Application of Spark Sintering to Preparation of Zn-50Sn-Al₂O₃/Cu Joints for AC-Low Voltage Fuse Elements Without Lead

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After the casting and cold drawing of fuse alloys, the joint between fuse alloys and Cu has been soldered using their parts for fabrication of AC-low voltage elements used in electric power line, which means to complex fabrication processes. As case study for the power saving, the Zn-50mass%Sn-8vol%Al₂O₃ and Cu was directly joined using their powders by spark sintering, for development of Pb-free fuse elements, which led to the keep of initial microstructural state because of the un-melting without soldering. The sintering of their powders showed a low sinter-ability because of the large difference in their melting points, which meant the difficulty in application of spark sintering to the fabrication of their joints. However, the joints of Zn-Sn-Al₂O₃ fuse alloy and Cu connectors were prepared successfully by controlling the maximum holding temperature of 433–473 K. The joints were sintered at the solid state of 433 K, showed a little of reaction area at the interface between the fuse alloy and Cu. In contrast, the joints with high relative density which were sintered at the liquid state, showed much amount of reaction area at the interface. The temperature dependence of specific resistivity, thermal conductivity and specific heat was measured for electrical and thermal calculations. The joint sintered at the solid state showed the close values in their properties to the solidified alloy as the reference. Both the melt and un-melt down performance for AC-low voltage fuse elements could be satisfied on the joints of fuse alloy and Cu sintered at the solid state, which resulted in the same performance shown in the standard alloys by complex fabrication processes consisting of casting and soldering as previously manufacturing method. [doi:10.2320/matertrans.MD201512]

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1. Introduction

Lead and its alloys or compounds are considered environmental hazards because of lead's toxicity therefore many countries are going to ban their use^{1,2)}. The practical Pb-Sn alloys used as solders in electrical and electronic industries are classified into two groups (Pb-5mass%Sn and Pb-60mass%Sn) by their melting temperatures. The Sn-40Pb system alloys have been also used as AC-low voltage fuse elements in electric power line^{3,4)}. Due to the world-wide legislative requirements^{5,6)}, it is important to develop viable alternative Pb-free alloys for AC-fuse elements used in electric power line. The main requirements for alternative fusible alloys are:

(1) Low melting point: The melting points should be comparable to practical Sn-Pb system alloys.

(2) Availability: There should be adequate supplies or reserves available of candidate metals.

(3) Ability of manufacture: The production of raw materials should not be difficult.

A eutectic composition in binary alloys consisting of zinc and tin has been investigated in our previous study as a Pb-free alloy for low-voltage fuse elements, except for the points of its performance in a break at high value (3000 A) in electric current, weather proof and wettability on copper^{3,4,7)}. In contrast, since this eutectic point (471 K) is similar to that (456 K) of the practically used Sn-40Pb, it has been also considered by other investigators as a candidate alloy system for a Pb-free solder material^{8,9)}. The Zn-Sn eutectic system which is basically classified as an anomalous eutectic alloy has a

broken-lamellar type eutectic structure¹⁰⁾. The faceting lamellae are Zn and the non-faceting phase is the Sn solid solution. Under rapid cooling conditions, the lamellar Zn becomes fibrous^{10,11)}, which means the sensitivity to solidifying conditions. It is considered that electrical and thermal conductivity of this eutectic system alloys are difficult to be estimated using Maxwell¹²⁾ and Landauer¹³⁾ models, because those properties are directly influenced by morphology of each phase in them as described in Fan's model¹⁴⁾. Also in our proposed estimations¹⁵⁾, not only volume fraction of pure Zn and Sn-solid solution containing Zn of less than 1 mass%¹⁶⁾ but morphologies of both phases were considered in the alloys, which led to excellent estimation for their electrical and thermal properties as the function of the composition and temperature¹⁵⁾. The dependence of solidifying conditions on microstructural morphologies such as eutectic structures are greater and greater as the eutectic amount increases in Zn-Sn system alloys. For instance, both primary Zn and eutectic were observed as the continuous phases in Zn-50Sn alloy¹⁵⁾ which showed the insensitive microstructure depending on the solidification condition, compared with the eutectic composition.

