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"Effect of Cu on Resistivity, Microstructure, and Thermal Properties of Sn-3.5Ag Lead-Free Solder Alloys"

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Abstract

The superior mechanical qualities of Sn-Ag-based solder make it a potential alternative to Pb solder. Sn-3.5Ag, Sn-3.5Ag-0.5Cu, and Sn-3.5Ag-0.7Cu were studied for their microstructure, thermal, and electrical properties after quickly solidifying from the melt utilizing a melt-spinning process for intermediate-step soldering. At room temperature and within the temperature range of 340K to 600K, the electrical resistivity of Sn-Ag solder alloys was evaluated using the four-point probe method. The investigation of thermal characteristics was carried out using Differential Thermal Analysis. When measuring the hardness of lead-free solder alloys, researchers found that the as-cast eutectic samples of Sn-3Ag-0.5Cu and Sn-3.5Ag-0.7Cu had melting points ranging from 215°C for the former to 221 oC for the latter. The properties of a commercially available Sn-3.5 wt.% Ag eutectic solder alloy were compared to those of the other solder alloys. Also included are presentations and discussions about micro-structure investigations.

Keywords: Lead-free solder alloys; SEM micrographs; DTA, Hardness; Electrical resistivity.

المستخلص:

إن الصفات الميكانيكية الفائقة للحام المعتمد على Sn–Ag تجعله بديلاً محتملاً للحام الرصاص. تمت دراسة Sn–3.5Ag و Sn–3.5Ag–0.5Cu و Sn–3.5Ag–0.7Cu لمعرفة بنيتها المجهرية وخصائصها الحرارية والكهربائية بعد التصلب السريع من الذوبان باستخدام عملية غزل الذوبان للحام ذي الخطوة المتوسطة. في درجة حرارة الغرفة وضمن نطاق درجة الحرارة من ٣٤٠ كلفن إلى ٦٠٠ كلفن، تم تقييم المقاومة الكهربائية لسبائك اللحام Sn–Ag باستخدام طريقة المسبار رباعي النقاط. تم دراسة الخصائص الحرارية باستخدام التحليل الحراري التفاضلي. عند قياس صلابة سبائك اللحام الخالية من الرصاص، وجد الباحثون أن العينات المصبوبة سهلة الانصهار من Sn–3Ag–0.5Cu و Sn–3.5Ag–0.7Cu لها نقاط انصهار تتراوح من ٢١٥ درجة مئوية للأول إلى ٢٢١ درجة مئوية للسبائك للأخير. وتمت مقارنة خصائص سبيكة اللحام سهلة الانصهار Sn–3.5% Ag المتوفرة تجاريًا مع خصائص سبائك اللحام الأخرى. تتضمن أيضًا العروض التقديمية والمناقشات حول تحقيقات البنية الدقيقة.

الكلمات المفتاحية: سبائك اللحام الخالية من الرصاص، صور مجهرية SEM، DTA، الصلابة، المقاومة الكهربائية.

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1. Introduction

As a substance for mechanically attaching and electrically connecting electronic components in their packaging, soldering is an essential tool in the electronics industry. The success of the assembly' packing is, hence, highly dependent on the soldiers' performance [1,2]. If you're looking for an electronic soldering solution that doesn't use tin-lead, there are a few key features to keep in mind. These include a lower solder melting point, enough strength, low cost, good wetting properties, availability of the base metal in enough quantities, and environmental concerns about toxicity. Rapid solidification, conventional casting, and unidirectional solidification are some of the alloy preparation procedures that primarily impact these qualities. Isothermal heat treatments, ageing time, the working process of the alloys, and isochronal heat treatments are all heavily influenced by the alloys' attributes [3-5].

