Lead-free Solders with Rare Earth Additions

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There have been significant research efforts to improve the properties of globally recognized lead-free solders, such as Sn-Ag-Cu, Sn-Cu, Sn-Bi, etc. Rare earth (RE) elements have been selected in the current research to be added into lead-free solders as alloying elements in an effort to improve the comprehensive properties, such as wettability, microstructural stability, creep resistance, and mechanical strength and integrity of the solders. This article overviews the background and rationale for the alloy development, the fabrication techniques for RE-containing solders, the roles of RE elements, as well as current status of RE-containing solder research. Examples of several representative RE-containing solders are introduced. The effects of RE element addition on the wetting behavior, microstructure, and mechanical properties of certain lead-free solder alloys are presented and analyzed.

INTRODUCTION

In recent decades, anticipation of the introduction of legislation (e.g., Restriction of Hazardous Substances directive and the Waste Electronical and Electronic Equipment directive) accelerated research into lead-free solders. As a result of those efforts, a number of lead-free alloys are currently commercially available. These solders are mostly Sn-based binary and ternary alloys with such alloying elements as Ag, Cu, Bi, In, Sb, and Zn. The new lead-free solder alloys, which will be used in the electronics industry, need to meet a variety of property requirements such as good wettability, low melting point, low cost, adequate strength, good thermomechanical fatigue resistance, excellent creep properties, etc.[1,2] Modern microelectronics products demand high reliability, so electronic lead-free solders and their interconnections need to be reliable over long-term use. Although many of the commercially available lead-free solders possess good attributes such as high strength, there is still room for improvement in certain properties to facilitate manufacturability and long-term reliability. Research efforts in this direction have been very active and are continuing in lead-free alloys.

One of the effective approaches is the addition of alloying elements to the existing lead-free alloys. It has been known that the addition of a small amount of rare-earth (RE) elements in metals can greatly enhance their properties. Due to the unique properties of RE elements, several major research groups in the world have attempted to improve the properties of solders by adding RE elements. General findings in such investigations revealed that several aspects of the properties of most popular lead-free solders, such as Sn-Ag, Sn-Cu, Sn-Zn, and Sn-Ag-Cu, could be tailored with the addition of trace amounts of RE elements.[1–10] The modification of the properties gives the solder alloys better wettability, creep strength, and mechanical integrity.[11–18] For example, Dudek et al. found that adding La to Sn-3.9Ag-0.7Cu solders could enhance the ductility of the alloy, which has profound implications for improving the mechanical shock resistance of electronic packages containing Sn-Ag-Cu solders.[11] Wu et al.,[1] Wang et al.,[9] and Zhao et al.[10] investigated the effect of RE elements on the microstructure of the lead-free solders. Experimental results showed that growth of intermetallic compounds could be dramatically repressed with

How would you…

…describe the overall significance of this paper?

Rare earth elements are considered as the vitamins for metals. This paper presents a viable means of improving the reliability of promising Pb-free solders by rare earth addition. The fabrication techniques of solders as well as the effectiveness of reliability improvement are presented, which serves as a general overview for people who are interested in both theoretical investigation and practical application.

…describe this work to a materials science and engineering professional with no experience in your technical specialty?

The current investigation discusses the alloying methods aiming at improving the mechanical strength, especially reliability, of several Pb-free solder alloys. The primary alloying elements employed were La, Ce, and Y type of rare earths. The effects of rare earth addition on the processing properties, microstructural stability, mechanical strengths, and reliability issues such as creep, thermomechanical fatigue, etc., are reported.

…describe this work to a layperson?

