

# Microstructural Refinement and Mechanical Strengthening of SAC305 Lead-Free Solder Through Cerium Microalloying

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## Research Article

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# “Microstructural Refinement and Mechanical Strengthening of SAC305 Lead-Free Solder Through Cerium Microalloying”

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## Abstract:

The effect of cerium (Ce) microalloying on the microstructural evolution, thermal behavior, and mechanical properties of SAC305 lead-free solder was investigated to establish quantitative correlations between microstructure and mechanical performance. SAC305 alloys containing 0.2, 0.3, and 0.5 wt.% Ce were systematically investigated. The addition of 0.2 wt.% Ce produced the greatest microstructural refinement, reducing the  $\beta$ -Sn volume fraction from  $35 \pm 3\%$  to  $26 \pm 2\%$  and the average  $\beta$ -Sn grain size from  $20 \pm 2 \mu\text{m}$  to  $10 \pm 2 \mu\text{m}$ . This refinement resulted in the highest tensile strength (51.5 MPa), toughness ( $12.9 \text{ MJ m}^{-3}$ ), and hardness (18.5 HV), corresponding to improvements of 22.6%, 30.3%, and 34%, respectively, compared with the base SAC305 alloy. Higher Ce additions diminished the refinement effect, leading to coarser  $\beta$ -Sn regions and reduced mechanical performance. DSC analysis revealed negligible changes in melting behavior, with peak melting temperatures remaining between 222.07 and 223.40 °C, indicating compatibility with conventional reflow soldering. Overall, 0.2 wt.% Ce provides the optimum balance of microstructural refinement, mechanical strengthening, and thermal stability in SAC305 solder.

**Keywords:** Cerium microalloying, Lead-free solder, Microstructural refinement,  $\beta$ -Sn phase evolution, Mechanical strengthening.

## Introduction

Electronic packaging technology is essential enabling platform for modern electronic systems, ensuring electrical connectivity, mechanical integrity, and thermal management throughout the service life of electronic devices. With the development of advanced integration, miniaturization, and multifunctionality in electronic products, the reliability of solder joints has become a key factor affecting device performance and longevity [1]. Solder materials are desired to sustain complex

service conditions involving thermal cycling, mechanical loading, high humidity, and corrosive environments, which collectively impose strict requirements on their microstructural stability and functional properties [1–4].

For many decades, Sn–Pb solders were widely employed in electronic packaging because of their excellent wettability, low melting temperature, stable microstructure, and favorable mechanical properties [5–8]. However, the toxicity of lead and its persistence in the environment pose serious threats to human health, resulting in strict global regulations such as the Restriction of Hazardous Substances (RoHS) directive, which restricts the use of Pb-containing materials in electronic products [5–10]. As a result, wide research efforts have been dedicated toward the development of environmentally friendly lead-free solder alloys to substitute conventional Sn–Pb systems. A wide range of lead-free solder systems have been examined, including Sn–Bi [11,12], Sn–Zn [13], Sn–Cu [14], Sn–Ag [15], and Sn–Ag–Cu (SAC) alloys [16,17]. Among these, SAC alloys particularly SAC305 have been documented as the most promising candidates for widespread industrial application due to their relatively balanced mechanical properties, good creep resistance, and acceptable thermal fatigue behavior [18–20]. Therefore, SAC305 has been extensively adopted in consumer electronics, automotive electronics, and communication devices.

Despite these advantages, SAC305 solder alloys still suffer from several basic limitations. The relatively high melting temperature ( $\sim 217^\circ\text{C}$ ) requires high reflow temperatures, which can persuade thermal stress, warpage of printed circuit boards (PCBs), and degradation of temperature-sensitive components [21–23]. In addition, SAC305 typically shows coarse  $\beta$ -Sn grains and rapid growth of intermetallic compounds (IMCs) such as  $\text{Cu}_6\text{Sn}_5$  and  $\text{Cu}_3\text{Sn}$  at the solder/substrate interface, which badly affect mechanical reliability and long-term service performance [18–20]. It has been reported that diminish soldering temperatures by approximately  $70^\circ\text{C}$  can lower  $\text{CO}_2$  emissions by nearly 35% and reduce PCB and substrate warpage by more than 50%, highlighting the importance of solder alloy alteration strategies aimed at lowering processing temperatures and improving reliability [24].