A vital sensation is the development and use of modern electronics. We can thank the vast world of electronics for every device we rely on on a daily basis. Electronics circuit and hardware assembly relies heavily on the soldering process. Tin-lead solders have a number of disadvantages when used in electronic soldering, including a high melting point, poor electrical and thermal conductivity, high cost, poor wetting properties, and an inadequate supply of the base metal, among other environmental concerns. Rapid solidification, conventional casting, and unidirectional solidification are some of the alloy preparation procedures that primarily impact these qualities. In addition, the workability, isothermal heat treatments, ageing duration, and isochronality of the alloys are all heavily influenced by their characteristics [6,7]. Assembling electronic equipment relies heavily on the soldering process. It is crucial to use high-quality solder materials when creating a solder junction. The variables that influence the quality of a solder junction are the following: strength, melting point, creep resistance, thermal expansion coefficient, creep characteristics, and solder ability. As an alternative to traditional solder, tin alloys show great promise for use in electronics. The most popular kind of solder contains lead, which is extremely harmful to both humans and the environment [8,9]. Developing a solder that does not include lead requires an in-depth understanding of surface characteristics including surface tension and interfacial adhesion. Reason being, these characteristics are recognized to be crucial for wettability and making good solder connections.
Superior electrical and mechanical capabilities, in addition to high reliability, are anticipated from the solder alloys [5]. In order to make binary Pb-free solders more suitable for use in electronic packaging, their characteristics were enhanced by the addition of other alloying elements. It is common practice to add trace amounts of copper to solder alloys in order to alter their melting point, improve their electrical properties, and stop the whisker development. Lower melting point of solder, sufficient strength, environmental concerns regarding toxicity, good electrical/thermal conductivity, cheap cost, good wetting properties, and availability in sufficient quantities of the base metal are some of the important characteristics and properties that are considered when looking for alternatives to tin-lead solders in electronic soldering. Rapid solidification, conventional casting, and unidirectional solidification are some of the alloy preparation procedures that primarily impact these qualities. In addition, the properties of these alloys primarily impact the working process, isochronal heat treatments, iso-thermal heat treatments, and ageing duration, among other parameters [10–12].

Along with mechanical and microstructural qualities, electrical resistivity—the inverse of electric conductivity—is a key metric to evaluate in soldering systems. Even in the solder junctions that connect electronic equipment, a decreased electrical resistivity showed improved electric conductivity flow. To determine the degree of reliability in terms of functionality and the flow current to electrical devices, the solder joint in electrical connections is carefully examined [13–16].

Questions of the study

- How do the thermal properties, as determined by Differential Thermal Analysis (DTA), vary among Sn-3.5Ag, Sn-3.5Ag-0.5Cu, and Sn-3.5Ag-0.7Cu solder alloys?
- What structural changes or phases are identified, and how are they influenced by the presence of copper?
- Are there advantages or disadvantages in the alloys containing copper compared to the commercially used alloy?

2. Objectives of the study

- To determine how do the thermal properties, as determined by Differential Thermal Analysis (DTA), vary among Sn-3.5Ag, Sn-3.5Ag-0.5Cu, and Sn-3.5Ag-0.7Cu solder alloys.
- To clarify the structural changes or phases that are identified, and how they influenced by the presence of copper.
- To show if there are advantages or disadvantages in the alloys containing copper compared to the commercially used alloy.

3. Statement of the problem

A major issue in lead-free solder technology is tackled by the research. Due to environmental concerns, the electronics industry is moving away from lead-based solder, so it's important to find alternatives. An alternative to lead solder with better mechanical characteristics is the Sn-3.5Ag solder alloy. Nevertheless, it is important to comprehend the effects of copper (Cu) addition on important characteristics like thermal behaviour, microstructure, and resistivity. To effectively use lead-free solder alloys in a variety of electronic applications, it is necessary to have a thorough understanding of how Cu affects these critical qualities; however, this information is currently lacking. This research aims to assist in the development of advanced and eco-friendly soldering materials for electronics by studying the thermal properties, microstructure, and resistivity of Sn-3.5Ag solder alloys with different Cu contents. The findings should shed light on the alloys' suitability and performance.