Use of Pb has been prohibited by law due to its toxicity in the traditional Pb-Sn soldering materials. However, Pb-free candidates to replace the Pb-bearing solders have encountered various reliability difficulties due to increasing miniaturization and severe service conditions of modern electronic components. Adding several rare earth elements has been successful in improving the reliability of the solder materials. This paper reports how such additions are accomplished and how the reliability problems are solved based on both experimental facts and theoretical considerations.

the appropriate addition of RE, resulting in a fine microstructure. Other studies have also shown that the addition of RE elements to existing lead-free solders could make them able to wet the surface of silicon and optical materials.[2] Apart from these studies, Chuang et al. have investigated the effect of RE elements on the growth of tin whiskers.[19–21]

Significant research efforts have been conducted for the past decade on RE-containing Sn-based solders in the Advanced Electronic Interconnection Materials Laboratory at Beijing University of Technology. This article reports the fabrication technique of RE addition, as well as the effects of RE addition, especially La and/or Ce addition, on the microstructural evolution, tensile behavior, and creep properties of lead-free solders. See the sidebar for a description of fabrication techniques.

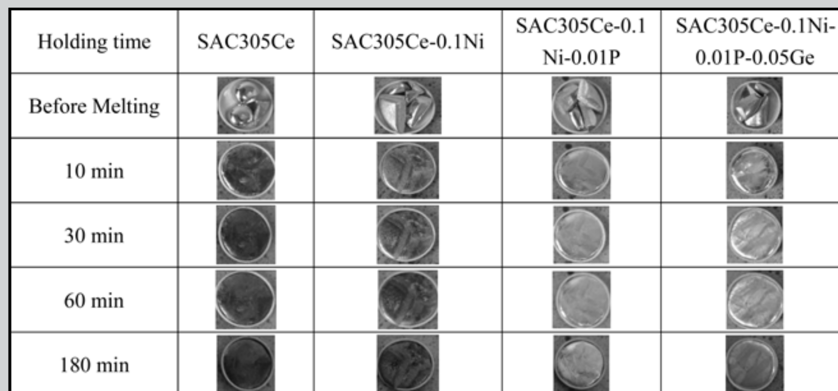

Figure 1. The surface conditions of the SAC305Ce based molten solders at the different holding times at 260°C.

PROCESSING PROPERTIES

Wetting Characteristics

One important aspect investigated after RE-containing solders were fabricated was the effect of the addition of such an RE mixture on the melting, wetting, and spreading properties of the original alloy. Here we use spreading property as an example, which reflects the wettability of solder on the substrate under a certain environment. When RE was less than 0.5 wt.%, the spreading area of Sn-3.8Ag-0.5Cu solder was found to increase a little as RE content was increased, but not significantly. The area decreased obviously when RE was added up to 1.0 wt.%. The RE is a surface-active element that can lower the surface tension of liquid solder, which is supposed to favor the wetting of the solder on the substrate. However, since RE is prone to oxidation, the formation of oxide residue during soldering will on the other hand limit the wettability of the solder. With the increase of RE, such negative effect exceeds the favorable aspect of RE. From this point of view, it is not desirable that the RE element exceeds 1.0 wt.%.[25]

Oxidation Properties

Improving the anti-oxidation ability of solder not only can improve the wettability, but also decrease the cost of solder by reducing the waste residues during processing. The effect of adding 0.05 wt.% Ce to Sn-3.0Ag-0.5Cu solder (SAC305Ce) on the oxidation behavior was investigated and did not show evident improvement with such RE addition.[26,27] The anti-oxidation ability of solders was characterized by the color of oxidation film in liquid solder surface at different holding times, based on the proportionality between the oxidation film thickness and the wavelength of the incident light. The color of the oxidation film became darker with increasing film thickness. Preliminary findings revealed that the addition of Ni did not help improve the anti-oxidation properties. However, it was observed that the oxidation of the SAC-305Ce solder was significantly reduced with the addition of P and Ge. Figure 1 shows the surface conditions of the molten SAC305Ce based solders at different holding times. It is obvious that the SAC305Ce-0.1Ni-0.01P and the SAC305Ce-0.1Ni-0.01P-0.05Ge solders did not show significant color change even after 3 hours of holding at 260°C. A much shinier surface could be observed on the SAC305Ce-0.1Ni-0.01P-0.05Ge solder specimen. As a contrast, solders without P and Ge addition became increasingly darker after exposure in air for more than 10 minutes. It was believed that P and Sn are more likely to form complex oxygen-containing acid salts on the solder surface. The dense oxidation barrier, as a protective film to prevent the further oxidation of SAC solders, enhances the anti-oxidation of the solder.

