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# Microstructure characteristics and mechanical properties of rheoformed wrought aluminum alloy 2024

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**Abstract:** The microstructure characteristics and mechanical properties of 2024 wrought aluminum alloy produced by a new rheoforming technique under as-cast and optimized heat treatment conditions were investigated. The present rheoforming combined the independently developed rheocasting process, named as LSPSF (low superheat pouring with a shear field) process, and the existing squeeze casting process. The experimental results show that LSPSF can be used to prepare sound semi-solid slurry within 25 s to fully meet the production rate of squeeze casting. The primary  $\alpha$ (Al) presents in mean equivalent diameter of 69 µm and shape factor of 0.76, and features zero-entrapped eutectics. Compared with conventional squeeze casting, the present LSPSF rheoforming can improve the microstructures and mechanical properties. An optimized heat treatment results in substantial reduction of microsegregation and significant improvement of mechanical properties, such as yield strength of 321 MPa, ultimate tensile strength of 428 MPa and elongation of 12%.

Key words: semi-solid; rheoforming; wrought aluminum alloy; microstructure; mechanical properties

## **1** Introduction

Through more than 30 years of development, semi-solid metal(SSM) processing has been successfully established as a unique technique for production of metallic components with high integrity and improved mechanical properties, which can be further divided into rheoforming and thixoforming[1-3]. Recently, many researchers have discovered that thixoforming has some disadvantages to be spread out into casting industry because of the complicated process and high cost associated with the feedstock[3-5]. Thus, increasing attention is paid to the rheo-route, in which a liquid alloy is transformed into semi-solid slurry followed directly by component shaping[5-7]. These works are mainly focused on two sorts of technologies. One is the technology to produce semi-solid slurry. The other is the technology to deform directly the semi-solid slurry into required near-net shape products. In recent years, several novel rheoforming processes have been developed, including the new rheocasting (NRC<sup>TM</sup>)[6], the twin-screw rheomoulding[7–8], the semi-solid rheocasting  $(SSR^{TM})[9]$ , the sub-liquidus casting  $(SLC^{\circledast})[10]$ , the continuous rheoconversion process (CRP)[11], the SEED[12], and the H-CNM[13], etc. These rheoforming processes combine high pressure diecasting, rolling or squeeze casting to directly deform the semi-solid slurry. In essence, all of the above mentioned processes are based on the same fundamental concept to produce semi-solid slurry: controlled nucleation and limited growth to achieve the semi-solid structure as the alloy melt is cooled below the liquidus temperature. However, there are some limitations to overcome for mass production, such as high production cost, low productivity, and limited alloy systems that can be used.

An advanced rheocasting process to produce sound semi-solid slurries, named as LSPSF (low superheat pouring with a shear field)[14–17] has been developed at Nanchang University, China. The LSPSF process can handle a large range of casting and wrought alloy compositions, e.g. A356, A380, 201, 2024, 6082 and 7075, and is being now scaled up for industry applications. In this study, an important application of the LSPSF to the squeeze casting is presented. The micro-

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structure characteristics and mechanical properties of LSPSF rheoformed 2024 wrought aluminum alloy under as-cast and optimized heat treatment conditions are investigated.

## 2 Experimental

Currently, almost all of researches on rheoforming are mainly focused on commonly used casting alloys such as A356, A357 and AZ91D, which provide high fluidity and castability. Although these alloys are capable of meeting most product requirements, they do not exhibit the strength and ductility achieved with wrought alloys. One of objectives of this work is to spread the advantages of rheoforming with wrought alloy. The experimental alloy used was a commercial wrought aluminum alloy 2024, which has a wide solidification range with solidus and liquidus of 503  $^{\circ}$ C and 638  $^{\circ}$ C, respectively. Its nominal chemical compositions are Cu 4.4, Mg 1.6, Si 0.3, Fe 0.4, Mn 0.6, Cr 0.1, Zn 0.2 (mass fraction, %) and balance Al. The ingots were supplied in the form of extruded rods of 69 mm in diameter.

The LSPSF rheoforming process innovatively adapted the well-developed LSPSF to the task of in situ fabrication of semi-solid slurry with fine and spherical particles. This was followed by the direct shaping of the produced semi-solid slurry into a component using the existing direct squeeze casting process. The LSPSF rheoforming equipment consisted of two basic functional units, a SLPSF slurry-maker and a standard direct squeeze casting machine. No modification to the squeeze casting was required. The LSPSF slurry-maker worked in a batch manner, providing semi-solid slurry within 25 s. When the temperature of semi-solid slurry reached the casting temperature, it was subsequently released into the cavity of the lower die. The upper die (plunger) was moved downwards. The pressure was activated to close off the die cavity and to pressurize the slurry. Further solidification occurred in the die cavity at high pressure. Finally, the plunger was withdrawn and the component was ejected.

