PHYSICAL METALLURGY OF MULTISTAGE RSP Al–Li BASED ALLOYS①

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ABSTRST

Based on series author's study of Al–Li alloys in recent years, the formation theory of powders, characteristics, powder compaction, the microstructures and the mechanical behaviors of RSP Al–Li based alloys are analyzed and discussed in this paper

Key words: Rapid Solidification Al–Li alloy, Powder metallurgy, microstructure, mechanical property.

1 INTRODUCTION

Combined with traditional powder metallurgy, rapidly solidified processing (RSP) has opened a new way for further improving the mechanical properties of Al–Li alloys. The composition of RSP Al–Li alloy, such as Li, Zr, can be selected in a wider range because the solidification of the melt is very fast and is far away from the equilibrium crystallization process. Compared with ingot metallurgy (IM) Al–Li alloy, RSP Al–Li alloys exhibit higher specific strength and modulus. However, powders prepared by RSP are very fine, which are easily oxidized and easily absorb gasses. After compaction, original powder particle boundaries (PPB) and a quantity of grain boundaries in the alloy play an important role in the subsequent processing and then affect the deformation and fracture of the alloy[1]. In order to deepen the understanding of physical metallurgy features in this kind of alloys and seek the method to further improve the ductility of the alloys, the authors have provided some research results of recent years and analyzed some physical metallurgy aspects of RSP Al–Li alloys in this paper.

2 FORMATION THEORY OF POWDERS DURING MULTISTAGE RAPIDLY SOLIDIFIED PROCESSING (MS–RSP)

The key of RSP technique is to decrease the solidified melt volume of unit time, increase the heat dissipation surface area of the melt and enhance the heat conductivity rate of intermediate contacted with the melt. The solidification rate of the powder[2] is

$$T = \frac{k\ (T_0 - T)}{\rho \cdot c \cdot r} \qquad (2)$$

where T_0— the melt temperature; T— ambient temperature; ρ— the density of the alloy; c—average specific heat of the alloy; k—heat exchange efficiency, r—radius of the melt drop. It can be seen, the finer the melt drop

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and the lower the temperature of intermediate, the higher the solidification rate of the melt. Therefore, the matter of the cooling rate in RSP technique is turned to how to make the melt dispersive and how to make the tempera-ture of intermediate decrease as much as possible. Obviously, the technique of single crushing melt, such as single gas atomization or single melt spining atomization, can not get the ideal effect. Thus, increasing the crush mechanisms of the melt and further decreasing the temperature of intermediate should be considered. Based on this assumption, a MS-RSP apparatus has been developed[3] (Fig.1). The working principle of this apparatus is to disperse the melt into melt drops using high pressure inert gas at first, then to make the supercooling drop further cracked by copper wheel with a high spinning rate. The time in which melt contacted with the intermediate should be controlled in order to avoid the solidification of the melt drop before cracking. In addition, some coolled liquid jet (no pollution with powder) can also be used to make the melt further dispersive and to cool the copper wheel. The author's study showed that the solidification rate of the melt could be changed, different powder particle size and various ratios of dispersive powder to foil shape powder could be obtained by adjusting atomization parameters, such as diameter of the tundish hole at the bottom of the crucible, the ring gas spacing in the jet nozzle, inert gas pressure and the spinning rate of the copper wheel.

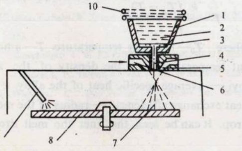

Fig.1 MS–RSP apparatus

3 POWDER CHARACTERISTICS

3.1 *Morphology of the powders*

Powders prepared by MS–RSP technique existed in dispersive and foil shape forms. Dispersive powder appeared as spherical or ellipitcal. Dentrite morphology on the surface of larger particles can be seen under SEM at high magnification (Fig.2a) , this arose from solidification shrinkage of the melt. The surface area of foil shape powders is 0.5–2 cm^2 and their thickness is 0.1–0.2 mm; the

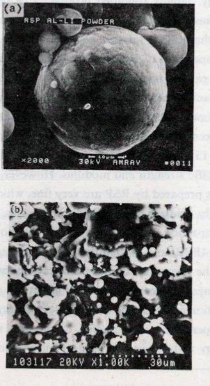

Fig.2 Morphology of MS-RSP Powders

particle surface close to copper wheel is smooth, but on the opposite surface, tiny particles are piled up (Fig.2b). Foil shape powder were essentially composed of jet-deposition of many quasi-solidified melt drop with different size at inert atmosphere.