FABRICATION TECHNIQUES

Using Sn-3.8Ag-0.7Cu-RE as an example, the RE-alloyed lead-free alloys were usually fabricated with pure Sn, Ag, and Cu as starting materials. The purity of all the materials was 99.9 wt.%. The RE elements to be added to Sn-3.8Ag-0.7Cu alloy were primarily RE mixtures, which were mainly composed of ~40 wt.% to 50 wt.% of Ce and ~20 wt.% to 30 wt.% of La. The remainder of the composition contained ~15 wt.% Nd, 8 wt.% Pr, and other minor elements. The raw materials of Sn, Ag, and Cu were placed in an Al_2O_3 ceramic crucible and melted at 550°C to 600°C for 20 min. With the help of a stainless steel bell with holes on the sidewall, the RE mixture was pushed down into the molten solder. After the RE was melted, the molten alloy was held for 40 min. The molten alloy was mechanically stirred every 10 min. with a stainless steel rod to promote uniformity of the solder alloy. A eutectic salt KCl + LiCl with a weight ratio of 1.3:1 was used during smelting to cover the surface of the liquid solder to prevent oxidation. The molten solder was finally chill cast into a cylindrical ingot before further machining for different testing purposes.[22,23] The fabrication of other RE-containing solder alloys, such as Sn-Bi-RE, and Sn-Ag-Bi-RE, etc., followed the same techniques.[24] The methodology for which wetting experiment, metallographical specimen preparation, microstructural observation, mechanical testing, and creep test set-up were conducted were all included in the published literature which will not be reiterated here.[22–41]

RE EFFECT ON MICROSTRUCTURAL STABILITY

The most significant influence of RE addition on microstructural stability of the solder alloy was refining the microstructure while suppressing intermetallic compound (IMC) growth.[22,25,28–33] Figure 2 shows the microstructure of the Sn-3.8Ag-0.7Cu alloys with different minute amounts of mixed RE. The microstructure of SnAgCuRE was similar to Sn-3.8Ag-0.7Cu. The binary and ternary eutectic microstructure dispersed between the dendrites as well as some intermetallics. As the RE content was increased, the microstructure of solder followed a refining trend. The acicular binary-eutectic β-Sn +Ag_3Sn microstructure disappeared and the Ag_3Sn phase formed as fine particles. The number of the RE compound phases also increased and they distributed between the dendrites, coexisting with the eutectic microstructure. When RE was added up to 0.1 wt.%, the RE compound phase began to appear in the microstructure of the Sn-3.8Ag-0.7Cu-0.1RE alloy. The shape of the RE phases usually took on a regular morphology, similar to a snowflake, and some could even fall into a line, as shown in Figure 2c.

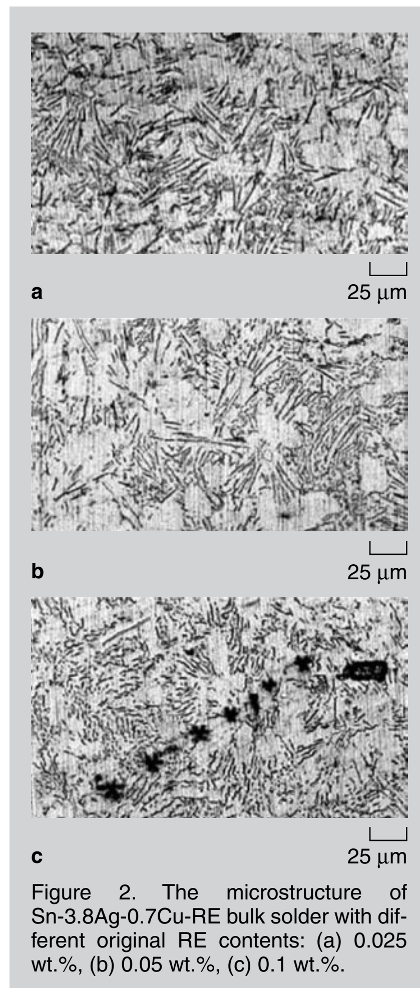

Figure 2. The microstructure of Sn-3.8Ag-0.7Cu-RE bulk solder with different original RE contents: (a) 0.025 wt.%, (b) 0.05 wt.%, (c) 0.1 wt.%.

