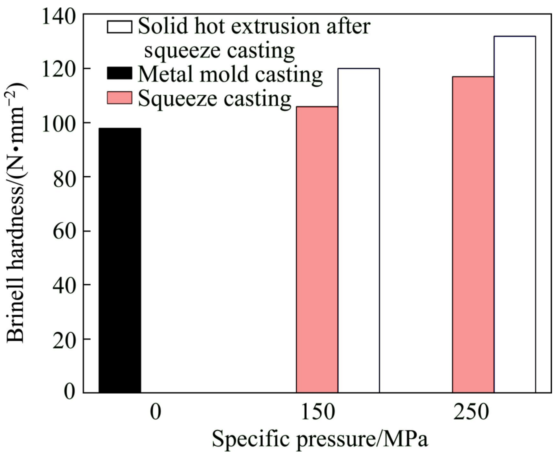

Fig. 6 Brinell hardness of alloy by different forming processes

by solid hot extrusion after squeeze casting is higher than that of squeeze casting, and the Brinell hardness of squeeze casting is higher than that of the metal mold casting. From the Brinell hardness of the billets prepared under the pressures of 150 MPa and 250 MPa, it can be concluded that the higher the mechanical properties of squeezing casting billets, the higher the mechanical properties of solid hot extruding bars.

The specific pressure will affect equilibrium phase diagram. The effect can be described through the Clausius−Clapeyron equation [5]. According to the equation, the solidification point of the alloy generally increases with the increase of the specific pressure. Under the same casting temperature, the undercooling is higher under high specific pressure and refines the grain size. Under the specific pressure of 250 MPa, there is a higher degree of grain refinement. FRANKLIN and DAS [23] found that the applied pressure causes a greater undercooling during the solidification process, which increases the nucleation rate and refines grain size. CHADWICK and YUE [24] obtained that the specific pressure can effectively affect the heat transfer coefficient and contact area between the solidified shell and the mold, which increases the solidification rate. When the specific pressure increases from 250 MPa to 350 MPa, the nucleation rate decreases and then the Brinell hardness decreases.

Mechanical properties of the ingots prepared with graphite crucible were tested, but the results are not as high as expected. We melted this alloy with a new magnesia crucible.

Tensile properties of the billets prepared by the metal mold casting, squeeze casting and solid hot extrusion are shown in Table 2. The tensile properties of the billets prepared by solid hot extrusion after squeeze casting are higher than those of squeeze casting and the metal mold casting, the ultimate tensile strength is 605.67 MPa and the elongation is approximately 8.1%. Comparing the results of squeeze casting with those of metal mold casting, the ultimate tensile strength increases from 458.05 MPa to 585.12 MPa. Grain refinement increases the area of grain boundary, which results in the dislocation movement more difficult, and then improves the mechanical properties. The solid hot extrusion refines the grain size effectively and the refined crystalline strengthening improves the tensile strength and elongation of the alloy. This tensile property is different from that of Ref. [25]. In Ref. [25], the alloy was melt with an old graphite crucible, which contaminated the alloy. In this work, this alloy was melted with new magnesia crucible and the crucible contamination was almost eliminated. Although the values are different, the tendency of the processing influencing the tensile properties is similar.

Table 2 Tensile properties of billets prepared by different processes

Process	σ_b/MPa	$\sigma_{0.2}$/MPa	δ/%
Metal mold casting	458.05	424.16	7.0
Squeeze casting(250 MPa)	585.12	543.05	6.8
Solid hot extrusion after squeeze casting(250 MPa)	605.67	585.14	8.1

3.3 Fracture morphology of Al−6Zn−2.5Mg−1.8Cu alloy

The fracture morphologies of the alloys by different forming processes are shown in Fig. 7. The fracture mode of metal mold casting is the intergranular fracture, which seriously decreases the mechanical properties, as shown in Fig. 7(a). The fracture mode of squeeze casting is quasi-cleavage fracture and the fracture surface has obvious quasi-cleavage flats, as shown in Fig. 7(b). The aluminium alloy is the face-centered cubic crystal with a lot of sliding systems and there is almost no cleavage fracture. The small flat connects with each other by tearing way, and the tearing edges can be clearly observed. After the solid hot extrusion, the fracture mode is dimple fracture in Fig. 7(c). Dimples are shallow and small, and the residual precipitated phases are found in the center of dimples.

The micro cracks are eliminated after squeeze casting, which will improve the mechanical properties. The morphologies of micro cracks are marked in Fig. 7(a). Because the solidification temperature range is larger, the specific pressure increases the flow of liquid metal and fills the space in the dendrite during squeeze casting, which eliminates the casting defects to improve mechanical properties [26]. The grain size affects the fracture morphology. The smaller the grain size is, the more the dimples form. The edge of the dimple is elongated with the shape of parabola, which forms in the tear stress. The opening direction of the parabola is the maximum stress direction.