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4. Significance of the study

The study holds significant importance in the realm of materials science and electronic packaging. Lead-free solder alloys, particularly those based on tin and silver (Sn-Ag), have gained prominence as environmentally friendly alternatives to traditional lead-based solders. The addition of copper (Cu) to these alloys introduces a crucial variable, and investigating its impact is pertinent for several reasons. The findings can guide engineers and material scientists in tailoring solder alloys with optimal electrical, thermal, and microstructural properties, thereby contributing to the production of more reliable and environmentally sustainable electronic devices.

5. Theoretical framework

The research is driven by a desire to find lead-free solder replacements that have better mechanical qualities. Sn-Ag is being considered as a possible substitute for the more conventional Pb solder. In order to learn about the microstructure, thermal behaviour, and electrical resistivity of Sn-Ag-based alloys, this study uses a melt-spinning method to quickly solidify Sn-3.5Ag, Sn-3.5Ag-0.5Cu, and Sn-3.5Ag-0.7Cu. Adding Cu as an alloying element makes it possible to measure how it affects the relevant properties.

The electrical resistivity of solder alloys can be determined by measuring them using the four-point probe method throughout a temperature range. To learn how the alloys, react to changes in temperature, Differential Thermal Analysis is used to investigate their thermal properties. In addition, the melting points of various alloy compositions can be better understood by measuring the hardness of as-cast eutectic samples, which provide insights into the mechanical properties. The Sn-3.5 wt.% Ag eutectic solder alloy that is commercially available is used as a comparison [25].

The solder alloys' organization and properties can be visually understood through microstructure research. A thorough comprehension of how copper affects the resistivity, microstructure, and thermal characteristics of Sn-3.5Ag lead-free solder alloys is achieved by comparing the outcomes of these several analyses.

6. Study limitations

Limited Alloy Compositions: The study primarily focuses on three specific alloy compositions (Sn-3.5Ag, Sn-3.5Ag-0.5Cu, and Sn-3.5Ag-0.7Cu). While these variations are informative, a broader range of copper concentrations or additional alloying elements could provide a more comprehensive understanding of the alloy's behavior. The use of a melt-spinning technique for intermediate-step soldering is a distinct method employed in this study. The limitations associated with this technique, such as potential differences in cooling rates or solidification dynamics compared to traditional soldering methods, should be acknowledged.

Limited Temperature Range for Resistivity Measurement: The electrical resistivity measurements are conducted within a specific temperature range (room temperature and 340K-600K). A more extensive temperature profile could reveal nuanced variations and better capture the alloy's electrical behavior across a wider operational range.

Comparison to Commercial Alloys: The study compares the results with commercially available Sn-3.5 wt.% Ag eutectic solder alloy. However, variations in manufacturing processes and alloy compositions between the studied alloys and commercial counterparts may limit the direct applicability of the comparisons.

Environmental Considerations: The study does not explicitly address potential environmental considerations related to the use of these solder alloys. Assessing factors such as recyclability and environmental impact could enhance the study's practical implications.

7. Materials and Methods:

7.1. Preparation of the alloy

The samples were synthesized using Sn, Ag, and Cu that were more than 99.99% pure. To avoid sample oxidation in air, this study's synthesis used alloys made by melting their ingredients in a Pyrex tube under a fluxing agent (Colophony). For about fifteen minutes, the tube was agitated to homogenise the melt as it was melted over a benzene flame. The alloy was obtained by breaking the tube after solidification. To get rid of the extra flux, the ingot was soaked in pure carbon tetrachloride (CCl_4) for long enough. The sample's density was determined by employing the displacement method with CCl_4 as the immersion liquid. Table (1) shows that the results validate the current preparation technique for these alloys.

8. Results and Discussions

8.1. Phase equilibria and alloy design

The phase diagram of every solder system was computed in order to ascertain the optimal solder composition. As shown in Fig. (1), the liquidus temperature decreases to below 221 °C for Cu levels up to 0.7%, but it rapidly rises for Cu contents more than 0.9%. A liquidus temperature of 219 ± 2 °C is shared by the Sn-3.5Ag-(0-1.0) Cu system. It was demonstrated by Park et al. [17] that the reflow method could make use of a solder alloy having a liquidus temperature greater than the reflow temperature. This investigation utilised three different compositions: Sn-3.5Ag, Sn-3.5Ag-0.5Cu, and Sn-3.5Ag-0.7Cu. Sn-3.5Ag, Sn-3.5Ag-0.5Cu, and Sn-3.5Ag-0.7 Cu were chosen as potential Pb-free solder systems by phase diagram calculations.