3.2 *Microstructures within powder particles*

After powder are inlaid, polished and etched, crystallization feature (Fig.3) within powder particles can be seen under SEM.

The solidification process of the melt and the crystallization features varied with the melt drop: for particles with diameters smaller than 50 μm there was a featureless shell with different thickness. The finer the particle, the thicker the shell, dentrite and cellular structure in the center of the particles appeared in turn (Fig.3a–3c). For particles with diameters smaller than 4 μm, dentrite and cellular structure within the particles disappeared and the microstructure within the particles appeared featureless (Fig.3d).

In order to further understand the micro structure of the featureless area and then analyse their solidification process, tiny powder particls were put under TEM, it was found that the featureless area was composed of

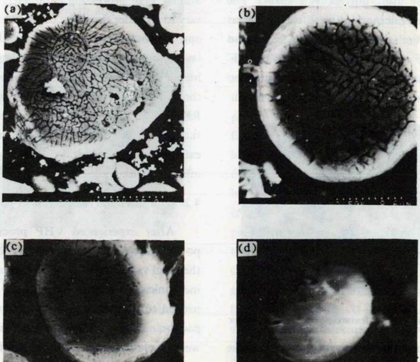

Fig.3 Crystallization features within powders with different particle sizes

micrograins with the sizes of about 0.5 μm (Fig.4) . The formation of micrograins was correlated with a high nucleation rate, a high growth velocity of the solid-liquid interface and intense heat exchange between the melt drop and the cooling intermediate. The melt corresponding to the featureless area could get quite high supercooling before solidfication. In this case, nucleation rate was very high, the melt would be solidified as distribution free form. Therefore, featureless areas can not exhibit composition contrast under SEM. Different from the second electron image on SEM, central bright field (BF) image on TEM is an image mainly using diffraction contrast. The orientation of micrograins in featureless areas was different relative to the electron beam; and the extent of satisfied Bragg diffraction condition was also different, so microgains morpho logy could be observed on TEM.

Fig.4 microstructure of featureless area within powder

In addition, by measuring second dentrite arm spacing (DAS) within powder particles, the relation between particle size (d) and DAS could be obtained. According to the relation between DAS and cooling rate (T) of the melt suggested by Matyia[4]

$$c = DT^a$$

where D is the DAS value; a, c are constants; the cooling rate of the melt achieved using MS–RSP technique was evaluated as 10^5–10^6 k / s.

4 COMPACTION OF POWDER

4.1 *Vacuum hot pressure* (VHP)*compaction*

VHP processing included vacuum heating degassing and VHP compaction. During vacuum heating degassing, the absorbed vapour would be gone. Subsquently, powder were compacted at a given temperature and pressure.

It should be pointed out, while determining VHP processing parameter, it is necessary to consider the effect of vacuum degassing and the compaction extent of powder. Higher temperature can get better degassing effect and higher compaction extent, but it is easy to lose the sub-stable structure of RSP powders. For RSP Al–Li alloy used for engineering structure materials, the effect of degassing and the extent of the compaction of billet should be most important.

4.2 *Hot extrusion compaction of powder billet*

After experienced VHP processing, the powder density can approach 92% of the theorical value. In order to further improve the mechanical propertics of the alloy, the billet needed to be treated with hot extrusion compaction. During extrusion powder particles were shear-deformed and elongated. Oxide film on the surface of particles were crushed and particles were combined by forming new surfaces at connected areas. The study showed[5] that increasing the temperature could decrease the resistance force during extrusion, but if the temperature were too high, the surface quality of extrudate would deteriorate.

Higher extrusing deformation degree favoured the crush of oxide film on powder particles and then benefited the ductility of the alloy.

