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Effect of Solid Solution Treatment on Semisolid Microstructure of Zn-22Al Alloy

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ABSTRACT

The effect of solid solution treatment on semisolid microstructure of Zn-22Al with developed dendrites was investigated. Zn-22Al is a zinc-based alloy with aluminium as its main alloying element. Producing Zn-22Al product by semisolid metal processing (SSM) offers significant advantages, such as reduction of macrosegregations, porosity and low forming efforts. Meanwhile, thermal and microstructure analyses of Zn-22Al alloy were studied using differential scanning calorimeter (DSC) and Olympus optical microscope. Solidus and liquidus of the alloy can be determined by DSC analysis. In addition, changes to the microstructures in response to solid solution treatments were also analyzed. The major effort of all the semi-solid technologies is the generation of small and spherical morphologies. Prior to the generation of spherical morphologies, the fine grains should be first produced. The as-cast samples were isothermally held at 315°C, ranging from 0.5 to 5 hours before they were partially re-melted at semisolid temperature of 438°C to produce solid globular grains structure in liquid matrix. The results indicated that a non-dendritic semisolid microstructure could not be obtained if the traditionally cast Zn-22Al alloy with developed dendrites was directly subjected to partial remelting. After solid solution treatment at 315°C, the black interdendritic eutectics were dissolved and gradually transformed into β structure when the treatment time was increased. The microstructure of the solid solution treated sample changed into a small globular structure with the best shape factor of 0.9 and this corresponded to 40±16µm when the sample was treated for 3 hours, followed by directly partial remelting into its semi solid zone.

Keywords: Semisolid metal processing (SSM), solidus; liquidus; dendritic; spherical morphologies

INTRODUCTION

The initial work that led to the interest in semi-solid metal processing (SSM) could be traced back to the studies by researchers at the Massachusetts Institute of Technology in the early 1970s. This work was originally directed at the problem of hot tearing in alloy castings, but it was later realised that a new technology for near-net shaping of complex shapes had been discovered (Flemings, 1991). This SSM technology can be generally defined as a forming process that shapes metal components in their semi-solid state (Flemings, 1991; Kirkwood, 1994; Omar *et al*., 2005, 2009).

Semi-solid metal forming (SSM) has become a widely accepted metal processing technique because it combines the elements of both casting and forging, offering significant advantages, such as the reduction of macrosegregations, porosity and low forming efforts (Kirkwood, 1994; Chen *et al*., 2002, Omar *et al*., 2005, 2009). Other major advantages include prolonged die life

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due to decreased thermal shock (forging below liquidus as against castings), weight savings in components with less porosity than conventional, plus improved usage of feedstock materials because of improved designs (Omar *et al*., 2009).

Continuous searches have been carried out for alternative materials in engineering applications to reduce the cost of production without sacrificing the functional requirements of the components. Zinc-based alloys are of recent interest in this regard, offering a number of benefits over their conventional counterparts like aluminium casting alloys, copper-based alloys, bearing bronzes and cast iron in various engineering applications. These alloys feature clean, low-temperature, energy-saving melting, excellent castability, as well as high strength and equivalent or often superior bearing and wear properties as compared to standard bronze bearing. Meanwhile, cast Zn–Al (ZA) alloys have been widely used for engineering components, including those in the automotive industry (Abou El-khair *et al*., 2004).

The main requirement for alloys to be shaped in the semi-solid state is that they should exhibit a fine spheroidal or non-dendritic grain structure (Chen *et al*., 2002). From the previous research, non-dendritic semisolid microstructure could not be obtained if the traditional cast alloy with developed dendrites was directly subjected to partial remelting. The alloy must be pretreated, such as by mechanical stirring during solidification, modifying prior to pouring, or subjected to deformation prior to remelting. These pre-treatments not only brought about melt contamination and gas absorption during mechanical stirring or melt contamination from refine, but also needed some special high-cost equipments (e.g. stirring equipment for magnetohydrodynamic stirring and rolling or pressing machine for predeformation) (Chen *et al*., 2006). In this study, the development of non-dendritic microstructures of Zn-22Al was investigated by applying isothermal heat treatments in a single phase area at various holding times, followed by a direct partial remelting into its semi-solid zones.

EXPERIMENTAL PROCEDURES

Materials

Zn-22Al is zinc based metal alloy with the alloying elements of aluminium, magnesium and copper. Adding copper and magnesium was found to increase strength and inhibit intergranular corrosion, respectively. Typical uses are automotive parts, household appliances and fixtures, office and computer equipment, as well as building hardware (Robert, 1984). The chemical composition of the as-cast material is given in Table 1.