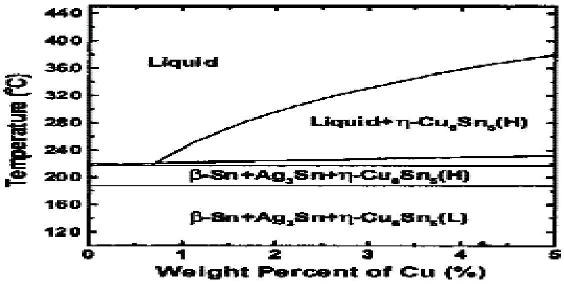


Fig. 1: Calculated phase diagram of Sn-3.5Ag-Cu.

9.2 Thermal properties of Cu-containing Sn–3.5 Ag solders

The melting and solidification behaviours of alloys can be uncovered by analyzing the results of Differential Thermal Analysis (DTA) measurements. Overheating and undercooling data, for instance, can yield liquidus and solidus temperatures. In the DTA thermograms, two distinct phenomena stand out. The first one is the endothermic melting point Tm, which is at the point where the two linear segments that are next to the DTA's transition shoulder meet. The second one has to do with the endothermic zone, which is where melting occurs.

Table 1: Calculated and experimental densities of the pewter Sn solder alloys.

The eutectic alloys (Sn-3.5%Ag-0.5wt.%Cu, Sn-3.5%Ag-0.7wt.%Cu) are depicted in figures (2a, b& c) correspondingly, in the differential thermal analysis thermogram. The melting points of the Sn-3.5, Sn-3.5Ag-0.5Cu, and Sn-3.5 Ag-0.7 Cu alloys are displayed in Table (2), according to the figures.

Table 2: Melting point and heat of fusion of the Sn-Ag solder alloys.

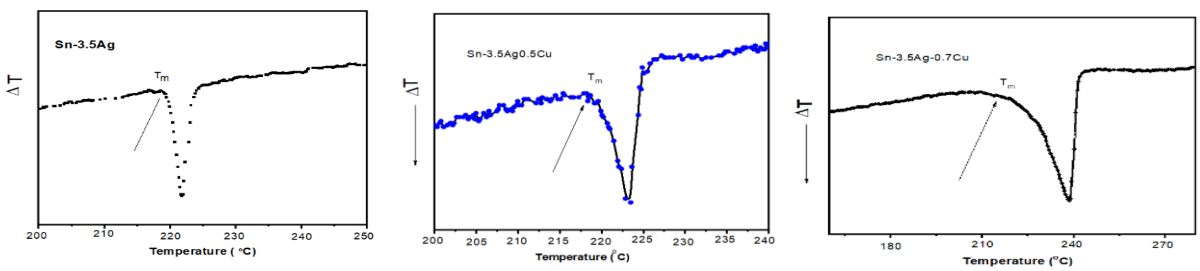
Solder Alloys	M.P (K)	ΔH (cal./gm)
96.5Sn-3.5wt.%Ag	494	8.54
Sn-3.5Ag-0.5Cu	490	6.32
Sn-3.5Ag-0.7Cu	488	6.01

Fig. 2: The differential thermal analysis thermogram of the prepared alloys.

Sample	Alloys	Density calculated	Density Experimental
Sample1	96.5Sn-3.5wt.%Ag	7.04	7.11
Sample2	Sn-3.5Ag-0.5Cu	7.46	7.39
Sample3	Sn-3.5Ag-0.7Cu	7.10	7.01

9.2.1. Calculation of the Enthalpy (Latent Heat of Fusion)

Addition of solute elements to a common solvent causes enthalpy effects, which are well studied. The latent heat of fusion, an indicator of thermal stability, has been determined by computing the enthalpy released during the transformation [18].