5 MICROSTRUCTURE OF RSP Al– Li ALLOYS

5.1 *Original powder particle boundariee in the alloy*

After experienced VHP and hot extrusion compaction, original spherical or elliptical particles were elongated and the oxide film on the particle surface were crushed into discontinous tiny particles (Fig.5) .It should be pointed out that good combination between particles is the premise of good strength and ductility of the RSP Al–Li alloy.

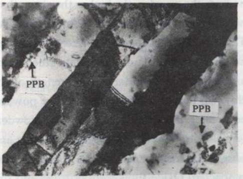

Fig.5 grain structure of the extrudate

5.2 *Grain structure*

Grain structures of the alloy mainly aim at its grain size and grain morphology. Zr played a decisive role in the grain structure of RSP Al–Li alloy.

At rapid solidification, there were minor undisolutioned Al_3Zr particles in the melt before atomization. After atomization, most Zr existed as supersaturation in solid solution. Subsequenty. tiny spherical Al_3Zr particles (10–20 nm) coherent with the matrix were precipitated in VHP process. During extrusing, grain were deformed as bamboo shape due to axial flow of the billet. If recrystallization and grain growth occur within the grains, the movement of grain boundaries should modify the low energy interfaces between Al_3Zr particles and the matrix, which need more energy. Therefore, in adddition to refining the grain size during solidification, Al_3Zr particles also strongly inhibit the recrystallization of RSP Al– Li alloy, and grain morphology still retained the bamboo shape (Fig.5) .

5.3 *Precipitation within the matrix*

Comparison study on aging response of a series of the both IM and RSP Al–Li alloys showed that the precipitates and precipitaion sequences of IM and RSP Al–Li alloy with the same composition were basically the same, the difference was only that the dynamics of the precipitation reaction of RSP Al–Li alloy was faster. During ageing, supersaturated solid solution decomposed and precipitated spherical δ' (Al_3Li) phases. If the aging temperature is higher and the aging time is longer, S' (Al_2CuMg) and T_1(Al_2CuLi) would be also precipitated. Many factors affect the precipitation dynamics of δ' and S' phases, among them the amount of Zr, dislocation substucture and grain size are most important. The authors found that when the Al–Li alloy containing Zr was quenched, dislocation loops would appear around Al_3Zr particles due to the gathering of vacancy (Fig.6) . It is possible that tube diffusion of dislocations made Li easily close to Al_3Zr and form Al_3Li / Al_3Zr compound particles, and then accelerated the precipitation process of δ' phases. We also found that the tiner the grain size, the faster the precipitation process. This phenomena was essentially correlated to the dislocation substructure and the grain boundaries, which promoted the diffusion of Li, Cu, Mg in solid solution. For the Al–Li alloy prepared by RSP, the amount of Zr is higher and the grain size of the alloy is also finer, these two factors all favoured the precipitation of δ' and S′ phases and led to faster aging dynamics.

Fig.6 dislocation loops around Al_3Zr in quenched Al–Li alloys

5.4 *Grain boundary characteristics*

Grain boundary characteristics of Al–Li alloys include the segregation of harmful impurities, the precipitation of equilibrium phases and accompanied δ' precipitate-free zone (PFZ) at grain boundaries, which strongly affect the mechanical properties of the alloy and is a kind of key microstructure factor. For the alloy prepared using MS–RSP the grains were finer (1.5–2.5 μm). Compared with IM Al–Li alloy, the ratio of grain boundaries in the alloy is higher, the concentration of harmful impurities at grain boundaries may expect to be decreased. It was also found that there were equilibrium phases and PFZ at grain boundaries. Furthermore, the amount of equilibrium phases and the width of PFZ increased with aging time[6].