To overcome the limitations of SAC305, alternative lead-free solder systems such as Sn–Bi, Sn–Zn, and Sn–Cu alloys have been extensively investigated [11–14]. Sn–Bi solders offer low melting temperatures and relatively high strength but suffer from brittleness and phase coarsening during service, which can reduce mechanical reliability [25–30]. Sn–Zn alloys exhibit favorable melting characteristics and low cost; however, their practical application is limited by oxidation, poor wettability, and corrosion susceptibility [31–34]. Sn–Cu solders are economically attractive and widely used in wave soldering processes, yet they show relatively high melting temperatures, lower wettability, and excessive intermetallic compound growth that can adversely affect joint performance [35,36]. These limitations highlight the need for effective alloy modification strategies capable of enhancing the microstructural stability and reliability of lead-free solder systems.

Microalloying has emerged as an effective strategy for enhancing the performance of Sn-based lead-free solders. Trace additions of alloying elements can alter solidification behavior, regulate intermetallic compound growth, and improve mechanical, thermal, and corrosion properties. Various alloying additions, including In, Bi, Zn, Sb, Co, Ni, Fe, Mg, and Mo, have been reported to improve solder reliability through microstructural refinement and interfacial modification [31–34,37–39]. Among these, indium has attracted considerable attention because it can decrease melting temperature and improve ductility, wettability, and thermal cycling reliability [40–42]. However, its relatively increase cost and concerns regarding long-term phase stability have encouraged the exploration of alternative microalloying elements with comparable benefits.

In recent years, rare-earth (RE) elements have attracted increasing attention as microalloying additions for Sn-based lead-free solders because they can influence solidification behavior, modify interfacial reactions, and improve microstructural stability. Among these, cerium (Ce) is particularly attractive due to its reported ability to refine  $\beta$ -Sn grains, regulate intermetallic compound morphology, and improve microstructural stability. Recent studies have demonstrated that RE-containing microalloying strategies can modify the thermal, microstructural, and interfacial characteristics of SAC-based solders, while reviews have emphasized the importance of microstructural control for improving the corrosion resistance and reliability of SAC alloys [43,44].

Despite these advances, quantitative relationships between Ce-induced microstructural evolution and the resulting thermal and mechanical response of SAC305 solder remain insufficiently explored. Therefore, the present study systematically evaluates the influence of Ce addition on  $\beta$ -Sn volume fraction,  $\beta$ -Sn grain size, eutectic morphology, melting behavior, and mechanical properties to establish quantitative correlations between  $\beta$ -Sn phase evolution and the resulting thermal and mechanical performance and to identify the optimum Ce concentration for SAC305 solder alloys.

## **2 Experimental procedures:**

### ***2.1 Alloy processing***

To investigate the effect of Ce microalloying on SAC305 solder, four solder alloys were prepared from high-purity elemental constituents. These included the base SAC305 alloy and SAC305 alloys containing nominal Ce additions of 0.2, 0.3, and 0.5 wt.%. High purity elemental constituents of tin (Sn, 99.99 wt.%), silver (Ag, 99.99 wt.%), copper (Cu, 99.99 wt.%), and cerium (Ce, 99.9 wt.%) were used for alloy preparation. To ensure accurate control of alloy composition, the required amounts of Sn, Ag, Cu, and Ce were weighed according to the nominal alloy compositions listed in Table 1 using a high-precision microbalance.

*Table 1. Chemical compositions of SAC305 and SAC305–Ce solder alloys (wt.%)*

| <b>Alloy composition</b> | <b>Sn (wt.%)</b> | <b>Ag (wt.%)</b> | <b>Cu (wt.%)</b> | <b>Ce (wt.%)</b> |
|--------------------------|------------------|------------------|------------------|------------------|
| SAC305                   | 96.5             | 3.0              | 0.5              | 0                |
| SAC305–0.2Ce             | 96.3             | 3.0              | 0.5              | 0.2              |
| SAC305–0.3Ce             | 96.2             | 3.0              | 0.5              | 0.3              |
| SAC305–0.5Ce             | 96.0             | 3.0              | 0.5              | 0.5              |