The area beneath the DTA peak and the calibration curve of the DTA, with Tin as a reference, were used to estimate the experimental evaluation of the enthalpy (ΔH) during the melting process (ΔH_{ref}). Once the formula is applied:

$$\Delta H = \Delta H_{ref}\ A/M \text{ ---------------(1)}$$

the mass of the sample (M) and the area beneath the DTA peak (A) are defined. Table (2) displays the computed enthalpy ΔH values for the Sn-3.5wt.%Ag, Sn-3.5wt.%Ag-0.5wt.% Cu, and Sn-3.5wt.%Ag-0.7wt.% Cu alloys, respectively.

9.3. The Electrical Resistivity Measurements

The four probe approach was used to study the alloys' temperature dependency d.c electrical resistivity readings. To avoid contact resistance interference, the resistivity of arbitrary shaped specimens can be measured using the four-probe approach. An oven, measuring cell, and sensitive thermocouple were custom-built for the measurements, and the four-probe electrode was used for each. Using shielded wires, the voltage drop and current were measured. We used a Model PS-1830 D constant current D.C. power supply to supply the electric current, and a Model (HP) 425 D.C. micro-voltmeter to measure the voltage drop as a function of temperature. Figure (3) shows the measurement circuit that was utilized. By measuring just, a little fraction of the specimen, the four-probe technique can get an average resistivity reading. Therefore, compared to the two-probe method, this one is better for studying specimen homogeneity.

In measuring the resistivity of large specimens in which the distances between their boundaries and the probes are greater than those between the probes (semi-infinite specimens), the following formula can be used [3-9].

$$\rho = \frac{V_X}{I_X} \frac{2\Pi}{\left(\frac{1}{b_1}\frac{1}{b_3} - \frac{1}{b_1+b_2}\right) - \left(\frac{1}{b_2+b_3}\right)} \text{ ----------------(2)}$$

Where, ρ is the resistivity, in ohm. Cm, V_x is the voltage drop between inner probes 2,3, in Volt; I_x is the current following through outer probes 1,4, in Amp. and b_1, b_2, b_3 are the spacing between the probes, in cm. When the spacing between probes is equal, i.e., $b_1 = b_2 = b_3$ the formula is typically, b = 0.5 cm. $\rho = \frac{V_X}{I_X} 2\pi b.$----------------- (3)

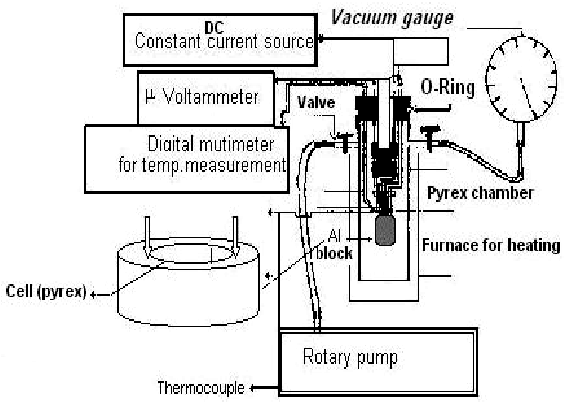
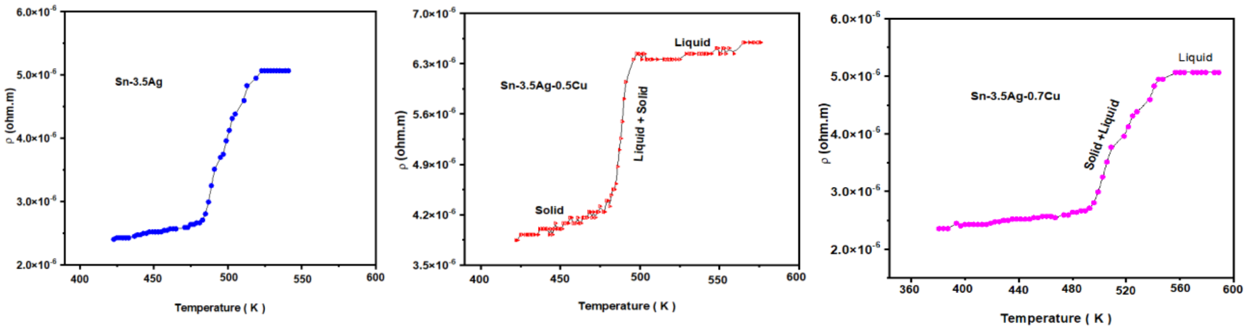


Fig. 3: setup of electrical conductivity.