Optimization of fuse elements is carried out by both methods of their alloy- and shape-design. In the shape-design, temperature distributions in the fuse alloy element - Cu connector - electric wire system, have to be exactly known in some conditions evaluating the main requirements (period showing melt or un-melt down and temperature increment at fixed current flow conditions) for AC-low voltage fuse elements⁷⁾. The period for melt down of fuse elements increased on Zn-rich alloys, or the melt down could not be achieved less than the control period, because of small amount of heat gen-

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eration due to small value of the specific resistivity (Sn-9Zn:12.1, Zn-50Sn:9.7, Sn-37Pb: 17.0 $\mu\Omega\text{ cm}$)¹⁵⁾. The values of thermal diffusivity are 4.3×10^{-5} , 4.4×10^{-5} and $2.6 \times 10^{-5} \text{ m}^2/\text{s}$ in Sn-9Zn, Zn-50Sn and Sn-37Pb, respectively, which means almost its same value in both Zn-Sn alloys. In contrast, the control period for un-melt down becomes to be sufficient in Zn-rich alloys, compared with the eutectic composition. Therefore, there is the reciprocal relation between melt and un-melt down performance for the fuse elements, and both electrical and thermal properties must be controlled for the practical application. It is reported that the microstructural, electrical and thermal properties of Zn-50mass%Sn were controlled by addition of 8 vol% Al₂O₃ with high melting point, high and low values in specific resistivity and thermal diffusivity, respectively^{17,18)}. Even in the Zn-50Sn-8vol%Al₂O₃ alloy, the electrical and thermal properties were varied depending on Al₂O₃ distribution states, or microstructural heterogeneity by the partial melt and solidification in the soldering for joining between this alloy and Cu connectors¹⁸⁾. It is considered to be beneficial that joining between Zn-50Sn-8vol%Al₂O₃ and Cu is carried out using their powders by spark sintering^{19–21)}, because of microstructural keeping. Spark sintering¹⁹⁾ is a rapid densification process which has the potential to minimize grain growth by enhancing sinter-ability through both dielectric breakdown of an oxide film between powder particle surfaces by the pulse discharge process²⁰⁾ and rapid heating by unsteady heat flow in the resistance heating process²¹⁾. For manufacturing of their joints, the application of the single manufacturing process only by the spark sintering instead of the complex processes of casting, cold drawing and soldering, leads to both effects of the power saving and the keep of initial microstructures and homogeneous distribution of Al₂O₃ particles in alloys because of an un-melting process without soldering. However, it is considered that their powders showed a low sinter-ability because of the large difference (887 K) in their melting points (Zn-50Sn: 471 K, Cu: 1358 K).

This study aimed to apply the spark sintering to the joining of Zn-50Sn-8vol%Al₂O₃ fuse and Cu connectors without the microstructural change by the partial melting, as one case study of the single process for their manufacturing. The temperature dependence of electrical and thermal properties was experimentally measured on their joints and temperature simulation was carried out using their values, in order to control their properties for the development of lead-free fuse elements used in electric power line.

2. Experimental Procedures

2.1 The construction of a fuse box and model for electrical and thermal calculations

The schematic illustration showing the assembly of some parts in a practically used fuse box⁴⁾ is shown in Fig. 1(a). The fuse box consisted of a fuse element, copper connector, copper sleeve, cover, case and vinyl coated electric copper wire with a diameter of 3.2 mm^{4,7)}. For AC-low voltage fuses used in electric power line, both main requirements are the melt down of a fuse element to the period less than 600 s at 99 A, and the un-melt down for 3 s at 210 A after current discharge of 72 A^{3,4)}. The values of 72 and 99 A correspond to

Fig. 1 (a) The construction of a fuse box. (b) The schematic illustrations showing the size of the copper wire, copper connector and Sn-Zn-Al₂O₃ fuse element in the fuse element-connector-electric wire system for simulation. Units are given in millimeters.

130 and 180% of a rated current, respectively. The assembly of the Zn-50Sn-8vol%Al₂O₃ fuse element, copper connector and electric copper wire, and the size of each object used in this calculation is shown in Fig. 1(b).

A three-dimensional Cartesian coordinate system was used in the fuse element–Cu connector–electric wire system. The governing equations are based on Ohm's and Fourier's laws for electrical and thermal analyses, which can be written as the following eqs. (1) and (2), respectively,

$$\rho_e c_e \frac{\partial E}{\partial t} = \frac{\partial^2 E}{\partial x^2} + \frac{\partial^2 E}{\partial y^2} + \frac{\partial^2 E}{\partial z^2} = 0 \quad (1)$$

where ρ_e , c_e , E and t represented the specific resistivity, capacitance, voltage and time, respectively,

$$\rho c_p \frac{\partial T}{\partial t} = \lambda \left(\frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2} + \frac{\partial^2 T}{\partial z^2} \right) + Q \quad (2)$$

where T and Q represented the temperature and amount of heat generation per 1 sec, respectively. Q and the governing equation for boundary mesh points such as the fuse element/Cu connector, were described in the early report¹⁵⁾. The finite difference method, FDM, was used to solve the above equations for voltage and temperature as a function of time and position.