The weighed elements were placed in a graphite crucible and melted in an electric resistance furnace. To minimize oxidation during alloy preparation, the furnace chamber was purged with high purity argon gas prior to melting. The furnace temperature was increased to 450 °C to ensure complete melting of all constituent elements. During melting, the molten alloy was mechanically stirred using a quartz rod to improve compositional uniformity. Each alloy was re-melted three times to minimize compositional variations and improve chemical homogeneity prior to casting. The molten alloy was then poured into a pre-designed metallic die to produce miniature dog bone shaped tensile specimens. Owing to the limited volume of the laboratory cast solder alloys, customized tensile specimens with an overall length of 40 mm, gauge length of 10 mm, and gauge width of 4 mm were employed instead of standard ASTM-sized specimens. All alloy compositions were cast using the same die, pouring temperature, and ambient laboratory conditions to ensure consistent solidification conditions. The specimens were allowed to solidify naturally inside the metallic die at room temperature ( $25 \pm 2$  °C), without the application of forced air or water cooling.

No post-casting heat treatment, solution treatment, annealing, or artificial aging was performed. All specimens were mechanically tested and characterized in the as-cast condition.

## **2.2 *Microstructure and phase characterization:***

Metallographic specimens were prepared using standard metallographic procedures. The specimens were first hot-mounted in Bakelite resin and subsequently ground using silicon carbide (SiC) abrasive papers of progressively finer grit sizes. Final polishing was performed using diamond suspensions to obtain a mirror-like surface finish. The polished specimens were then chemically etched using a solution containing 95% ethanol and 5% hydrochloric acid (HCl) to reveal the microstructure. The microstructural characteristics of the alloys were examined using scanning electron microscopy (SEM), while energy-dispersive X-ray spectroscopy (EDX) attached to the SEM was employed to determine the elemental composition and distribution of the different phases. Phase identification was carried out using X-ray diffraction (XRD), with diffraction patterns recorded over a  $2\theta$  range of 5°–100° to identify the  $\beta$ -Sn matrix and intermetallic phases present in the alloys. Quantitative microstructural analysis was performed using ImageJ image analysis software. Five SEM micrographs obtained from different regions of each alloy were analyzed to ensure representative measurements. The  $\beta$ -Sn phase was segmented using grayscale thresholding, and its measured area fraction was assumed to represent the corresponding volume fraction according to stereological principles. The average  $\beta$ -Sn grain size

was determined from the equivalent circular diameter of the identified  $\beta$ -Sn regions, and the results are reported as the mean  $\pm$  standard deviation (SD).

### 2.3 Mechanical testing:

Uniaxial tensile tests were performed using a universal testing machine at a constant crosshead speed of **0.5 mm min<sup>-1</sup>**. Owing to the limited dimensions of the laboratory-cast solder alloys, customized miniature dog-bone-shaped tensile specimens with a gauge length of **10 mm** were employed instead of standard ASTM specimens, as shown in **Figure 1**. The tensile testing procedure followed the general principles of ASTM E8/E8M; however, a customized miniature specimen geometry was employed because of the limited dimensions of the laboratory-cast solder alloys. During testing, the applied load (N) and elongation (mm) were continuously recorded and subsequently converted into engineering stress–strain curves using standard engineering relationships. The ultimate tensile strength (UTS), elongation to fracture (ductility), and toughness, defined as the area under the engineering stress–strain curve, were determined from the resulting stress–strain curves. The tensile testing conditions are summarized in **Table 2**.