The alloy was melted in a graphic crucible using a resistance heating furnace and refined at 750 °C. To obtain sound semi-solid slurry of 2024 alloy, the effects of three important parameters of LSPSF on the semi-solid microstructure were investigated. 1 500 g of the melt alloy was fed into LSPSF slurry-maker at temperatures of 700–680 °C (pouring temperature, PT). The rotation speed of barrel(RSB) was set at 0–120 r/min, the inclined degree of barrel(IDB) was set at 0–45°, the rotational barrel was operated at a temperature of 200 °C, and the temperature of slurry holder was kept at (620±1) °C to utilize a lower cooling. As the temperature of semi-solid slurry held in the slurry holder

reached 628  $^{\circ}$ C, the metallographic samples were taken by sucking a small amount of semi-solid slurry into a quartz tube and then quenched in a salt water pool.

For LSPSF rheoforming, the lower die and the upper die were both preheated to about 300 °C. A typical hot forging lubricant was used on the tool surfaces to facilitate the ejection of component. The speed of the punching upper die, to which the lower die was fixed, was about 200 mm/s, and the compression holding time was about 20 s. A casting temperature of 720 °C was applied in the conventional squeeze casting. In order to determine the applied pressure, casting temperature  $(T_c)$ of semi-solid slurry and the variation of microstructure and mechanical properties of product, the applied pressure was operated between 60 MPa and 90 MPa, and the casting temperature of slurry was changed to 628 °C and 636 °C. Some rheoformed products were treated by the optimized solution treatment and T6. The mechanical properties both before and after heat treatments were measured.

The microstructures of salt water quenched semisolid slurry and rheoformed products at as-cast state and after solution treatment were examined using an optical microscope with quantitative metallography (Zeiss-Axio Imager type). Microsegregation in products was determined by scanning electron microscope(SEM) equipped with energy-dispersive spectroscope(EDS). All metallographic samples were prepared using the standard technique, and etched with Keller's reagent. A closer description of microstructure was given by the grain equivalent diameter  $D=2(S/\pi)^{1/2}$  and shape factor  $F=4\pi S/L^2$ , where S is the area, and L is the perimeter.

## **3 Results**

#### 3.1 Fabrication of semi-solid 2024 alloy

There are three important parameters in LSPSF, including the pouring temperature, the rotation speed of barrel and the inclined degree of barrel. The combination effects of the three parameters on semi-solid microstructure of 2024 alloy were deeply investigated in Ref.[17]. The optimized conditions to produce sound semi-solid 2024 alloy are as follows: the pouring temperature is between 660 °C and 680 °C, the rotation speed of barrel is 90–120 r/min, and the inclined degree of barrel is 20–35°. Under the optimized conditions, LSPSF can prepare sound semi-solid slurry within 25 s, and the typical microstructures are shown in Fig.1. The primary  $\alpha$ (Al) presents in a mean equivalent diameter of 69 µm and shape factor of 0.76, and features zero-entrapped eutectic.

#### 3.2 As-cast microstructures

Fig.2 shows the typical microstructures obtained by



**Fig.1** Microstructures of water quenched semi-solid 2024 alloy rheocast by LSPSF process: (a) RSB 90 r/min, IDB 20°, PT 680 °C; (b) RSB 90 r/min, IDB 20°, PT 660 °C; (c) RSB 120 r/min, IDB 20°, PT 680 °C; (d) RSB 90 r/min, IDB 35°, PT 660 °C



**Fig.2** As-cast microstructures of 2024 castings produced under different processing conditions: (a) Conventional squeeze casting, casting temperature 720 °C, applied pressure 90 MPa; (b) LSPSF rheoforming, casting temperature 636 °C, applied pressure 90 MPa; (c) LSPSF rheoforming, casting temperature 628 °C, applied pressure 60 MPa; (d) LSPSF rheoforming, casting temperature 628 °C, applied pressure 90 MPa

conventional squeeze casting and LSPSF rheofroming. The casting process affects the shape and distribution of present phases. The conventional squeeze cast microstructure shows primary  $\alpha$ (Al) dendrites with the intermetallic compounds consisting of Al<sub>2</sub>Cu and Al<sub>2</sub>CuMg, as shown in Fig.2(a). Fine and spherical primary  $\alpha$ (Al) is typical for the LSPSF rheoformed microstructure and the intermetallic compounds are concentrated in the regions between the primary  $\alpha$ (Al) phases.