TABLE 1
Chemical composition of the Zn-22Al alloy compared to the nominal

Zn-22Al	Chemical composition (wt%)							
	Al	Cu	Mg	Cd	Pb	Fe	Sn	Zn
Nominal	21 – 23	0.4 – 0.6	0.008 – 0.012	0.01 max	0.01 max	0.002 max	0.001 max	balance
Analysis	21.46	0.5	0.004	-	-	-	-	balance

Differential Scanning Analysis (DSC)

DSC analysis was carried out primarily to estimate the solidus, liquidus and liquid fraction within the semi-solid zone of the supplied material. The alloy was cut into small pieces (less than 20 mg) for the DSC test using Mettler Toledo DSC 822e. The heating rate employed is 10°C per minute.

Heating was carried out in a nitrogen atmosphere to prevent oxidation. From the heating curve, phase transformation reactions were also observed.

Solid Solution Treatment and Partial Remelting

Solid solution treatment and partial remelting procedures were carried out using an electric furnace. Zn-22Al samples were solution treated at 315°C in β zone, for a range of holding times from 0.5 to 5 hours. Subsequently, the samples of solution treated were quickly removed from the furnace and water quenched before the partial remelting treatment was carried out at 438°C (i.e. in the semi-solid temperature of Zn-22Al) for 1 hour.

Image Analysis

The microstructural characterisation was carried out using Olympus optical microscope and Philips XL30 scanning electron microscope prior to the analysis using imageJ software. All the samples were etched in a solution of 100ml water + 0.5-5ml HNO_3 (Frank 1991).

RESULTS AND DISCUSSIONS

The equilibrium phase diagram of zinc-aluminum alloy is shown in *Fig. 1*. Zn-22Al alloy with aluminium content of about 22% has a single β phase when it is heated above the eutectoid temperature. The microstructures of the as-cast and solid solution treated Zn-22Al alloy are presented in *Fig. 2*. Meanwhile, the as-cast microstructure of Zn-22Al [shown in *Fig. 2*(a)] consists of developed dendrites and interdendritic eutectics. Solid solution treatment procedure was carried out at 315°C in β zone, with various holding times between 0.5 to 5 hours. The black interdendritic eutectics (mainly A1 rich) shown in *Fig. 2*(a) gradually dissolved into the primary dendrite structures

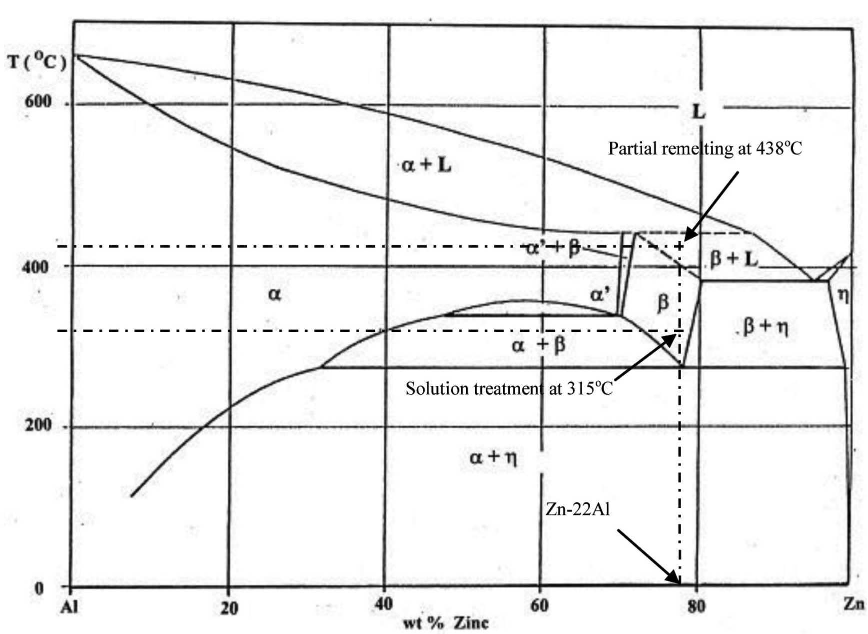

Fig. 1: Binary phase diagram of Zn-Al system (Zhu et al., 1999)

(as shown in *Fig. 2*(b), *2*(c) and *2*(d)) during solid solution treatment. It resulted in a decrease of the eutectic phases which gradually evolved into β phase until no obvious dendritic structures were observed when the treatment time was long enough (*Fig. 2*(d)).