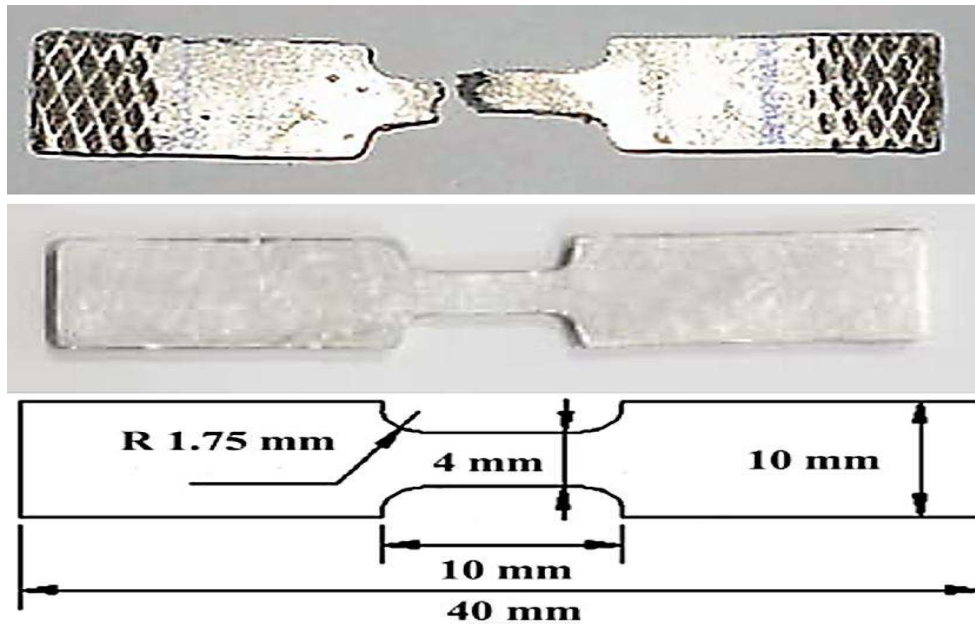


Figure 1. Tensile test specimen geometry and representative tensile samples before and after testing

Table 2. Tensile testing parameters used for mechanical characterization of SAC305 and Ce-modified SAC305 alloys

| Specimen Type   | Gauge Length | Crosshead Strain Rate | Speed / | Environmental Conditions | Data Measurement          |
|-----------------|--------------|-----------------------|---------|--------------------------|---------------------------|
| Dog-bone shaped | 10 mm        | 0.5 mm/min            |         | Room temperature (~25°C) | Load (N), elongation (mm) |

The microhardness of the SAC305 and Ce-modified SAC305 alloys was measured using a Vickers microhardness tester. Prior to testing, the specimens were ground using progressively finer silicon carbide (SiC) abrasive papers and subsequently polished to obtain a mirror-like surface finish. Micro-Vickers hardness measurements were performed using a load of 100 gf with a dwell time of 15 s at room temperature. A minimum of three indentations were made at different locations on each specimen to minimize the influence of local microstructural heterogeneity, and the average value was reported. The reported hardness values represent the mean  $\pm$  standard deviation (SD) and are expressed in Vickers hardness (HV). To ensure the reproducibility of the mechanical properties, at least three independent tensile specimens were tested for each alloy composition under identical experimental conditions. The reported values of UTS, ductility, and toughness represent the mean  $\pm$  standard deviation (SD) of three independent measurements.

#### **2.4 Differential Scanning Calorimetry:**

Differential scanning calorimetry (DSC) was performed to evaluate the melting behavior of the as-cast SAC305 and Ce-microalloyed SAC305 solder alloys containing 0.2, 0.3, 0.4, and 0.5 wt.% Ce. Small specimens weighing approximately 8–12 mg were sectioned from the bulk alloys, mechanically cleaned to remove surface oxides and contaminants, and sealed in standard hermetic aluminum DSC pans, with an empty sealed pan used as the reference. The measurements were carried out using a differential scanning calorimeter under a high-purity nitrogen atmosphere (50 mL min<sup>-1</sup>) to minimize oxidation during heating. Prior to testing, the instrument was calibrated for temperature and heat flow using high-purity indium and zinc standard reference materials. Each specimen was heated from 30 °C to 300 °C at a constant heating rate of 10 °C min<sup>-1</sup>. The onset melting temperature (Tonset), peak melting temperature (Tp), endset melting temperature (Tendset), melting interval ( $\Delta T = \text{Tendset} - \text{Tonset}$ ), peak heat flow, and enthalpy of fusion ( $\Delta H$ ) were determined from the heating curves using the instrument's analysis software. The onset and endset temperatures were obtained using the tangent intersection method, while the enthalpy of fusion was calculated from the integrated area under the melting endotherm after baseline correction. Three independent specimens were analyzed for each alloy composition to ensure repeatability.