The size and morphology of the interfacial Cu_6Sn_5 IMC layer of Sn-Ag-Cu solder joints differed greatly with various RE contents.[22] First, as shown in Table I, the thickness of reaction layers in the solder joints with RE addition was thinner than that of the Sn-Ag-Cu solder joint. However, the thickness did not linearly change with the RE content. The thickness of the reaction layers reached the lowest value when the RE was 0.1 wt.%, and increased when the RE content was 0.05 wt.% or 0.25 wt.%. Second, there also existed a difference in the morphology of the IMCs. In the solder joint without RE, IMCs often grew perpendicularly from the interface and were well knit, while in the Sn-Ag-Cu0.1RE-Cu solder joint, the number of Cu_6Sn_5 IMCs was much less, and consequently, the IMCs were arranged loosely with various growing directions instead of growing vertically.

The phenomenon described above can be explained from two points of view. One is from the effect of RE on the driving force of the Cu_6Sn_5 IMC layer growth. The Cu atom diffuses through the IMC layer to Sn, which means the driving force of the Cu_6Sn_5 layer growing depends on the activity of Sn at the Cu_6Sn_5/Cu interface. Thermodynamic parameters of element affinity indicate that it is easy for RE to interact with Sn in the Sn-Ag-Cu alloys to form IMCs, resulting in increased resistance of Cu_6Sn_5 IMC layer growth. The other aspect arises from the difference of Sn atoms and RE atoms. The diameter of an Sn atom is 0.141 nm, while those of Ce and La are 0.183 nm and 0.181 nm, respectively. The radius of the RE atom is 33% larger than that of Sn. Consequently, it is difficult for RE atoms to replace Sn atoms in the matrix. As a result, it is easy for RE atoms to gather at crystal defects, such as dendrite boundaries, and resist growth of the IMCs. Earlier research[28] showed that the interlocking of β-Sn dendrite and Cu_6Sn_5 layer was present, where RE elements were enriched at the boundaries in the form of a web. This inserted structure can increase the resistance of grain growth, and hence, to some extent, repressed grain coarsening.

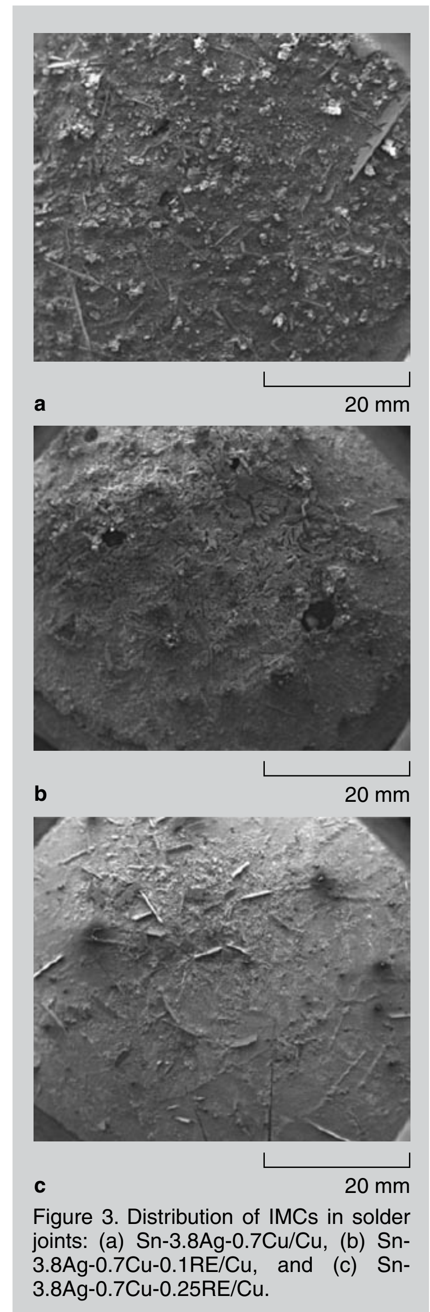

Figure 3. Distribution of IMCs in solder joints: (a) Sn-3.8Ag-0.7Cu/Cu, (b) Sn-3.8Ag-0.7Cu-0.1RE/Cu, and (c) Sn-3.8Ag-0.7Cu-0.25RE/Cu.