6 MECHANICAL BEHAVIOUR OF THE MS–RSP Al–Li ALLOYS

Table 1 Powder characteristic and tensile properties of the alloy

Powders Characteristic		YS (MPa)	UTS (MPa)	EL (%)
foil	shape	445	512	8.6
+100	mesh	440	500	5.2
−100—+250	mesh	475	525	5.6
−250—+320	mesh	480	535	6.5
−320—+500	mesh	486	542	5.7
−500 目	mesh	490	547	5.1

6.1 *Effect of powder characteristic*

The Effect of powder particle size on mechanical properties of RSP Al–Li alloy can be divided into the effect of both strength and ductility of the alloy. Our study indicated that the finer the powder, the higher the strength of the alloy prepared using this kind of powder (Table 1). This is because the finer the powder, the finer the grain size, the greater the contribution of grain boundaries on strength of the alloy. On the other hand, the finer the powder, the greater the specific surface. The powders were easy to be oxidized during collecting and subsequent treating. While the powders were compacted, Oxide films were crushed and distributed along PPB. Oxides weakened the cohension of PPB and led to the failure of the alloy along PPB. Thus, the beneficial effect of grain refining on the ductility of the alloy was cancelled out or weakened by the harmful effect of the oxides on the surface of the particles. Therefore, the ductility of the alloy prepared using middle powder particle size is better. If using foil shape powder for preparing the alloy, the strength of the alloy was little bit lower but the ductility was obviously improved. This is due to the less specific surface and lower oxide content of foil shape powder.

6.2 *Effect of extrusing texture*

During extrusion, powder billet experienced axial deformation and then formed very strong <111> texture. For fcc metal, <111> is slip plane, <111> texture makes the <111> planes within grains perpendicular to the longitudinal direction of the extrudate (i. e the applied pulling direction) which led to the increase of deformation resistance. Therefore, the extrudates exhibited higher strength and lower ductility. Extrusion-upsetting-extrusion processing can make adequate crush of oxides at PPB, weaken the <111> texture and refine the grain size in the extrudates, then benefit the ductility of the alloy[5].

6.3 *Effect of step aging*

Table2 Tensile properties of RSP Al–Li alloy with different treatment

Processing	YS (MPa)	UTS (MPa)	EL (%)
525℃, 40min, water quenching	236	365	12.8
170℃, 4h preageing	416	480	7.0
190℃, 20h single ageing	460	520	6.4
170℃, 4h+190℃, 18h double ageing	445	512	8.6

Study[7,8] on deformation and fracture mechanism of Al–Li alloy showed that intense slip localization at grain boundaries caused by shearing of δ' (Al_3Li) with dislocation and strain localization along PFZ at grain boundaries are the main causes of grain boundary failure. The author's study showed that step aging was the most effective method to improve the ductility of RSP Al–Li alloys (Table 2).

Kin et al[9] believed that step ageing favouring ductility was correlated with the existence of more compound Al_3Li / Al_3Zr particles. XiaXin[10]suggested that step ageing make S′ (Al_2CuMg) more dispersive. Author's study[11]on single–double ageing of a MS–RSP Al–2.52Li–1.60Cu–1.2Mg–0.22Zr alloy found that suitable step ageing not only made more compound Al_3 Li / Al_3Zr particles but also decreased the equilibrium phases and narrowed the PFZ at grain boundaries. When the alloy is deformed, because the center of compound particles have higher hardness, it is difficult to be sheared by dislocation and intense planar slip does not occur easily, deformation of the alloy is more homogenous. On another hand, narrowing of PFZ weakened the strain localization at grain boundary. Two factors above all favoured improvement of ductility of the alloy.

7 CONCLUSION

(1) The key of RSP technique is to decrease the volume of the melt solidified at unit time, to increase the release-heat surface area and the conduction heat rate of intermediate contacted with the melt. MS–RSP technique can make the melt multiple crush before solidification. The cooling rate can approach 10^5–10^6 K / sec.

(2) The powders prepared by MS–RSP existed in both dispersive and foil shape powder. The later were essentially composed of jet-deposition of quasi-solidified melt drops during atomization in inert atomosphere. Compared with dispersive powder, foil shape powders have less specific surface and less oxidization. The ductility of the alloy prepared using foil shape powders were obvtiously improved.

(3) Extrusion-upsetting-extrusion processing can make the oxides on particle surface adequately crush, produce more new surface at PPB. It can also weaken the <111> extrusion

(*To be continued on page 97*)