2.2 Preparation of joints consisting of fuse element/Cu connector

Scaly pre-alloyed Zn-50Sn, spherical Al₂O₃ and atomized

Fig. 2 Scanning electron and optical micrographs showing (a) the morphology and (b) microstructure of used pre-alloyed powders with the composition of Zn-50mass%Sn-8vol%Al₂O₃, and their characteristic X-ray images of (c) Sn and (d) Zn.

Fig. 3 A Scanning electron micrograph showing the morphology of used Cu powders.

Cu powders with the 100, 50 and 50 μm size were used in this experiment. Their appearances are shown in Figs. 2 and 3. The pre-alloyed Zn-50Sn powders consisted of primary Zn and eutectic of Zn and Sn solid solution, and both were present as the continuous phases, seen in Fig. 2(b)–(d).

Both Zn-50Sn and Al₂O₃ powders were ball-milled for 8.6 ks, and they and Cu powders were poured into the graphite die having inner and outer diameters of 10 and 40 mm, and the die was plugged at both ends with graphite punches having a diameter of 10 mm. In more detail, the Zn-50Sn-8vol%Al₂O₃ powders were put between Cu powders in the die, as the simplification of the fuse-Cu connector assembly shown in Fig. 1. It was assumed that final sintered joints with a true density had the diameter of 10 mm and length of 20 mm consisting of the Zn-50Sn-Al₂O₃ fuse and both Cu connectors with the length of 4 and 16 mm, respectively.

The spark sintering process consisted of pulsed electrical discharge and continuously electric current discharge. The direct current pulse of rectangular waves was discharged from the upper electrode to the lower one. The powders were pulse electrical discharged for approximately 0.8–0.9 ks under the conditions of the punch pressure of 15 MPa and the pulse current density of 1.3 A/mm², the pulse-discharge time and -cut time of 100 ms. The compacts were resistance-heated to fixed maximum temperatures of 433 to 473 K and were held at the temperatures at the punch pressure of 50 MPa, in the following continuously electric current discharge. The temperature of compacts was estimated from that of the die, by the method that had been reported earlier²²⁾, and the estimated temperatures on compacts were used in this paper. Both the pulse discharging and continuous current discharging processes were carried out under vacuum conditions less than 10^{−2} Pa.

The microstructural observation of sintered compacts was carried out using an optical microscope and scanning electron microscope.

2.3 Measurement of electrical and thermal properties

The specific resistivity, ρ_e , was simultaneously measured from 293 K to about 420 K by the standard four probes DC-method in air using a computer-controlled equipment. The size of samples was 1 × 1 × 20 mm. The temperature gradient along the length (20 mm) of joints for the measurement of ρ_e was about 5 K. The thermal conductivity, λ , was measured from 293 K to 460 K using samples with the diameter of 10 mm and length of 20 mm, under the steady-state condition in air. The construction of a joint sample, copper heating rod with a cartridge heater and cooling plate and detail proce-

ture were early reported for measurement of the heat conduction¹⁵⁾. The specific heat, c_p , was measured from 293 K to about 420 K at the rate of 2 K/min, in a nitrogen stream, by the differential scanning calorimetry.

3. Results and Discussion

3.1 Sintering behaviors

The changes in temperature to 433 K and displacement between punches meaning the relative density of compacts, with respect to current discharge time, are shown in Fig. 4 (a), as an example of the achievement of solid state sintering below the eutectic temperature of 471 K. The temperature curves consisted of the rising and holding processes showing the unsteady and steady in temperatures in the continuous current discharge after the pulse discharging during 0.8 ks. The temperature increased linearly during the continuous current discharging to approximately 0.2 ks and became almost constant after this period. The displacement between punches, or relative density of a compact increased linearly to the continuous current discharge time 0.3 ks and increased monotonously. The abrupt increase in the density was observed by increment of punch pressure from 15 to 50 MPa in the initial stage of the continuous current discharge at the time of 0.8 ks.

