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Effect of Ni additions on the microstructure of Zn based lead free solder alloys for high temperature applications

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ABSTRACT: The effects of nickel additions on the change in microstructure of Zn-*x*Ni lead free solder alloys were investigated. The investigation revealed that increasing Ni additions led to the increase in size of intermetallic Zn-Ni particles along with an increase in volume fraction of intermetallic particles. The microstructure also revealed the presence of the prominent delta (δ) phase in all three compositions of the solder alloy i.e. Zn-0.7 mass% Ni, Zn-1.0 mass% Ni and Zn-1.5 mass% Ni respectively. The presence of irregular shaped δ phase particles and conjoined δ phase particles were noticed in alloys with higher Ni content along with δ particles at grain boundaries. The most remarkable change occurred in the size of Zn grains, which decreased in size as Ni additions were increased. This reduction in size can be attributed to pinning effect of Zn grains by intermetallic particles.

KEYWORDS: Zn-*x*Ni lead free solder alloys, intermetallic Zn-Ni particles, delta (δ) phase, conjoined δ phase particles, pinning effect.

1 INTRODUCTION

Lead-free solders have received remarkable attention in the field of high temperature industrial applications. These applications include bonding of semiconductor devices onto substrates, step soldering technology, flip-chip connections, solder ball connections, etc [1]. There has been significant research in the field of lead-free solders but only a small degree of research specifically relate to high temperature lead-free solders [2]. High-temperature solder alloys are however, a key structural material for various industrial components and assemblies which require a high level of quality and reliability and also to be free from lead. As such, there is a need to identify the relative strengths and weaknesses of the current generation of lead-free solder alloys suitable for high-temperature applications and identify opportunities for further developments.

Solder alloy systems like Pb–Sn, Au–Sn, Au–Ge, Zn–Al, Zn–Sn, Bi–Ag and Sn–Sb alloys are high temperature solders typically designed to withstand temperatures between 150 and 200^{0}C [3]. These solders have been the subject of research on various functional properties [4-6] and reliability issues [7-9]. One of the basic criteria a high temperature solder should satisfy is having an appropriate melting range that allows ease of manufacture of soldered components. This melting range has been defined by industry as 270–350^{0}C in order to ensure efficient process control [10]. Selecting of the solder alloy for a specific job must take into consideration its melting temperature which should be higher than service temperature. At the same time, the solder alloy should possess a solidus temperature higher than 270^{0}C in order to withstand peak temperatures of second level soldering. The liquidus temperature of solder alloy should be below 350^{0}C to avoid thermal degradation of polymers commonly used in the substrate. These temperature criteria are fundamental considerations in the selection of high temperature solders. However, reasonable consideration must be given to alloys that fall outside of the temperature guidelines if properties are not adversely affected. For example, few Zn-base solders were developed for ultra-high temperature applications with the alloying elements: (4–6) mass% Al and (1– 5) mass% Cu [6]. These solders were designed to have liquidus temperatures between 382 and 402^{0}C.

An attempt was made to develop Zn based high temperature solders by addition of Ni. As a result the system attained higher melting point than 402^0C. The Zn-Ni eutectic temperature is around 420^0C that is higher than the Zn-Al-Cu ternary system as shown in the binary phase diagram in Fig. 1. In this study the effect of varying Ni content on the change in microstructure has been studied by preparing Zn-Ni solder alloys by varying the mass% of Ni.