Moreover, the microstructures produced by LSPSF rheoforming under different conditions are significantly different. When the casting temperature of semi-solid slurry is higher, the solid fraction of slurry is lower, resulting in relatively more remained liquid. Because the heat extraction ability of the die is very strong, some small irregular particles are formed during further solidification of the remained liquid in the die cavity, as shown in Fig.2(b). In Figs.2(c, d), the distributions of liquid and solid phases at the pressure of 90 MPa are denser than those at the pressure of 60 MPa. And the grain size of primary  $\alpha$ (Al) is also reduced because of the increase of the pressure. The reason is that heat loss between the semi-solid slurry and the die increases because of the increase of the pressure, so the cooling rate increases.

Samples were taken from different sections of the rheoformed components to reveal typical microstructures resulting from deformation and flow of semi-solid slurry. Figs.3(a–e) represent the microstructural variations along cross-section of Fig.3(f). The used casting temperature and applied pressure were 628  $^{\circ}$ C and 90 MPa, respectively.

During LSPSF rheoforming, a pre-specified amount



**Fig.3** Microstructures at different locations of LSPSF rheoformed 2024 casting under conditions of casting temperature of semi-solid slurry 628 °C, applied pressure 90 MPa, and preheating temperature of mold 300 °C: (a) Area A; (b) Area B; (c) Area C; (b) Area D; (e) Area E

of semi-solid slurry is poured into the preheated die cavity. The grains flow with liquid during pouring filling the bottom part of the cup-shaped component (designated E in Fig.3(f)), and there is not much flow near the bottom part, thus the primary grains are largely unchanged, which are nearly the same as those before deformation as shown in Fig.1. As the deformation continues, the semi-solid slurry flows upwards and fills the space between upper die and lower die. At A and B parts of the finished component, the outside wall of upper die and inside wall of lower die are parallel. There is not much pressure in these regions and consequently the shape of primary grains is not changed, but much liquid phase is observed. The liquid phase seems to flow upwards by the pressure of the upper die. In area C, the geometry figure of cup-shaped component changes, and the grains have been elongated along the flow direction. For area D, two types of grains can be found. Some tiny grains between larger particles can be seen and this does not seem to derive directly from the semi-solid slurry produced by LSPSF. The semi-solid slurry at this location might have gone severe deformation, and the fine grains might indicate that recrystallization has occurred while the larger irregular particles might comprise grains pressed/sintered together.

### **3.3 Mechanical properties**

Fig.4 shows the ultimate tensile strength, yield strength and elongation of as-cast specimen. Both the casting temperature of semi-solid slurry and applied pressure significantly affect the mechanical properties of LSPSF rheoformed specimens. As the applied pressure increases, the strength and elongation also increase. These results coincide with the results of KANG et al [18] for experiment using thixoforging. According to the above results, the improved mechanical properties can be obtained, which are higher than those of the conventional squeeze casting, under a suitable combination of applied pressure and casting temperature of semi-solid slurry, under which a denser microstructure can be obtained, as shown in Fig.2.

Table 1 shows a comparison of mechanical properties of 2024 alloy specimens processed by different semi-solid techniques and followed by T6 heat treatment. A large improvement of mechanical properties

of LSPSF rheoformed specimens is observed after T6 heat treatment. Table 1 demonstrates clearly that LSPSF rheoforming offers better strength than indirect squeeze casting and thixoforging techniques applying direct/ indirect squeeze casting, but a lower elongation than that of thixoforging techniques applying indirect squeeze casting. Compared with wrought process, LSPSF rheoforming can obtain similar ultimate strength and elongation, but a lower yield strength.

## **4** Discussion

The essential metallurgical feature of LSPSF is the forced multiple nucleation on the wall of the rotational barrel, which continues to grow in a spherical shape due to the controlled cooling under conditions that induce a stabilization effect on the morphological instability at the solid/liquid interface[15]. It appears that the rotation of



Fig.4 Mechanical properties of 2024 castings produced under various conditions

Table 1 Mechanical properties of 2024 alloy castings obtained from different processing techniques

Processing technique	Heat treatment	Yield strength/MPa	Ultimate strength/MPa	Elongation/%	Reference
LSPSF rheoforming	Т6	321	428	12	This paper
Thixoforging-1	Т6	277	366	9.2	[19]
Thixoforging-2	Т6	236	387	21	[18]
Indirect squeeze casting	Т6	230	420	8	[20]
Wrought	T6	393	476	10	[20]