In contrast, the changes in temperature to 473 K and displacement between punches meaning the relative density, with respect to current discharge time, are shown in Fig. 4 (b), as an example of the achievement of partially liquid state sintering above the eutectic temperature of 471 K. The abrupt increase of 1.8 mm in the punch displacement was observed

by the appearance of the liquid phase at the time of 1.9 ks showing the achievement of eutectic temperature.

3.2 The microstructures of spark sintered compacts

Figure 5 shows the outside appearance of Cu-Zn50SnAl₂O₃-Cu joints with the outer diameter of 10 mm and length 20 mm, and the longitudinal sections for obtained three compacts. The length of Zn-50Sn-Al₂O₃ fuse alloy was varied 4.4 to 4.1 mm depending on the maximum holding temperatures of 433 to 473 K, respectively, which corresponded to the change in displacement between punches, or relative density as shown in Fig. 4. Figure 6 shows the typical microstructures of Zn-50Sn-Al₂O₃ and Cu sintered parts. The spark sintering was achieved at the solid and liquid state reflecting to the microstructures of as initial pre-alloys seen in Fig. 2(b), and solidified structures of eutectic liquid for the compacts prepared at 433 K and 473 K, respectively, as shown in Fig. 6(a) and (b). The external shapes of initial particles were shown as the grain boundaries in the solid-sintered Cu part at 433 K. The values in density were 7.1, 7.9 and 8.1 g/cm³ in Cu-Zn50SnAl₂O₃-Cu joints sintered at 433, 463 and 473 K, respectively. The values in relative density were 81, 92 and 95% in sintered Zn50SnAl₂O₃-Cu joint compacts at 433, 463 and 473 K, respectively. In contrast, Zn-50Sn-8vol%Al₂O₃ alloys¹⁸⁾ were already prepared by casting method, having the microstructure shown in Fig. 6(d). This alloy is hereafter called the solidified alloy as a reference, and showed the homogeneous distribution of 8 vol% Al₂O₃ particles in both primary Zn and eutectic. The solidified Zn-50Sn-8vol%Al₂O₃ alloy¹⁸⁾ itself showed the density of 6.8 g/cm³.

Figure 7 shows the interface between Zn-50Sn-Al₂O₃ and Cu compacts. The reaction area was formed at the interface and its thickness increased as the sintered temperature was varied from 433 to 473 K as shown in Fig. 8. The reaction area was intermittently produced and its maximum thickness was 0.1 mm, at the interface of compacts solid-sintered at 433 K. In contrast, the reaction area was spread to the edge of Cu compacts from an origin of the interface, although the volume fraction of reaction phase was gradient-varied in joints liquid-sintered at 473 K. The reaction area consisted of all equilibrium phases at the room temperature in Cu and Sn or Zn binary systems, due to the gradient concentration distribution depending on the diffusion distance of their elements. The solid-sintered joint at 433 K showed mainly the formation of a little amount of CuZn, Cu₅Zn₈ and CuZn₅ phases. In contrast, the partially liquid-sintered joints at 463 and 473 K showed the formation of Cu-Zn system intermetallics (CuZn, Cu₅Zn₈, CuZn₅) and Cu-Sn system intermetallics (Cu₆Sn₅, Cu₃Sn), in the near and distant areas from the interface, respectively, as shown in Fig. 9. The behavior in formation of their intermetallic compounds agrees with open literatures^{23–25)}.

3.3 Electrical and thermal properties

The electrical resistivity, ρ_e , of Cu-Zn50SnAl₂O₃-Cu joints was measured at various temperatures in the range of 293–420 K. Figure 10 shows the temperature- and sintering states-dependence of the ρ_e . In this figure, 4 straight lines approximated by a least squares method using experimental values of ρ_e for each joint mean the qualitative tendency of its

Fig. 4 The relation between temperature or displacement between punches showing densification of powders, and sintering time, during spark sintering consisting of pulse discharge and continuous current discharge heated up to maximum holding temperatures of (a) 433 and (b) 473 K.

Fig. 5 (a) An appearance of spark sintered joint consisting of a Cu – fuse alloy (Zn-50Sn with 8vol%Al₂O₃) – Cu with the diameter of 10 mm and total length of 20 mm, (b), (c) and (d) longitudinal section of the joints sintered at 433, 463 and 473 K, respectively.