Impurities and lattice flaws are the primary determinants of a material's electrical resistivity. It is common practice to monitor the melting and precipitation of alloys subjected to heat treatment by measuring their electrical resistivity [18-21]. Ari et al. studied Sn-Ag alloys' thermo-electrical characteristics across a range of compositions. The results showed that the electrical resistivity of the samples rose linearly with temperature, and that the temperature coefficient had nothing to do with the proportion of Sn, Ag, or Cu in the samples [22].

As illustrated in Figure (4), the isothermal electrical resistivity curves for the Sn-3.5%Ag, Sn-3.5%Ag-0.5Cu, and Sn-3.5%Ag-0.7Cu alloys are displayed. Anything that enhances the resistivity, which in turn increases the frequency of electron-ion collisions, A decrease in conductivity can be caused by thermal vibration, foreign atoms in solid solutions, or plastic deformation of the lattice. According to the results shown in figures (4), the electrical resistivity of the melt-spun alloys investigated in this study grows as the temperature increases. When combined with thermal vibration, their effects are amplified. The lattice thermal disturbance at higher temperatures can be modelled as quantized elastic waves or phonons, and the electron-phonon collisions that cause the electrical resistivity to increase can be seen graphically.



Cell (pyrex) Holder (Aluminum)

Fig. 4: The electrical resistivity behavior of the prepared samples.

Table 3: Latent Heat (Q) from Resistivity Measurement for Sn solder Alloys.

Solder Alloys	ρ_L/ρ_S	Q (cal/gm)	dρ/dT (Ω.m/deg) Liquid	dρ/dT (Ω.m/deg) Solid
Sn-3.5Ag	2.4	3.51	5.1×10^{-6}	2.5×10^{-6}
Sn-3.5Ag-0.5Cu	3.8	3.91	6.42×10^{-6}	3.81×10^{-6}
Sn-3.5Ag-0.7Cu	6.1	4.51	5.2×10^{-6}	2.5×10^{-6}

Figure (4) shows curves that summarize the electrical resistivity behaviour of Sn-Ag-Cu alloys. There is a temperature dependence of the electrical conductivity of metals. The behaviour of the resistivity versus temperature is typically used to describe this change. While ρ has a small and relatively steady value between 360 and 470 K, it begins to increase with T at a slower rate but then follows a linear relationship as the temperature rises over that range. This practically constant linear behaviour maintains itself all the way up to the melting point [20] The reasons why pure crystals can nevertheless experience scattering are as follows: (i) the ion cores can vibrate at their equilibrium position regardless of the temperature; (ii) alien atoms or impurities can be present; and (iii) the crystal can have lattice flaws. An rise in ρ (T) due to electron-phonon scattering is a physical manifestation of the thermal disturbance at higher temperatures, which can be defined in terms of quantized elastic waves or phonons. Foreign atoms in solid solution in the matrix metal create the residual resistivity ρ. The high resistivity of Sn-Ag-Cu alloys is attributable to the higher dispersion of conduction electrons caused by the random arrangement of the atoms.