Thxioforming-1applies direct squeeze casting; Thixoforming-2 applies indirect squeeze casting

barrel can result in a large and ever-renewing surface area between the melt alloy and the barrel wall, which together with a lower superheat pouring is ideal for enhancing the efficiency of heat extraction. According to the separation theory[21] and simultaneous nucleation theory[22], localized rapid cooling, combined with vigorous mixing during the initial stage of solidification, can enhance heterogeneous nucleation, crystal separation and crystal survival, resulting in substantial grain refinement of the semi-solid slurry. For LSPSF, the semi-solid conditions are mainly controlled by pouring temperature, rotation speed and inclined degree of barrel. When the above mentioned three parameters are properly controlled, the outlet temperature of melt alloy (when the melt alloy flows out of the barrel) can be below its liquidus temperature[17], and the excellent semi-solid slurry can be achieved. From experimental observation, there is a large available range for pouring temperature. Compared with NRC<sup>TM</sup>, SSR<sup>TM</sup> and SLC<sup>®</sup> etc, the pouring temperature of the melt alloy in LSPSF process can be increased to a much higher level. These factors indicate that the LSPSF can be more easily controlled and can be used as the core technology to produce sound semi-solid slurry in rheoforming processes.

As the cooling rates are similar in squeeze casting stage, the different microstructures in the as-cast state must be attributed to the difference of material states in the casting. Conventional squeeze casting starts from a superheated melt. The non-equilibrium solidification begins with nucleation of primary  $\alpha$ (Al). Subsequently, dendritic growth occurs and the remaining liquid in the inter-dendritic regions finally solidifies as eutectic consisting of intermetallic compounds. In LSPSF rheoforming, the squeeze casting journey starts from SSM slurry (solid fraction 0.4–0.6), i.e. near equilibrium solidification at solid fraction 0–60% and subsequent non-equilibrium solidification. During forming of semi-solid slurry, the eutectic is concentrated in the remained liquid regions between primary  $\alpha$ (Al) particles. As a result, the intermetallic compounds do not form individual particles in conventional squeeze casting, but form a continuous network instead.

The presence of intermetallic compounds (brittle phase) at grain boundaries results in microsegregation, which significantly affects the mechanical properties of castings[23]. Fig.5 indicates that the degree of microsegregation in LSPSF rheoformed specimen is lower than that of conventional squeeze casting. The good combination of strength and elongation of LSPSF rheoformed samples is mainly derived from the lower microsegregation and the fine/uniform microsegregation still presents in as-rheoformed 2024, as shown Fig.5(b).



**Fig.5** Microsegregation in 2024 alloy under various conditions (applied pressure of 90 MPa): (a) Squeeze cast; (b) Rheoforming; (c) Rheoforming and solution treatment at 495 °C for 16 h; (d) Distribution of Cu

Therefore, heat treatment should be undertaken to reduce microsegregation and to further improve the mechanical properties.

The present liquid segregation (Fig.3) and microsegregation can affect precipitation, including solution heat treatment, and it would be expected that time at solution temperature would be expected to be increased with respect to standard practice, viz. approximate 2 h for the wrought 2024 alloy. In the present work, an optimized T6 for rheoformed 2024 was undertaken, that is 16 h solution at 495 °C followed by water quenching, then ageing for 12 h at 170 °C. Under the optimized solution treatment, most soluble Cu-rich phases such as Al<sub>2</sub>Cu and Al<sub>2</sub>CuMg have been dissolved (Fig.5(c)), and the distribution of element Cu becomes more uniform (Fig.5(d)), indicating a significant reduction of microsegregation, and resulting in the improved mechanical properties that can be close to the levels of wrought 2024 alloy targets. Better mechanical properties were obtained by LSPSF rheoforming than by thixoforging[18]. This may be associated with the high quality semi-solid slurry (fine grain size and zero-entrapped eutectic) produced by LSPSF process and the optimized post-forming T6 heat treatment.

## **5** Conclusions

1) LSPSF process can be used to prepare sound semi-solid slurry of wrought aluminum alloy 2024 within 25 s to fully meet the production rate of squeeze casting process. The primary  $\alpha$ (Al) presents in mean equivalent diameter of 69  $\mu$ m and shape factor of 0.76, and features zero-entrapped eutectic.

2) LSPSF rheoforming can improve the microstructures and mechanical properties. The higher the applied pressure between 60MPa and 90MPa, the more dense the microstructure, resulting in an increase of mechanical properties.

3) Microsegregation exists in LSPSF rheoformed 2024 alloy. Proper solution treatment can markedly reduce the degree of microsegregation. Under an optimized T6 condition, the improved mechanical properties of LSPSF rheoformed 2024 alloy can be close to the levels of wrought 2024 alloy targets.

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