The addition of RE in general also affected the size, quantity, and distribution of various types of IMC, including Cu_6Sn_5, Ag_3Sn, and the plate-like Sn-Ag IMCs. The low-magnification micrographs in Figure 3 show the distribution of IMCs in joints of three solder alloys with various RE additions on Cu substrates. In Sn-3.8Ag-0.7Cu/Cu solder joints (Figure 3a), large plate- or needle-like phases are clearly

Table I. Dimension of the IMCs in Sn-3.8Ag-0.7Cu Solders with and without RE

Alloy IMCs	Solders			
	Sn-3.8Ag-0.7Cu	Sn-3.8Ag-0.7Cu-0.05RE	Sn-3.8Ag-0.7Cu-0.1RE	Sn-3.8Ag-0.7Cu-0.25RE
Large Sheet of Ag_3Sn				
Length (mm)	0.7–1.3	0.5–0.9	0.2–0.3	0.5–0.7
Thickness (μm)	15–30	9–18	5–6	10–14
Cu_6Sn_5 Diameter (μm)	10–25	15	7–10	21
Cu_6Sn_5 Reaction Layer (μm)	40–60	40–50	25–40	35–50

shown. They interlock each other and some break attached on the surface of solders. Compared with that, the IMCs of Sn-3.8Ag-0.7Cu-0.1RE/Cu joints decreased dramatically in both quantity and size, as shown Figure 3b. The surface seemed to be smooth and only a few IMCs could be seen in a microscope with low magnification. However, with the increase of RE, the quantity of IMCs of Sn-3.8Ag-0.7Cu-0.25RE/Cu solder joint increased and could be easily observed again, as shown in Figure 3c. According to the data given in Table I, it is suggested that the morphology of the IMCs is related to the RE content. Appropriate RE addition can resist the growth of IMCs dramatically, because RE atoms gathered at the dendrite boundaries can serve as barriers to the atomic diffusion and consequently restrain the growth and movement of the dendrites.

Another research effort involved the addition of erbium (Er) to Sn-3.8Ag-0.7Cu alloys.[31] With the addition of 0.15 wt.% Er to the Sn-3.8Ag-0.7Cu lead-free solder alloy, the thickness of the Cu-Sn IMC at the interface was also found to decrease during soldering, and the growth of the Cu-Sn IMC was inhibited during the aging process. The effects of Er addition on the microstructure evolution of Sn-3.8Ag-0.7Cu solder during high temperature aging (170°C) are presented in Figure 4. It can be seen from the micrographs that at 170°C, the microstructure of Sn-3.8Ag-0.7Cu solder experienced evident coarsening during long time aging, and micro-cracks were formed inside the solders after 1,000 h. However, the RE-containing lead-free solders exhibited stable microstructure during long time aging, and no micro-cracks were present. $ErSn_3$ IMCs formed from the molten solder were considered to provide heterogeneous nucleation sites for the precipitated Ag-Sn IMCs. The increase in nucleation probability consequently led to the refinement of the Ag-Sn IMCs. The small and uniform Ag-Sn IMCs obtained in the Er-containing solder joints slowed down the ripening rate, and the refinement effect was achieved. However, for an Er-free solder joint, massive Ag-Sn IMCs in Er-free solder joints were precipitated in the solder matrix, and the compound/solder boundaries provided favorable sites for cracks to nucleate and propagate without significant consumption of energy.