This study demonstrates that, at temperatures below the melting point, the solid's resistivity is controlled by the amount of impurities, dislocations, and residual scattering of conduction electrons caused by crystal defects. Another factor that contributes to resistivity as temperature increases is thermal lattice vibrations, which are caused by electron phonon scattering mechanisms. All of these contributions, known as "disorder scattering," are likely to grow as the material melts; in fact, the resistivity typically doubles during melting [19]. On the other hand, Mott [23] proposed a formula that might link the ratio of liquid to solid resistivities (ρ_L/ρ_S) to the latent heat of melting (Q) using the following equation:

$$Q = \frac{3}{2} k T_m . Ln\left(\frac{\rho_L}{\rho_S}\right) \text{-----------------(4)}$$

in where ρ_L denotes the sample's resistivity in its liquid state and ρ_S its resistivity in its solid state, k is the Boltzmann constant, and Tm is the sample's melting temperature. The data that is now available [21] show that the impact of metallic impurities on the resistance of liquid metals is far smaller than that of solid materials. The most up-to-date quantum electronic theory of metals in liquid state also confirms this result [20]. For both pure metals and Sn-Ag-Cu alloys, Table (3) displays the ratio of the liquid resistivity to the solid resistivity (ρ_L/ρ_S). Experiments have shown that the scattering mechanisms do not interact with one other; rather, the overall resistivity of a metal is the product of its impurity content, plastic deformation, and thermal vibrations. The following is a mathematical representation of this:

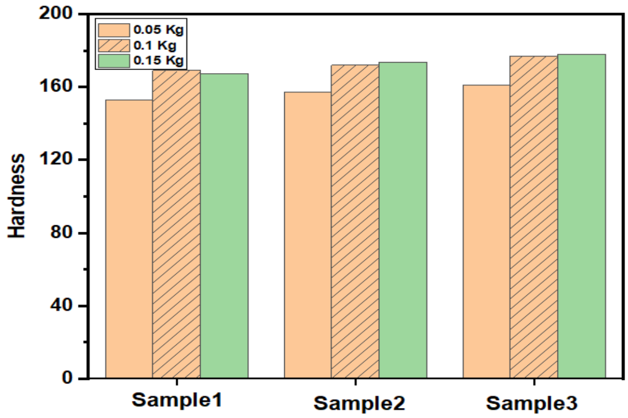
$$\rho_{total} = \rho_T + \rho_i + \rho_d \qquad \text{-----------------(5)}$$

in which ρ_T, ρ_I, ρ_d represent the individual thermal, impurity, and deformation resistivity contributions, respectively. From resistivity measurements, it is found experimentally that bismuth atoms, as semimetal, act as scattering centers, and increasing the concentrations of bismuth results in an enhancement of resistivity.

9.4 Hardness Measurement

When it comes to detecting structural changes in various soft solders, microhardness testing is incredibly sensitive. It is often the simplest method to find out the mechanical properties of the various parts of a building, and it is also non-destructive. The Vickers hardness number of Sn-Ag alloy samples varied with loads of 0.05, 0.1, and 0.15 Kg, as shown in Figure 5.

Fig 5: Variation of Vickers hardness



9.5 internal Friction

rapidly solidified alloys. It shows that, the value of internal friction changes with the alloying elements. It increases to the maximum value when adding Ag, and decreases to the minimum value when adding Cu fig.(6). This variation can be attributed to the change of the dissolving atoms in the Sn-matrix as substitutional solid solutions; which have different mobility due to the change of their atomic radii and the change of the axial ratio that may hinder the motion of substitutional atoms from one site to another. The change of the dissolving atoms in Sn-matrix as substitutional solid solutions, which have different mobility's due to the change of their atomic radii, causes the change of internal friction of these alloys.