Fig. 6 Microstructures of spark sintered compacts of Zn-50Sn with 8 vol% Al₂O₃ prepared at (a) 433 or (b) 473 K, and pure Cu prepared at 433 K. The (d) microstructure of a reference Zn-50Sn with 8 vol% Al₂O₃ alloy¹⁸⁾ prepared by both melted and solidified methods.

change. As can be seen in the figure, the ρ_e increased monotonously with increasing temperature, regardless of sintering states. The solid-sintered joint at 433 K showed the close values in ρ_e , compared with the solidified Zn50Sn-8vol%Al₂O₃

alloy¹⁸⁾ as a reference. The ρ_e of this solidified alloy in Fig. 10 was estimated using the ρ_e of both Zn-50Sn-8vol%Al₂O₃ and pure Cu¹⁸⁾. The ρ_e in each sample is roughly represented as the functions of the temperature using 4 equations in Fig. 10.

Fig. 7 Morphologies showing the reaction area at the interface between Zn-50Sn with 8 vol% Al_2O_3 and Cu prepared at (a) 433, (b) 463 K and (c) 473 K.

Fig. 8 Relation between sintering temperature and thickness in reaction layer between a fuse alloy and Cu connectors

There were large scatters among 4 specimens, because of large amount of intermetallics formation at the interface in the liquid state sintering. It is found that the solid state sintering at 433 K is useful as the spark sintering temperature condition, because of the minimization in formation of reacted phases.

In contrast, the thermal conductivity, λ , of Cu-Zn50Sn Al_2O_3 -Cu joints was measured at various temperatures in the range of 293–460 K. Figure 11 shows the temperature dependence of the λ , and the tendency of its change

using 4 lines in the same manner with Fig. 10. As can be seen in this figure, the λ decreased monotonously with increasing temperature, regardless of manufacturing conditions or methods. The values of λ in the solid-sintered joint at 433 K showed the closer values in λ , compared with the solidified Zn50Sn-8vol% Al_2O_3 alloy¹⁸. The λ in each sample is roughly represented as the functions of the temperature using 4 equations in Fig. 11. The change of the λ can be explained by the difference between both manufacturing condition and method, and the optimum temperature in spark sintering can be decided to 433 K in the same manner with the ρ_e .

The specific heat, C_p , of Cu-Zn50Sn Al_2O_3 -Cu joints was measured at various temperatures in the range of 293–420 K. Figure 12 shows the temperature dependence of the C_p , and the tendency of its change using 4 lines in the same manner with Fig. 10. As can be seen in this figure, the C_p increased monotonously with increasing temperature, regardless of manufacturing conditions or methods. The C_p in each sample is roughly represented as the functions of the temperature using 4 equations in Fig. 12, although there were almost the same values among three sintered joints.

It is found in the following potential and thermal calculations⁷⁾ below the melting point of the Zn-50Sn alloy that the values of ρ_e , λ and c_p for each mesh point can be generally represented using equations listed in Figs. 10 to 12 as the function of the temperature.

Fig. 9 The characteristic X-ray images of (a) Al, (b) Sn, (c) Cu and (d) Zn, and (e) a corresponding SEM image of the interface between Zn-50Sn with 8 vol% Al_2O_3 and Cu in the joint sintered at 473 K.

Fig. 10 Specific resistivity measured in this study for the joints consisting of Cu – fuse alloy (Zn-50Sn with 8vol% Al_2O_3) – Cu prepared at 433, 463 and 473 K, and it of previously reported solidified Zn-50Sn with 8vol Al_2O_3 alloy¹⁸⁾ as a reference.

Fig. 11 Thermal conductivity measured in this study for the joints consisting of Cu – fuse alloy (Zn-50Sn with 8vol% Al_2O_3) – Cu prepared at 433, 463 and 473 K, and it of previously reported solidified Zn-50Sn with 8vol Al_2O_3 alloy¹⁸⁾ as a reference.

Fig. 12 Specific heat measured in this study for the joints consisting of Cu – fuse alloy (Zn-50Sn with 8vol% Al_2O_3) – Cu prepared at 433, 463 and 473 K, and it of previously reported solidified Zn-50Sn with 8vol Al_2O_3 alloy¹⁸⁾ as a reference.