RE ELEMENTS: EFFECT ON MECHANICAL PROPERTIES

Tensile Properties

The addition of RE enhanced the ultimate tensile strength of the Sn-3.8Ag-0.7Cu solder in a very narrow range but gave a 30% increase at most in elongation, as show in Table II.[25] With the increase of RE, elongation gradually descended to the level of Sn-3.8Ag-0.7Cu when RE content exceeded 0.25 wt.%. The addition of RE resulted in the formation of hard RE compounds, and as RE was increased, the number of RE compounds increased, too, which was assumed to lower the elongation. The

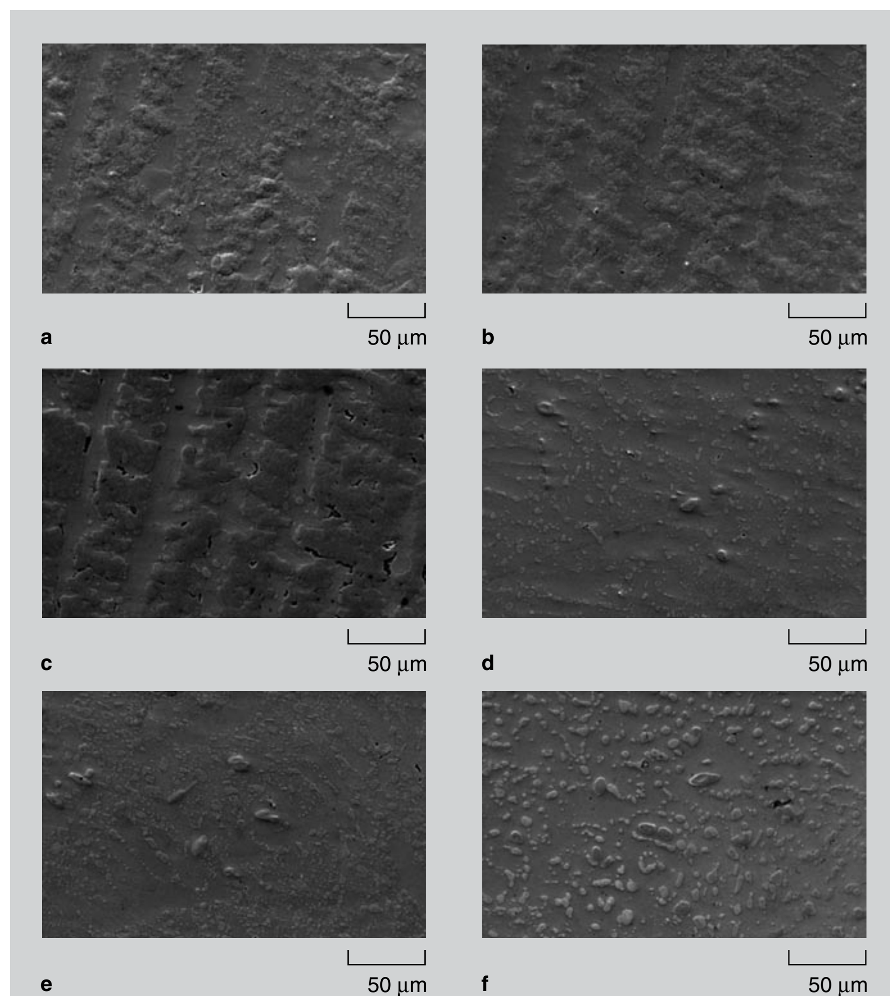

Figure 4. Microstructure evolution of Er-containing Sn-3.8Ag-0.7Cu solders during high temperature aging (170°C). (a) Sn-3.8Ag-0.7Cu, 200 h; (b) Sn-3.8Ag-0.7Cu, 500 h; (c) Sn-3.8Ag-0.7Cu, 1,000 h; (d) Sn-3.8Ag-0.7Cu-0.15Er, 200 h; (e) Sn-3.8Ag-0.7Cu-0.15Er, 500 h; (f) Sn-3.8Ag-0.7Cu-0.15Er, 1,000 h.

value of the fracture energy reflects comprehensive performance relevant to tensile strength and elongation of materials. From the tensile curve, the fracture energy could be assessed by calculating the area between the curve and horizontal axis. The fracture energy of the SnAgCuRE alloy increased first before a decrease was observed, which resembled the altering trend of elongation. While it could be concluded that adding RE improved the resistance to elastic deformation of the SnAgCu solder, as indicated by the increasing trend of elastic modulus with the increase of RE content, it has to be emphasized that the tendency of the effect of RE on the strength of the solder joint was not obvious. When RE was added up to 1.0 wt.%, the strength decreased greatly.