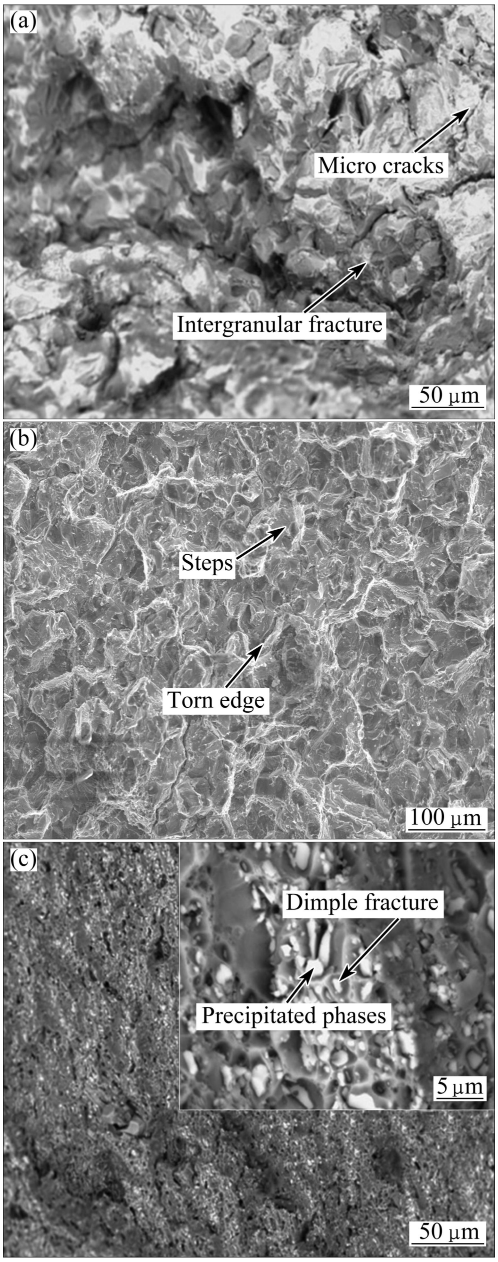

Fig. 7 Fracture morphologies of Al−6Zn−2.5Mg−1.8Cu alloy: (a) Metal mold casting; (b) Squeeze casting; (c) Solid hot extrusion after squeeze casting

4 Conclusions

1) The specific pressure increases from 0 to 250 MPa, and the dendrites become round and small. The applied pressure increases the undercooling and the nucleation rates, eventually refines the grain size. The applied pressure increases the solid solubility of alloying elements, resulting in the decrease of $MgZn_2$ phases. The specific pressure increases from 250 to 350 MPa, and the grain size increases with decreasing the nucleation rate. After solid hot extrusion, the α(Al) grains are refined obviously and the $MgZn_2$ phases are uniformly dispersed. During the solid hot extrusion, the continuous dynamic recrystallization occurs and the grain size is refined.

2) When the specific pressure increases from 0 to 250 MPa, the Brinell hardness is increased from 98 to 122 N/mm^2. When the specific pressure increases from 250 to 350 MPa, the Brinell hardness is decreased from 122 to 113.6 N/mm^2. After the solid hot extrusion, the ultimate tensile strength is 605.67 MPa and the elongation is 8.1%. After the squeeze casting, the ultimate strength is 585.12 MPa and the elongation is 6.8%.

3) The fracture modes of the billet prepared by the metal mold casting and by squeeze casting are intergranular and quasi-cleavage fracture, respectively, whereas, that of the solid hot extrusion is mainly dimple fracture. The refined crystalline strengthening is the main reason to improve the strength and the elongation.

4) The processing of solid hot extrusion after the squeeze casting is an effective method to improve the strength and the elongation of alminium alloys.

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挤压铸造和固态热挤压 Al−6Zn−2.5Mg−1.8Cu 合金的显微组织和力学性能

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摘 要：利用挤压铸造技术，在不同比压下制备 Al−6Zn−2.5Mg−1.8Cu 合金坯料，然后对性能最好的挤压铸造坯料进行固态热挤压。结果表明：比压从 0 增加到 250 MPa 时，树枝晶变得细小而圆整。由于外加压力增加了合金元素的固溶度，因此，$MgZn_2$ 相数量减少。当比压从 250 MPa 增加到 350 MPa 时，合金的晶粒尺寸变大。固态热挤压后，α(Al) 树枝晶被明显细化，并且 $MgZn_2$ 相均匀弥散地分布在合金的显微组织中。固态热挤压后，合金的极限抗拉强度为 605.67 MPa，伸长率为 8.1%。与金属型铸造合金相比，抗拉强度增加了 32.22%，伸长率增加了 15.71%。金属型铸造和挤压铸造的断裂方式分别为沿晶断裂和准解理断裂。然而，挤压铸造成形后固态热挤压工艺的合金断裂方式为韧窝断裂。细晶强化作用是合金抗拉强度和伸长率提高的主要原因。

关键词：铝锌合金；挤压铸造；固态热挤压；动态再结晶；显微组织；力学性能

(Edited by Yun-bin HE)