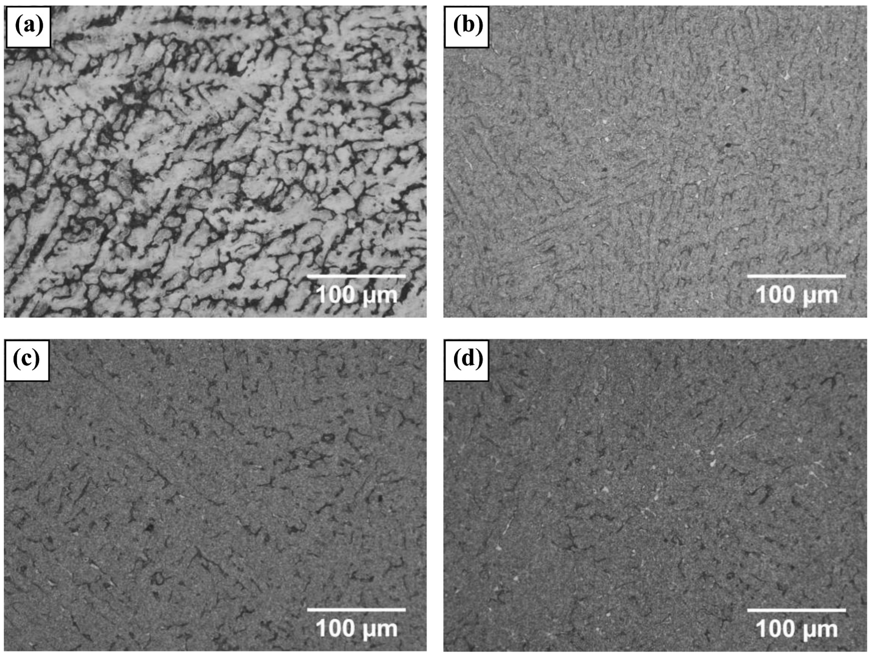

Fig. 2: Microstructures of Zn-22Al (a) as-cast, and after solution treatment for (b) 1.5 hrs, (c) 3 hrs, (d) 5 hrs

The DSC heating curve, together with its corresponding liquid distribution of the as-cast Zn-22Al, is shown in *Fig. 3*. The solidus is estimated at 380°C, and this is at 468°C for the liquidus, while 20% and 50% liquid fractions correspond to 419°C and 438°C, respectively. Note that thixoforming is normally carried out at liquid fraction between 20-50% (Kirkwood, 1994). Hence, when the solution treated Zn-22Al alloy was partially re-melted at 438°C, the corresponding liquid fraction is approximately 50%. *Fig. 4* shows some of the typical semi-solid microstructures of Zn-22Al alloy after the solid solution treatment for different periods. Meanwhile, *Fig. 4*(a) shows the as-cast microstructure after direct partial re-melting. It could be seen that the liquid phase appeared along the grain boundaries as well as entrapped within large solid agglomerates of β phase. When the sample was solution treated at 315°C for 1.5 hours before partially re-melted, however, the number of irregular grains was relatively higher and the size was smaller than the semi-solid microstructure of the re-melted as-cast sample (see *Fig. 4*(b)). Large and irregular structures gradually transformed into a structure of spheroidal particles when the holding time of solid solution treatment was increased to 3 and 5 hours (*Fig. 4*(c) and *Fig. 4* (d), respectively).

The microstructure evolution is focused on the change of shape and size of grains as a function of time in the semi-solid state. To quantitatively represent the shape of grains, shape factor was commonly used to characterize SSM microstructures. Shape factor is defined as $P^2/4\pi A$, where P is the perimeter and A is the area of the particle (shape factor of a circle is equivalent to 1). For optimum semi-solid processing characteristics, the shape factor should be as close to 1 as possible (Legoretta *et al*., 2008). The average size of the primary particles is defined as $[\sum 2(A_i/\pi)^{1/2}]/N$, where A_i is the area of each particle and N is the total particle numbers in each image (Chen *et al*., 2008). It can be observed that the shape factor has changed to be as close to 1 and the particle size has gradually reduced with an increase in the treatment time.