A systematic shear testing on the Sn-3.8Ag-0.7Cu-0.1RE alloy conducted later on also validated the results obtained in the above study.[34] The shear strength of the solder joints as-reflowed and after aging at 150°C for 168 and 336 h was measured at a constant loading rate of 0.3 mm/min. and room temperature. The results indicated that the shear strength of Sn-3.8Ag-0.7Cu-0.1RE solder joints was slightly lower than that of the Sn-3.8Ag-0.7Cu solder joints. The shear strength of both solder joints was reduced after aging at elevated temperature. However, the shear strength reduction rate of the Sn-3.8Ag-0.7Cu-RE solder joints was much slower. The addition of RE could obviously suppress the deterioration of the shear strength of the solder joints during aging, though no initial strengthening effect was observed. Even after thermomechanical fatigue testing between –40°C and 125°C for 1,000 cycles, RE-containing solder joints exhibited a slower deterioration rate as compared with the joints without RE addition. Such improvement was believed to be attributed to the refinement of microstructure, especially bulk IMCs, which could effectively alter the path for crack propagation from along the solder/Cu substrate interface to the bulk solder region.[35] Wu et al.[1,2] investigated the mechanical properties for Sn-9Zn and Sn-0.7Cu alloy. The results also indicated that adding Ce and La type of RE elements did not have significant influence on the ultimate tensile strength of the solder alloys.

Creep Properties

As Figure 5 shows, a minute amount of RE could remarkably increase the creep-rupture life of the Sn-3.8Ag-0.7Cu solder joint at room temperature, up to 7 times more than that of the control sample when RE content was 0.1 wt.%. As RE exceeded 0.1 wt.%, the lifetime was moving in the direction of decreasing. When RE reached 1.0 wt.%, the lifetime of the SnAgCuRE solder joint was shorter than Sn-3.8Ag-0.7Cu, which was about the same level as Sn-37Pb. At 65°C, the atoms were more thermally activated than at room temperature, and the improvement was not apparent. Under such conditions, as shown in Figure 5b the creep-rupture life of the solder joint increased to only about 2 times of the Sn-3.8Ag-0.7Cu. Both solder joints presented in Figure 5 exhibited much longer creep rupture lives at the optimal RE content as compared to SnPb solder joints under the same testing conditions.[25]

In order to understand if the heavy rare earth Er has a similar effect as light rare earth Ce or La on improving properties of Sn-3.8Ag-0.7Cu solder, creep rupture tests were also conducted on Sn-3.8Ag-0.7Cu-Er solder joints at 50°C under 16.5 MPa.[36] The average creep rupture life was increased up to 6.5 and 7 times that of Sn-3.8Ag-0.7Cu solder joints, when Er content reached 0.1 wt.% and 0.25 wt.%, respectively. As the Er content exceeded 0.25 wt.%, the creep rupture lifetime decreased. The proper Er content in such Sn-3.8Ag-0.7Cu solder alloys hence should be maintained in a range of 0.05–0.25 wt.% to achieve improved creep rupture life.

Systematic constitutive creep studies indicated that the creep deformation of both Sn-3.8Ag-0.7Cu and Sn-3.8Ag-0.7Cu-RE solder joints was controlled by a dislocation climb mechanism at both high and low stress regime. Detailed creep data and constitutive equations can be found in the published literature.[23] In addition to creep rupture lifetime enhancement, another study showed that adding a small amount of the RE elements could evidently improve the creep–fatigue rupture lifetime and the strain at failure of the Sn-3.8Ag-0.7Cu solder joint.[37] The

