



The effect of homogenization treatment on microstructure and properties of ZnAl15 solder

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ABSTRACT

The effect of homogenization treatment before extrusion process on microstructure and properties of ZnAl15 solders was investigated. The results of observing microstructure shown that homogenization treatment increased the structural homogeneity and stability of ZnAl15 solder. The results of microhardness measurements and reverse bend test indicated that the homogenized ZnAl15 solder resulted in lower microhardness and much higher ductility. Corrosion behavior of non-homogenized and homogenized ZnAl15 solders was studied in 3.5 wt.% NaCl solution using immersion test and Tafel plots and resulted in higher corrosion resistance of the homogenized ZnAl15 solder.

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1. Introduction

Zn–Al alloy have been widely used in wrought, die casting and coating, due to its favorable combination of physical, mechanical and technological characteristics (low melting point, high strength, exceptional castability, easy machinability, high corrosion resistance, as well as excellent bearing and wear resistance properties) [1,2]. In recent years, Zn–Al alloy is considered as an appropriate filler metal for Al/Cu brazing [3], especially the Zn–Al–RE alloy [4], owing to the fact that the Zn–Al alloy not only solve the environmental problems of Sn–Pb alloy and Zn–Cd alloy [5,6], but also have better corrosion resistance and mechanical properties than Zn–Sn alloy [7]. Unfortunately, “embrittlement” of Zn–Al alloy during storage, especially in rainy summer, restricts its application to Al/Cu brazing [8].

It was found that intergranular corrosion is the cause of “aging failure” in as-cast Zn–Al alloy and Zn–Al coating on steel [9–11]. Adding alloying element is one of the effective measures to refinement corrosion resistance of Zn–Al alloys. For example, adding 0.5–2 wt.% Cu to Zn–Al alloys does good for its corrosion resistance, because Cu can improve stability of the α phase and η phase and reduce the potential difference between η phase and impurity [12]. Adding around 0.06 wt.% Mg can refine the microstructure of Zn–Al alloys, and restrict the decomposition of α phase [13]. Adding 0.4 wt.% Mn is also beneficial for the corrosion resistance of Zn–Al alloy [14]. Rare earth additions enhance the corrosion

resistance of Zn–Al alloy through modification the microstructure [15], and formation a “lanthanide-doped” corrosion products layer [16]. Moreover, reducing the content of impurity elements (e.g. Pb, Cd, Sn, Sb), such as lowering the content of Pb and Sb down to 0.04 wt.% and 0.2 wt.% respectively, has a certain beneficial effect on relieving corrosion of Zn–Al alloys [11]. It is well known that the corrosion resistance of alloys may have a correlation with its microstructure and the solid solubility [17]. Heat treatment, such as T4 heat treatment, is also one of the useful methods to improve the corrosion resistance of Zn–Al alloys [18].

However, most of the research was done on as-cast Zn–Al alloys and research data on refining the “embrittlement phenomenon” of Zn–Al solders is still insufficient. In this paper, the effect of homogenization treatment before extrusion process on microstructures and properties of ZnAl15 solders was investigated, in order to study the effect of homogenization treatment on “embrittlement phenomenon” of ZnAl15 solders.

2. Experimental procedures

2.1. Materials

ZnAl15 solders including 15 wt.% Aluminum were prepared from high purity Zinc and Aluminum. The raw materials were melted using high frequency induction heating in a graphite crucible to 650 °C, and held for 10 min. Then cooled the melted metal to 560 °C, casted it into a preheated cast iron mould, and quenched it in water after cooled to a temperature around 300 °C, and got an ingot of 50 mm in diameter and 120 mm in length. Before ex-

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truded, the obtained rod was carried out homogenization treatment at 320 °C for 5 h and then furnace cooled to the room temperature. Whereafter, the obtained ingot homogenized and non-homogenized, previously heated at 250 °C, were placed in the extrusion die and a gross wire of 5 mm in diameter was obtained by employing a 10:1 extrusion rate. Finally, the gross wire was cold-drawn into a wire of $\varnothing 2.5$ mm.

2.2. SEM and EDX

The metallographic specimens were prepared metallographically with 0.05 μm alumina and subsequently observed by JSM-7500F scanning electron microscope (SEM), employing backscattered electrons at 10 kV. The chemical compositions were analyzed by Oxford INCA-PentaFET-X3 energy-dispersive X-ray (EDX) spectrometer.

2.3. Mechanical properties

Microhardness measurements were carried out at a load of 100 gf at room temperature. The load was applied for 10 s. Ten random hardness readings were taken for each specimen.

Reverse bend test was carried out at room temperature according to ISO 7801 [19]. The specimen's dimensions for reverse bend test were $\varnothing 2.5 \times 300$ mm. Number of reverse bends (N_b) was measured to indicate the tenacity of ZnAl15 solders. Three measurements were taken for each specimen.

2.4. Corrosion test

The immersion test and electrochemical polarization measurements were used to estimate the corrosion resistance of cold-drawn ZnAl15 solders. Corrosion rates of the homogenized and non-homogenized solders were determined by the weight loss method and using Tafel plots. All tests were performed in an approximately neutral NaCl solution (3.5 wt.% NaCl, pH = 6.7) in contact with air.