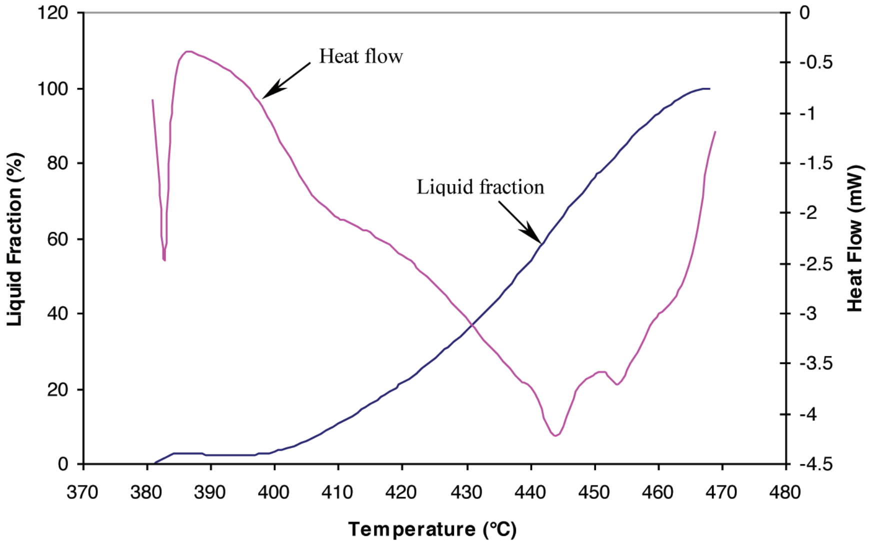

Fig. 3: DSC and liquid fraction curve of Zn-22Al

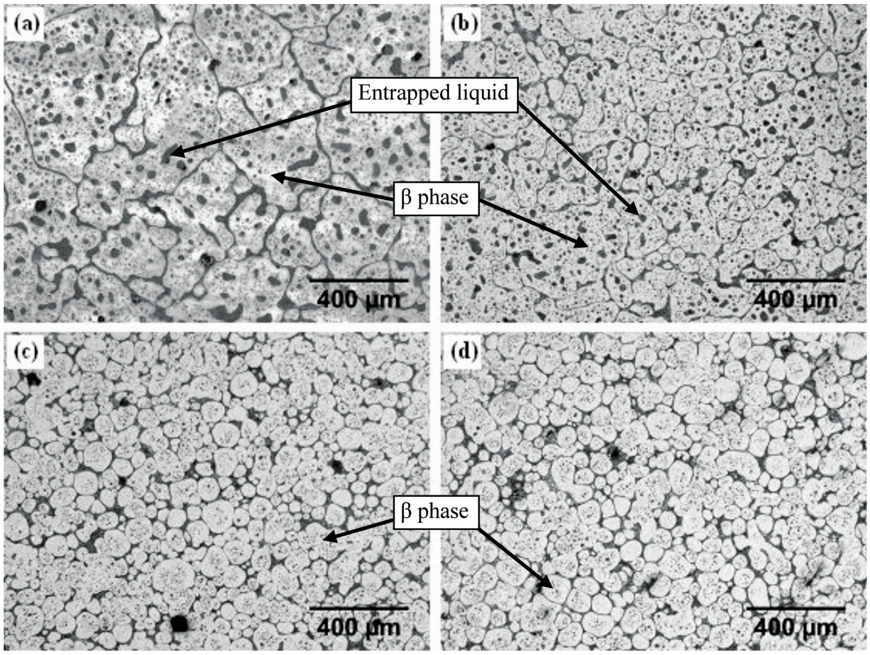

Fig. 4: Microstructure of Zn-22Al subjected to partial re-melting at 438°C for 1 hour on (a) as-cast sample, and on the samples which have previously solution treated at 315°C for (b) 1.5hrs, (c) 3 hrs, and (d) 5 hrs.

Meanwhile, the variations of shape factor and primary grain size of Zn-22Al subjected to partial re-melting are shown in *Fig. 5*. After the partial remelting at 438°C (i.e. semi-solid condition with 48% liquid fraction) for 1 hour for the samples that have previously been solution treated at 315°C, the average shape factor has continuously increased with further increases of treatment time. However, the shape factor of the semi-solid Zn-22Al has significantly decreased after 1.5 hours of holding time. Subsequently, the shape factors continued to increase with the increasing holding time until a maximum value of about 0.9 was achieved at 3 hours.