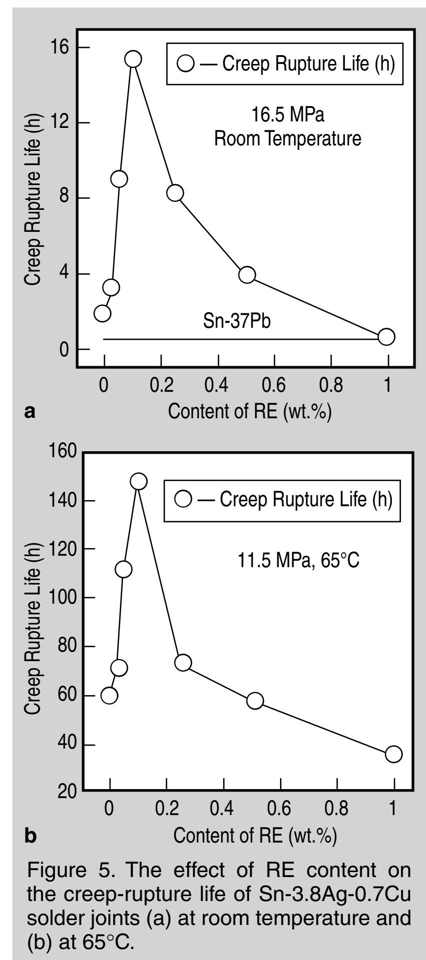

Figure 5. The effect of RE content on the creep-rupture life of Sn-3.8Ag-0.7Cu solder joints (a) at room temperature and (b) at 65°C.

Table II. Results of Tensile Tests of Sn-3.8Ag-0.7Cu-RE Alloys

RE (wt.%)	Ultimate Tensile Strength (MPa)	Elongation (%)	Elastic Modulus (MPa)	Fracture Energy (mJ)	Joint Strength (MPa)
0	43.31	21.0	7,398.0	1,138.20	23.0
0.025	43.77	25.7	7,485.7	1,539.97	23.2
0.05	44.95	27.2	7,531.3	1,649.80	19.4
0.1	45.04	23.5	8,058.0	1,282.03	20.0
0.25	45.17	22.2	10,106.7	1,302.03	19.9
0.5	45.38	16.2	10,787.0	1,025.60	20.3
1.0	45.80	14.8	11,520.0	970.18	11.7

increase in the creep-fatigue property was attributed to the reduction of the creep-fatigue damages and the change of microcrack propagation site during the fracture process, which depended on the refining of the IMC particles by RE addition. Such change in the microcrack propagation site was also observed in the thermomechanical studies of the same RE-containing solder joint.[35]

CONCLUSION

The addition of RE elements did not alter the processing properties, such as melting temperature, wettability, etc. However, RE addition was not able to promisingly solve the oxidation problems that most lead-free solder alloys encountered without further addition of P and Ge elements. In terms of mechanical strength of the RE-containing solders and their joints, RE addition did not show obvious influence on the strength of the Sn-3.8Ag-0.7Cu solder joint, but could provide significant improvement in IMC refinement and creep resistance of the solder joint. Such improvement implies these RE-containing solders could be applied to industrial settings where long-term reliability is a required factor. Since the IMCs in the microstructure could be controlled with the addition of RE and the microcrack propagation path could be altered to a favored direction, primary studies in thermomechanical behavior of RE-containing solder joints exhibited a much slower trend for residual strength deterioration as a function of thermal cycle. It is believed that lead-free solder alloys with RE addition are now ready for reliability studies to prove their feasibility as working solders, especially under severe service conditions.[38]

In spite of the many advantages reported for solders doped with RE elements, abnormally rapid growth of tin whiskers was found to occur during storage in air.[19–21] Our current study has also found that excessive addition of RE element to solders stimulated the growth of tin whiskers on the bulk specimens during storage with various morphologies from the $RESn_3$ phases which acted as the sources for compressive stresses to the neighboring Sn grains.[39–40] Meanwhile, with the miniaturization of electronic components, will RE addition be effective in inhibiting phase segregation and hillock/valley formation induced by electromigration under high current density? Though our preliminary studies in this area provided a positive answer,[41] there are still a significant number of remaining issues regarding the roles of RE-containing phases under a complex service environment, which have to be resolved before the industrial applications are realized.

ACKNOWLEDGEMENTS

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