9.6 Microstructure

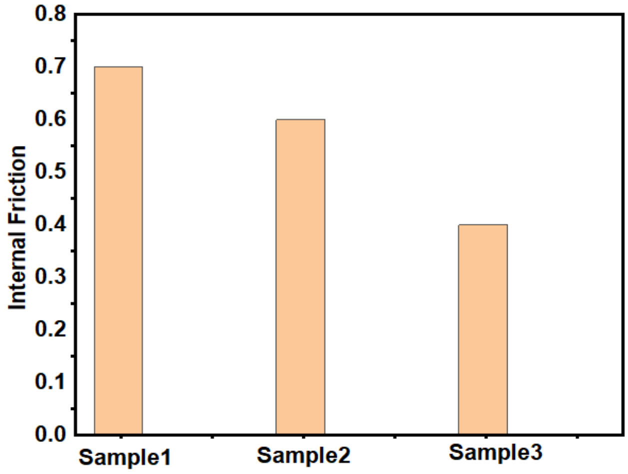
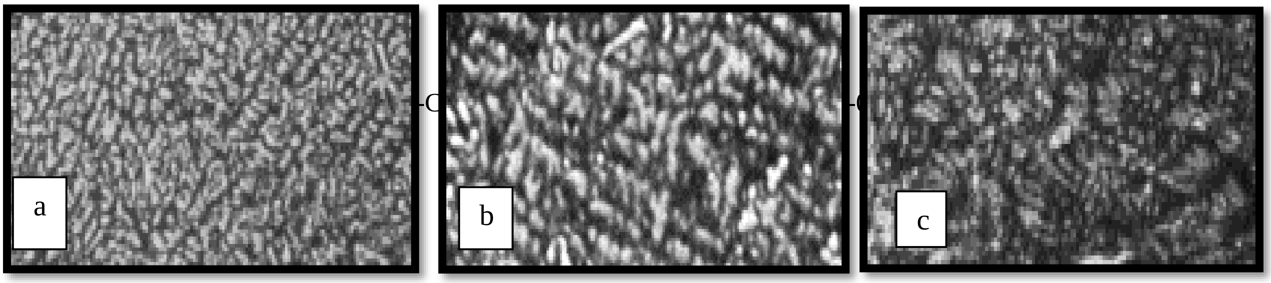


Figure 6: shows the variation of internal friction values of the prepared samples.

The microstructure of the solder is shown in Figure (7). Three solder compositions, namely Sn-3.5Ag, Sn-3.5Ag-0.5Cu, and Sn-3.5Ag-0.7Cu, were selected by phase equilibria calculations. The microstructure of solders gets finer and its distribution is more homogenous as the cu content increases, as illustrated in Fig. (7). A tiny amount of Cu creates a nucleation centre that is not uniform, which alters the crystal growth velocities and different crystallographic directions. Sn-3.5Ag has an irregular, block-like microstructure, as seen in Fig. (7a). Figure 7b shows substantially finer crystal grain than Figure 7a when the Cu concentration is 0.5 wt%. The shape changes to that of a bar or a short rod when the Cu content reaches 0.7% by weight, as seen in Figure 7c. The microstructure grows ever more fine-grained as the Cu content increases. As can be seen in Figure 7c, the microstructure becomes more homogeneous and eutectic as the Cu level increases.



9. Conclusion

The present study effectively synthesised lead-free solder alloys of Sn-3.5Ag, Sn-3.5Ag-0.5Cu, and Sn-3.5Ag-0.7Cu by quickly solidifying Sn-Ag melt using the melt-spinning approach. Results from the investigation of the material's microstructure, thermal characteristics, and electrical characteristics led to the following conclusions.

- The data on the typical peak temperatures of the Sn-Ag-Cu alloys that were studied (containing a high percentage of tin and minor percentages of silver and copper) were derived from the DTA curve. According to DTA curves, when the copper content of an alloy increases, the starting and ending points of phase transformation are shifted to lower temperatures. Their suitability for high-reliability component packaging is enhanced by the fact that their melting point drops from 221°C to 215°C as the Cu content increases. Using a Pb-free assembly solder to assemble the parts could be a safe option as well. Assembly temperatures up to 260 °C are usually necessary for these solders.
- While compared to Pb-Sn, the observed changes in the range of heats of fusion are lower. Thus, Sn-Ag-Cu alloys are the best material for reducing energy use.
- Electrical resistivity is a property that is quite sensitive to structural factors, and its addition of Ag content changed it. Adding Cu to Sn-Ag alloy makes it stronger, more resistant to wear and tear, and easier to work with by making the structure more uniform and fine-grained.
- The qualities that make these alloys useful for soldering technology are enhanced when Cu is added to the quickly solidifying Sn-Ag alloy.

Credit for author statement

Conceptualization S.E.N.; methodology, investigation, data curation, project administration, , S.I.; review and editing and A.A.B.; resources, supervision and visualization.

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