3.4 Temperature simulation under the constant current discharge of both 99 A and 210 A

3.4.1 The constant current discharge of 99 A for the performance of melt down

For AC-low voltage fuses used in electric power line, the fuse element with the diameter of 2.5 mm and length of 10 mm in the smaller diameter part has been designed for the fuse element- connector-electric wire system, as shown in Fig. 1(b)⁴⁾. Figures 10–12 also show the parameters as the function of temperature, used for the electrical and thermal calculations. The interface between Zn-50Sn with 8 vol% Al_2O_3 and Cu in sintered joints showed the some intermetallic compounds with the different amount and distribution depending on sintering temperatures, as shown in Figs. 7 and 8, which meant heterogeneous microstructures through the fuse elements and Cu connectors. In this paper, for convenience, it is assumed that the same values of ρ_e , λ and c_p were used at all mesh points, because of their average values via fuse and connector parts containing heterogeneously precipitated intermetallics, for equations listed in Figs. 10–12. The voltage

and temperature were calculated using eqs. (1) and (2), respectively. The electric current of 99 A was discharged from the edge surface of the copper electric wire to another one, as shown in Fig. 1. The current density on the edge surface of the electric wire was 12.3 A/mm² at room temperature. For instance, the measured value in the total electrical resistance was 706 $\mu\Omega$ throughout the fuse element-connector-electric wire system. The time step was 0.0001 s in temperature calculations. The largest heat source or the maximum value of the temperatures throughout the used system was shown at the center in the smaller diameter part of fuse elements⁷⁾, which meant the melt down at it firstly throughout this system.

The solid-sintered joints at 433 K which were optimized in sintering conditions, showed the close values in ρ_e and λ compared with the solidified Zn50Sn-8vol%Al₂O₃ alloy¹⁸⁾ having the standard microstructure with relative density of 100% as a reference. Therefore, the change in calculated temperature at the center of the fuse element made of the solid-sintered joint at 433 K, is shown in Fig. 13. In this figure, the result of temperature-calculations is also shown, for Zn-50Sn solidified alloys with and without 8 vol%. Al₂O₃ additions and practical Sn-38Pb solidified alloy as references¹⁸⁾. The temperature rose to the eutectic ones as the time increased, for fuse elements made of three alloys, except for Zn-50Sn without Al₂O₃ addition. The temperature in fuse elements made of the Zn-50Sn-8vol%Al₂O₃ solid-sintered and solidified alloys increased to the eutectic temperature for the periods of 200 and 300 s, respectively. This result can be easily explained on the basis of the different values between thermal diffusivity and specific resistivity for both alloys, as shown in Figs. 10–12. In this paper, for convenience, it is assumed that the melt down of fuse elements was caused at their eutectic temperatures. The melt down on the fuse elements after both periods (200 and 300 s) led to the satisfaction of the time limit (less than 600 s) at the melt down examination at 99 A. In contrast, the rate of increase in the temperature decreased with increasing the period in current flow, and the saturated value of the temperature under this

condition was shown below the melting temperature, as the current discharge time increased even to 600 s for the Zn-50Sn without Al₂O₃ under this condition. It is clear that the performance of melt down was satisfied on the Zn-50Sn-8vol%Al₂O₃ sample solid-sintered at 433 K in this study, for the usage of the practically used fuse size⁴⁾ as shown in Fig. 1, although the temperature distribution through fuse elements can be varied even by change in their sizes.

The suitability of the electric and thermal analyses was verified by a following procedure. K-type thermocouples with a diameter of 0.32 mm were spot-welded with metallic surface at the fixed positions of M1 and M2 in Cu wires shown in Fig. 1(a). The temperatures for the Sn-9Zn fuse element-connector-electric wire system were continuously measured by thermocouples at both positions of M1 and M2 under the condition of electric current of 99 A. There was the temperature-difference less than 3 K between the measured and calculated values at M1 and M2 positions shown in Fig. 1(a)(b), at the period showing the melting point at the center of the fuse element. This suggested the high accuracy calculations. The change in calculated temperature at the center in Sn-9Zn fuse element is also represented in Fig. 13. It was reported in the previous paper⁷⁾, that potential and temperature distributions could be also estimated by this calculation method, regardless of a kind and shape of the fuse elements, connectors and electric wires. Therefore, it is considered that the suitability in the potential and temperature calculations was also shown in this study using Zn-50Sn-8vol%Al₂O₃ sample solid-sintered at 433 K.