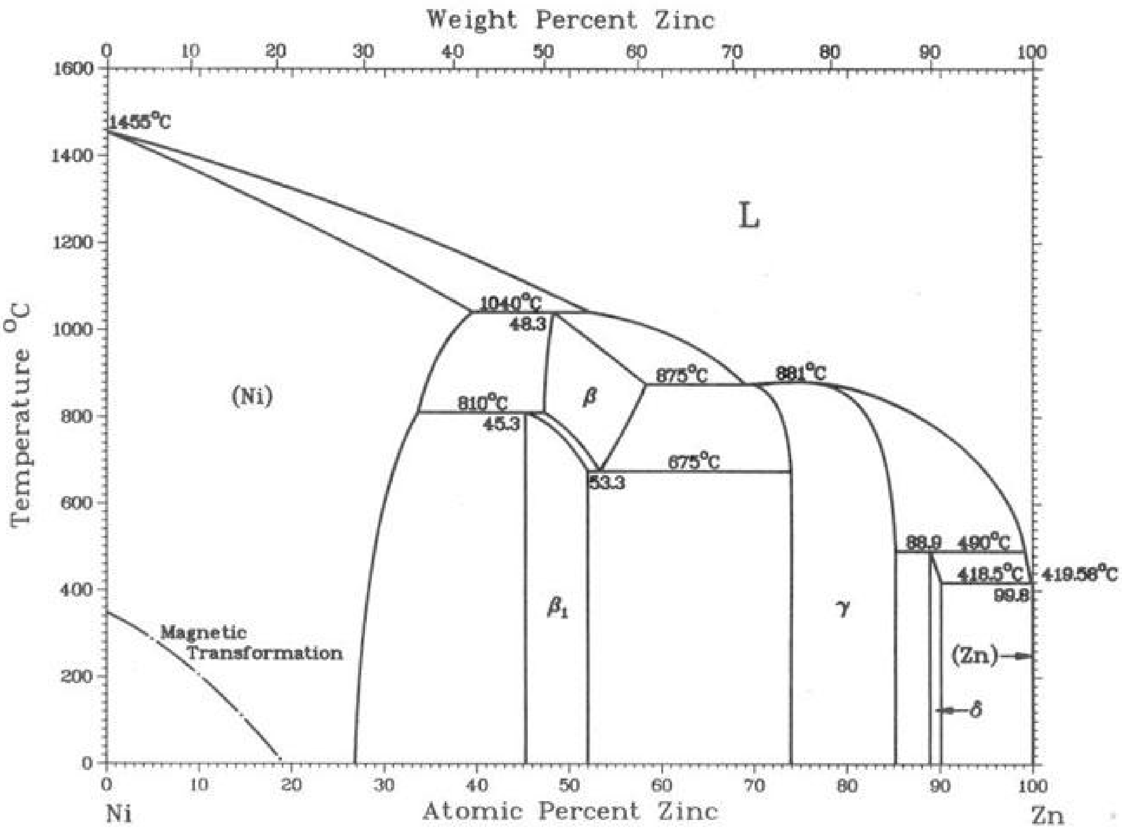

Fig. 1. Zn-Ni binary phase diagram

2 Materials and Methods

2.1 Preparation of Solder Alloy

The lead free solder alloys were prepared from commercially available pure Zn and Ni powders. Three Ni compositions were used for the respective study, i.e. 0.7 mass%, 1.0 mass% and 1.5 mass% Ni for the preparation of lead free Zn-Ni solder alloys. In the primary step, Zn briquettes were placed in a graphite crucible for melting in a gas fired pit furnace. The Ni powder to be added to the molten Zn was first preheated to around 250^0C before addition. After preheating, required amount of Ni powder was added to the melt. The melt was homogenized by manual stirring for few minutes and was finally poured in to preheated metal mold at a pouring temperature of 530^0C. This process was carried out for all the desired Ni compositions. The preparation steps for casting the solder alloy is shown in Fig. 2(a) to 2(e).