2.4.1. Immersion test

The specimens ($\varnothing 2.5 \times 40$ mm) for immersion test were prepared and exposure procedure were performed in accordance with ASTM: G31-72(2004). After weighing, the samples were exposed (in triplicate) in stagnant 3.5 wt.% NaCl solution at room temperature and were vertically immersed in the test solution. The period of exposure was 1 month. After exposure samples were removed from the test solution and rinsed with distilled water. Corrosion products were removed from the samples surface by chemical cleaning according to ASTM: G1-03(2011). The samples were then reweighed to determine their mass loss during exposure to NaCl solution. According to ASTM: G31-72(2004), the average corrosion rate, in mm/year, was calculated on the basis of the samples' mass loss during immersion test:

$$\text{Corrosion rate} = (K \times W)/(A \times T \times D) \quad (1)$$

where K is a constant, W is sample mass loss in grams, A is specimen area in cm^2 , T is time of exposure in hours and D is density of ZnAl15 solder in g/cm^3 .

2.4.2. Tafel plots

Polarization measurements were carried out at room temperature using a cell for flat specimens with a contact area of 0.049 cm^2 (the area of vertical section of cold-drawn ZnAl15 solder) between working electrode and electrolyte (3.5 wt.% NaCl, pH = 6.7). The cell contained the platinum electrode as a counter electrode and the saturated calomel electrode (SCE) as a reference electrode. All the potentials were referred to the SCE. Working electrode was

washed with acetone and distilled water before the polarization test. The open circuit potential (OCP) of working electrodes was monitored 30–60 min prior to application of electrochemical techniques. Tafel plots were obtained by starting the potential scan from a cathodic potential and increasing the potential towards the anodic side at a scan rate of 0.5 mV/s, using RST5000 electrochemical workstation. Each electrode was potentiodynamically polarized in the potential range ± 0.200 V over the OCP respectively. Three measurements were performed for each sample with good reproducibility. The corrosion rate, calculated from Tafel plots was as follows [20]:

$$\text{Corrosion rate} = (M \times i_{\text{corr}})/(z \times F \times \rho) \quad (2)$$

where M is the atomic weight of the element in g/mol , z is the number of electrons required to oxidize an atom of the element in the corrosion process, F is the Faraday's constant in $\text{A s}/\text{mol}$, ρ is the density in g/cm^3 (see ASTM: G1-03(2011)), i_{corr} is the current density in A/cm^2 .

3. Results and discussion

3.1. Microstructures

3.1.1. As-cast

According to Zn–Al phase diagram [21], the supersaturated Al-rich α_s phase precipitated out firstly to form the dendrites during solidification. With temperature decreasing, the composition of the molten metal around the first solidified α_s phase gradually changed to the composition of β_s phase and peritectic reaction occurred incompletely. The Al-rich β_s phase appeared at the edges of the α_s phase, around the cores of the dendrites. Meanwhile, β_s phase precipitated to form the dendrites just as the α_s phase. When cooling to 381 °C, the eutectic structure ($\beta_s + \eta_s$) solidified finally in the inter-dendrite regions. η_s phase was a Zn-rich phase. Due to bearing a rapider cooling rate in the solidification process, the equilibrium reaction could not carry out and eutectoid transformation of β_s phase was inhibited [22]. Therefore, the microstructure of as-cast ZnAl15 solder is composed of primary dendrites ($\alpha_s + \beta_s$ phase or β_s phase) and eutectic matrix ($\beta_s + \eta_s$ phase), as shown in Fig. 1a. Nevertheless, primary discontinuous precipitation of supersaturated α_s and β_s phase occurred at room temperature, and the corresponding microstructure was shown as Fig. 1b.

Fig. 1c displays the microstructure of as-cast ZnAl15 solder homogenized. As can be obtained from Fig. 1c, discontinuous precipitation of α_s and β_s phase occurred completely during homogenization treatment, and the corresponding microstructure was shown as Fig. 1d. The final microstructure of decomposed α_s was fine lamellar structures ($\alpha + \eta$). β_s phase in primary dendrites decomposed into coarse lamellar structures ($\alpha + \eta$). At the decomposition process of β_s phase, which locates at the edges of the α_s phase or in eutectic matrix, η phase grew along the original η phase. Therefore, the final microstructure was that α phase distributed in η matrix.

3.1.2. Extruded

During hot extrusion process, supersaturated α_s , β_s and η_s phases decomposed and the corresponding microstructure is shown in Fig. 2a, where the black phase is Al-rich α_E phase and the white phase is Zn-rich η_E phase [23]. Meanwhile, the dendritic structures were fragmented and refine microstructure was obtained. Unfortunately, there was still some coarse microstructure in the extruded ZnAl15 solder non-homogenized, which may be caused by the heating effect from decomposition of α_s , β_s and η_s phases [24]. After the homogenization treatment has been carried out before the extrusion process, the microstructure of extruded

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