3.4.2 The constant current flow of 210 A after discharge of 72 A for the performance of un-melt down

The electric current of 72 A was firstly discharged from the edge surface of the copper electric wire to another one, according to performance examination for the un-melt down. The current density on the edge surface of the electric wire was 8.95 and 29.4 A/mm² at 72 and 210 A, respectively, at room temperature.

The change in calculated temperature at the center of fuse elements with respect to current discharge time at a constant current discharge of 72 and 210 A is shown in Fig. 14 for the Zn-50Sn system alloys with and without Al₂O₃ manufactured by solid state sintering or solidification. The saturated values in the temperature were also shown after the current discharge for 360–700 s at 72 A in the calculation⁷⁾, although the constant current of 72 A was discharged via fuse elements for 1 ks in the practical experiments⁴⁾. The constant current of 210 A was discharged via the fuse element-connector-electric wire system after the current discharge of 72 A for 700 s, in the potential and thermal calculations. The temperature at the center of three kinds of fuse elements reached to the eutectic temperature after the current discharge times for 3.1, 3.4 and 13 s, for Zn-50Sn-8vol%Al₂O₃ solid-sintered, solidified Zn-50Sn with and without 8vol%Al₂O₃ alloys, respectively, at 210 A. However, it is considered that the achievement of the un-melt down performance becomes to be difficult on the solid state sintered alloy because of the larger amount of heat generation due to higher value in ρ_e , compared with the solidified alloy, as shown in Fig. 10. The period more than 3 s for the un-melt down requirement at 210 A could be satisfied even on the solid state sintered joint.

Fig. 13 The relation between the temperature and time obtained from the calculations under the constant current flow of 99 A at the center in the fuse element made of the joint consisting of Cu – fuse alloy (Zn-50Sn with 8vol%Al₂O₃) – Cu sintered at 433 K, and, and it of previously reported solidified alloys¹⁸⁾ as references.

Fig. 14 The relation between the temperature and time obtained from the calculations under the constant current flow of 72 and 210 A, for the center in the fuse element made of the joint consisting of Cu – fuse alloy (Zn-50Sn with 8vol%Al₂O₃) – Cu sintered at 433 K, and it of previously reported solidified samples¹⁸⁾ as references.

In the range between the liquidus and eutectic temperature, the presence of both the solid and liquid leads to satisfy the performance of un-melt down under the higher fixed value (210 A) of electric current as one of main requirements for fuse elements^{3,4)}, compared with the Sn-9Zn and Sn-38Pb alloys of eutectic compositions, even in the point of the post-reach of temperature in fuse elements to eutectic one. This is supported by the microstructural characteristic of Zn-50Sn-8vol%Al₂O₃ which means the presence of the primary Zn phase having the large amount and coarse size, as shown in Fig. 6(a), (b). The melting point of the primary Zn phase is higher than one in the eutectic consisting of fine Zn and Sn solid solution phases.

Both the melt and un-melt down performance for AC-low voltage fuse elements could be satisfied on the joints of fuse alloy and Cu sintered at 433 K with a little of reaction area at the interface between the fuse alloy and Cu connector, which resulted in the same performance shown in the reference solidified alloy by complex fabrication processes consisting of casting and soldering as previously manufacturing method.

4. Conclusions

This study aimed to apply the spark sintering to the joining of a Zn-50mass%Sn-8vol%Al₂O₃ fuse alloy and Cu connectors without the microstructural change by the partial melting, as one case study of the single process for their manufacturing. The temperature dependence of electrical and thermal properties was experimentally measured on their joints, in order to control their properties for the development of lead-free fuse elements used in electric power line. The results are summarized as follows:

(1) The joints of Zn-Sn-Al₂O₃ fuse alloy and Cu were prepared successfully by controlling the maximum holding temperature of 433–473 K. The joints were sintered at the solid state of 433 K, showed a little of reaction area at the interface between the fuse alloy and Cu. In contrast, the joints with high relative density which were sintered at the liquid state, showed much amount of reaction area at the interface.

(2) The temperature dependence of specific resistivity,

thermal conductivity and specific heat was experimentally measured for electrical and thermal calculations. The joint sintered at the solid state showed the close values in their properties to the solidified alloy as a reference.

(3) Both the melt and un-melt down performance for AC-low voltage fuse elements could be satisfied on the joints of fuse alloy and Cu sintered at the solid state with a little of reaction area at the interface between the fuse alloy and Cu connector, which resulted in the same performance shown in the standard alloys by complex fabrication processes consisting of casting and soldering as previously manufacturing method.

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