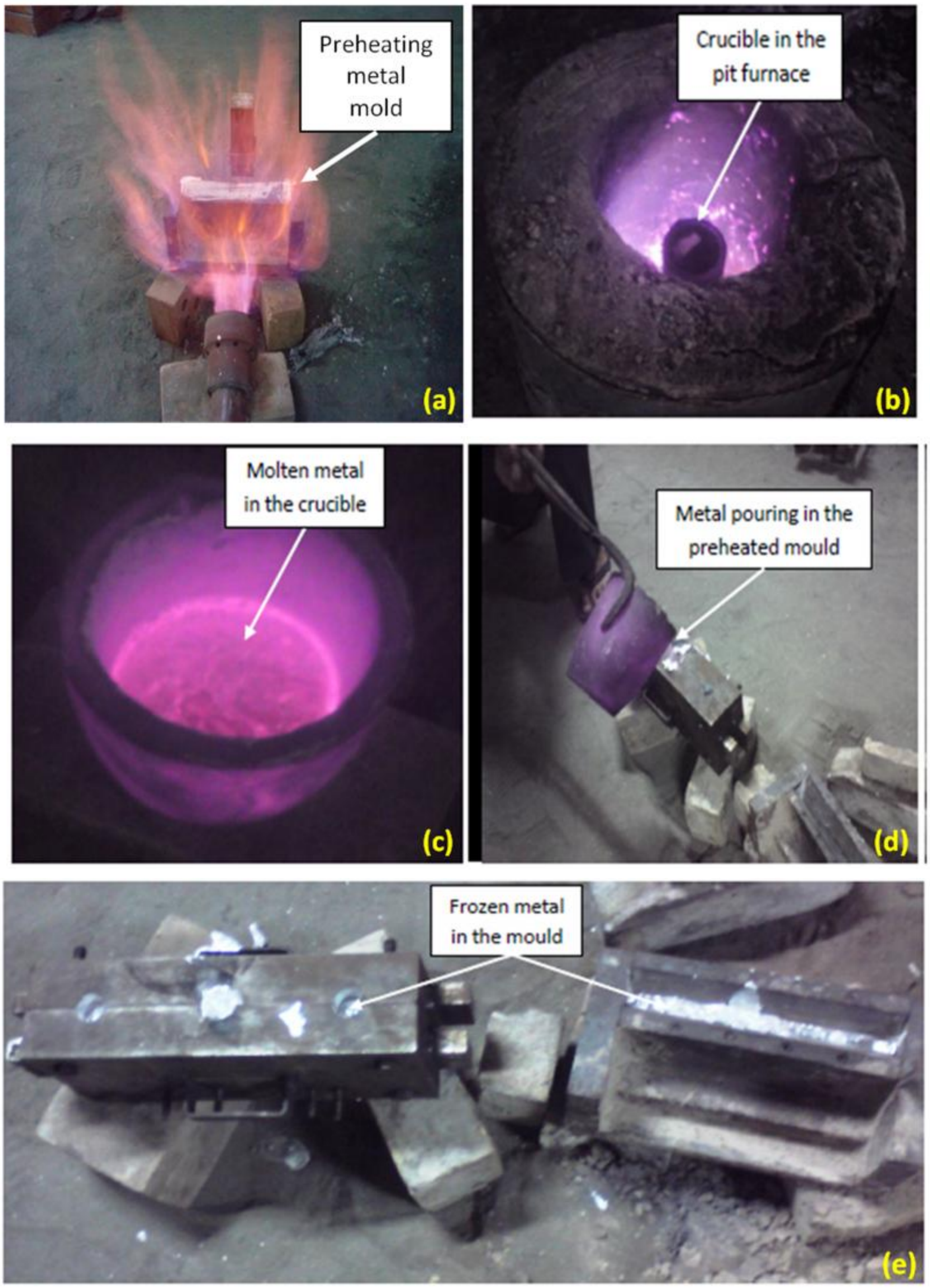

Fig. 2. Preparation of Zn-Ni Solder alloys (Casting Route)

2.2 MICROSTRUCTURAL ANALYSIS

Cylindrical samples were prepared for microstructural analysis. After progressive polishing on grit papers, samples were brought for fine polishing on a wheel where alumina powder was used as polishing media. Non-ferrous metallographic fine polishing standard technique was carried out with 0.05μm Al_2O_3 particles in order to obtain the microstructure. Then samples were cleaned with water and finally cleaned and dried in acetone. After drying the samples were etched in an etching solution containing 100 ml Ethanol (96%) and 1 to 5 ml Hydrochloric acid (32%). The samples were investigated by an optical microscope ((LEICA-MZFLIII, Italy)) and micrographs were recorded with a digital camera (OPTIKA Microscope B-600 MET).

3 RESULTS AND DISCUSSIONS

The microstructure of lead free Zn-Ni solder alloys are given in Fig. 3. The changes in microstructure with %Ni additions are readily visible with the increase in volume fraction of Zn-Ni intermetallic particles in the matrix. From Fig. 3 it is clearly evident that on increasing the mass% of Ni addition to the Zn based solders, the size of Zn-Ni intermetallic particles increases along with an increase in volume fraction of intermetallic particles. The matrix of the Zn-Ni lead free solder alloy consists of variation in the shape of intermetallic particles due to incomplete dissolution of Ni in Zn. These variations comprises a heart shaped phase known as delta (δ) phase, irregular δ shaped phase, conjoined δ phase and irregular shaped. The microstructure also shows the evidence of incomplete dissolution of Ni particles in Zn which are positioned preferentially at the grain boundaries in the form as a second phase. Due to the increase in volume fraction of intermetallic particles, there is a decrease in the size of Zn grains. This can be contributed to the pinning effect of Zn grains by the intermetallic particles/phase.