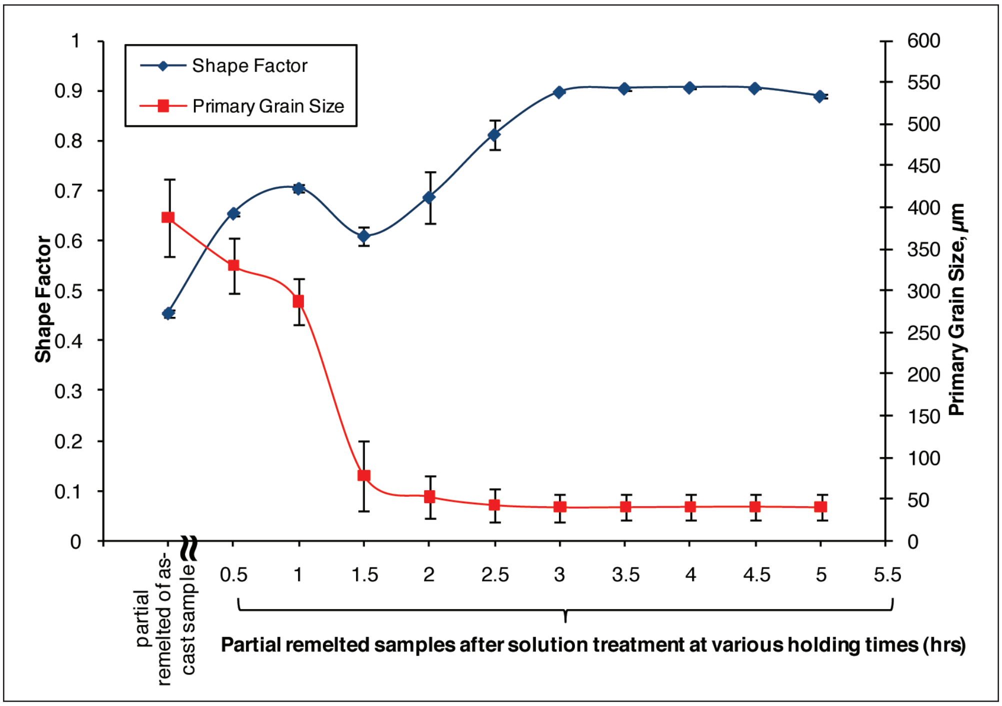

Fig. 5: Variations of shape factor and primary grain size of Zn-22Al subjected to partial re-melting at 438°C for 1 hour on the as-cast sample, and on the samples which had previously solution treated at 315°C (at various holding times).

The curve of the primary particle size in the semi-solid condition proved that the particles had become smaller when the treatment time was increased. It could be noted that the primary particle size decreased significantly when the sample was previously heated for 1.5 hours. Semi-solid non-dendritics, with the best shape factor of 0.9 and the smallest average primary particle size of about 40±16µm in the eutectic matrix, were achieved when the sample was solution treated for 3 hours. The shape factor and size of primary particles in the semi-solid condition have now remained almost constant when the solution treatment was carried out for more than 3 hours (i.e. up to 5 hours). These observations indicate that the solid solution treatment has a great influence on microstructural spheroidisation during partial remelting procedures.

It can be speculated that when the solution treatment holding time was increased, the original dendritic microstructure coarsened through the merging of the dendritic arms and the dendrites due to the dissolution of the interdendritic eutectics, a phenomenon which has also been observed by Chen *et al*. (2006). When the solutionised samples were subjected to partial re-melting, the liquid phase was first formed by melting residual eutectic along the sites at the former grain boundaries, arm boundaries, and arm roots during partial re-melting, resulting in a separation of the coarsened arms from the original dendrites into smaller truncated grains (Flemings, 1991; Chen *et al*., 2006). The truncated grains could perhaps further ripened into spheroidal grains resulting in the non-dendritic microstructures that are probably now suitable for the semi-solid metal processing application. Work on the disintegration of these solutionised dendrites and the dissolution of the interdendritic eutectics (for example by EDAX line scan) is still ongoing.

CONCLUSION

A semisolid microstructure with small and spherical morphologies could be obtained if a traditionally cast Zn-22Al alloy with developed dendrites was solid solution treated for 3 hours at 315°C, followed by partially re-melted at 438°C for 1 hour. In this study, the solid solution treatment was found to have resulted in the dissolution of the interdendritic eutectic structures and the coarsening of the dendritic structures through the merging of the dendritic arms and the dendrites. Partial re-melting has transformed these structures into non-dendritic microstructures which are probably suitable for the semi-solid metal processing application.

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