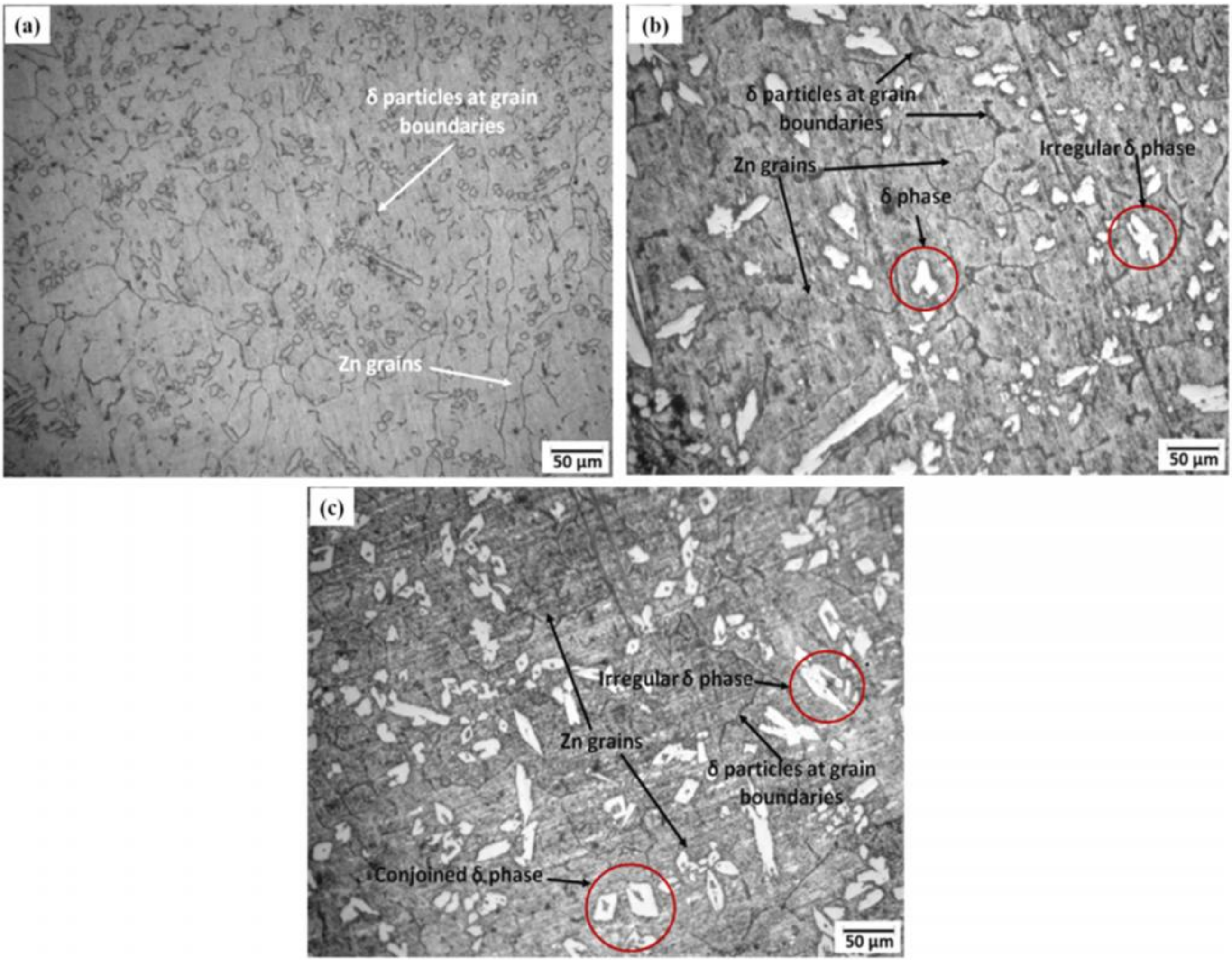

Fig. 3. Microstructures of Zn-Ni lead free solder alloy after (a) 0.7 mass% Ni addition, (b) 1.0 mass% Ni addition, (c) 1.5 mass% Ni addition [Magnification: 200X]

The microstructure of Zn-Ni free solder alloy at a higher magnification is shown in Fig. 4. The microstructure shows the presence of δ phase, conjoined δ phase/particles and intermetallic second phase at the grain boundaries. The cause of two δ phase joining head to head has yet to be studied. This type of structure is seen to increase as mass% Ni additions increase.

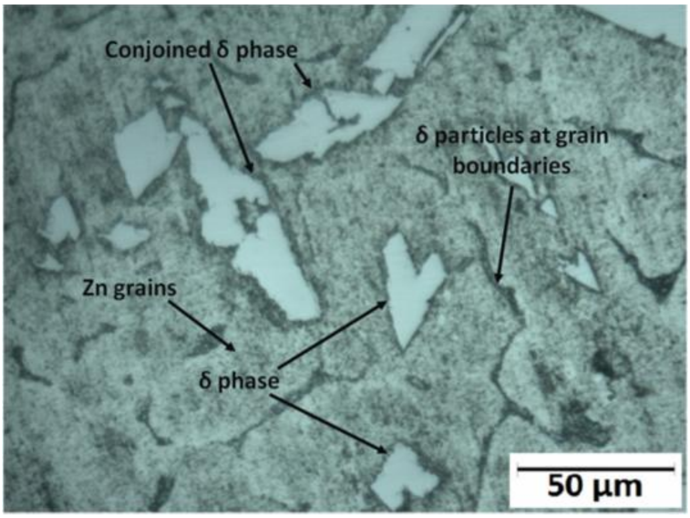

Fig. 4. Microstructure of Zn-1.5 mass% Ni lead free solder alloy [Magnification: 500X]

4 CONCLUSIONS

The change in microstructure of Zn-Ni lead free solder alloys was investigated and it was found that by increasing the mass% Ni additions the size and volume fraction of Zn-Ni intermetallic particles increased. The size of Zn grains decreased as Ni additions increased, which can be contributed to pinning effect by intermetallic particles during grain growth. The presence of intermetallic second phase particles were noticed due to incomplete dissolution of Ni in Zn.

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