### **3 Results and Discussions:**

#### **3.1 Microstructural Analysis (SEM / EDX / XRD)**

The microstructure of the SAC305 solder alloys was characterized using SEM, EDX, and XRD to evaluate the influence of Ce microalloying on solidification behavior. As shown in Figure 2(a–b), the as-cast SAC305 alloy consists of relatively coarse  $\beta$ -Sn regions surrounded by a discontinuous eutectic network containing Ag<sub>3</sub>Sn intermetallic compounds. The addition of 0.2 wt.% Ce noticeably refined the microstructure (Figure 2(c–d)), producing a finer and more homogeneous eutectic network together with a substantial reduction in coarse  $\beta$ -Sn regions. This observation

suggests that Ce influences the solidification behavior by promoting  $\beta$ -Sn grain refinement and restricting the growth of primary  $\beta$ -Sn. Similar refinement behavior has been reported for rare-earth-modified Sn-based solders, where Ce additions have been associated with enhanced heterogeneous nucleation and suppression of  $\beta$ -Sn grain growth [45]. However, the refinement efficiency decreased as the Ce content increased beyond 0.2 wt.%. As shown in Figure 2(e–h), progressively larger  $\beta$ -Sn regions reappeared in the SAC305–0.3Ce and SAC305–0.5Ce alloys, resulting in a less homogeneous microstructure. The EDX results indicate localized Ce enrichment in these alloys, suggesting that excess Ce may become non-uniformly distributed during solidification because of its limited solubility in the Sn matrix. The reduced refinement efficiency is consistent with this localized Ce enrichment, which may reduce the effectiveness of Ce as a grain-refining addition. Consequently, the reappearance of larger  $\beta$ -Sn regions at higher Ce contents supports the conclusion that excessive Ce additions diminish the grain-refining efficiency of SAC305 solder.

Quantitative image analysis (Figure 3 and Table 3) further supports the SEM observations. The  $\beta$ -Sn volume fraction decreased from  $35 \pm 3\%$  in the base SAC305 alloy to  $26 \pm 2\%$  following the addition of 0.2 wt.% Ce, while the average  $\beta$ -Sn grain size decreased by approximately 50%, from  $20 \pm 2 \mu\text{m}$  to  $10 \pm 2 \mu\text{m}$ . The concurrent reduction in  $\beta$ -Sn volume fraction and grain size indicates that the addition of 0.2 wt.% Ce is associated with the suppression of coarse primary  $\beta$ -Sn and the development of a finer and more homogeneous eutectic microstructure. This refinement improves the microstructural uniformity by reducing the size and spatial continuity of coarse  $\beta$ -Sn regions while promoting a more uniformly distributed eutectic structure. As the Ce content increased to 0.3 wt.% and 0.5 wt.%, both the  $\beta$ -Sn volume fraction ( $32 \pm 2\%$  and  $39 \pm 3\%$ ) and the average  $\beta$ -Sn grain size ( $14 \pm 2 \mu\text{m}$  and  $21 \pm 3 \mu\text{m}$ ) increased, demonstrating a progressive reduction in refinement efficiency. These quantitative results indicate that 0.2 wt.% Ce provides the highest degree of microstructural refinement among the investigated compositions, yielding the lowest  $\beta$ -Sn volume fraction and average grain size.

The local EDS point analysis provides complementary compositional information to support the observed microstructural evolution, as summarized in Table 4. No detectable Ce signal was observed in the SAC305–0.2Ce alloy, most likely because the Ce concentration was below the detection limit of conventional SEM–EDS. In contrast, measurable Ce concentrations of approximately 1.40 at.% and 2.20 at.% were detected in selected regions of the SAC305–0.3Ce and SAC305–0.5Ce alloys, respectively. These regions also exhibited relatively higher oxygen contents, indicating a possible association between Ce and oxygen-containing phases or surface oxidation products. However, the chemical state of Ce cannot be determined from EDS analysis alone. The localized increase in measured Ce concentration at higher Ce additions is consistent

with localized Ce enrichment, which may contribute to the reduced grain-refining efficiency observed in the corresponding SEM micrographs.

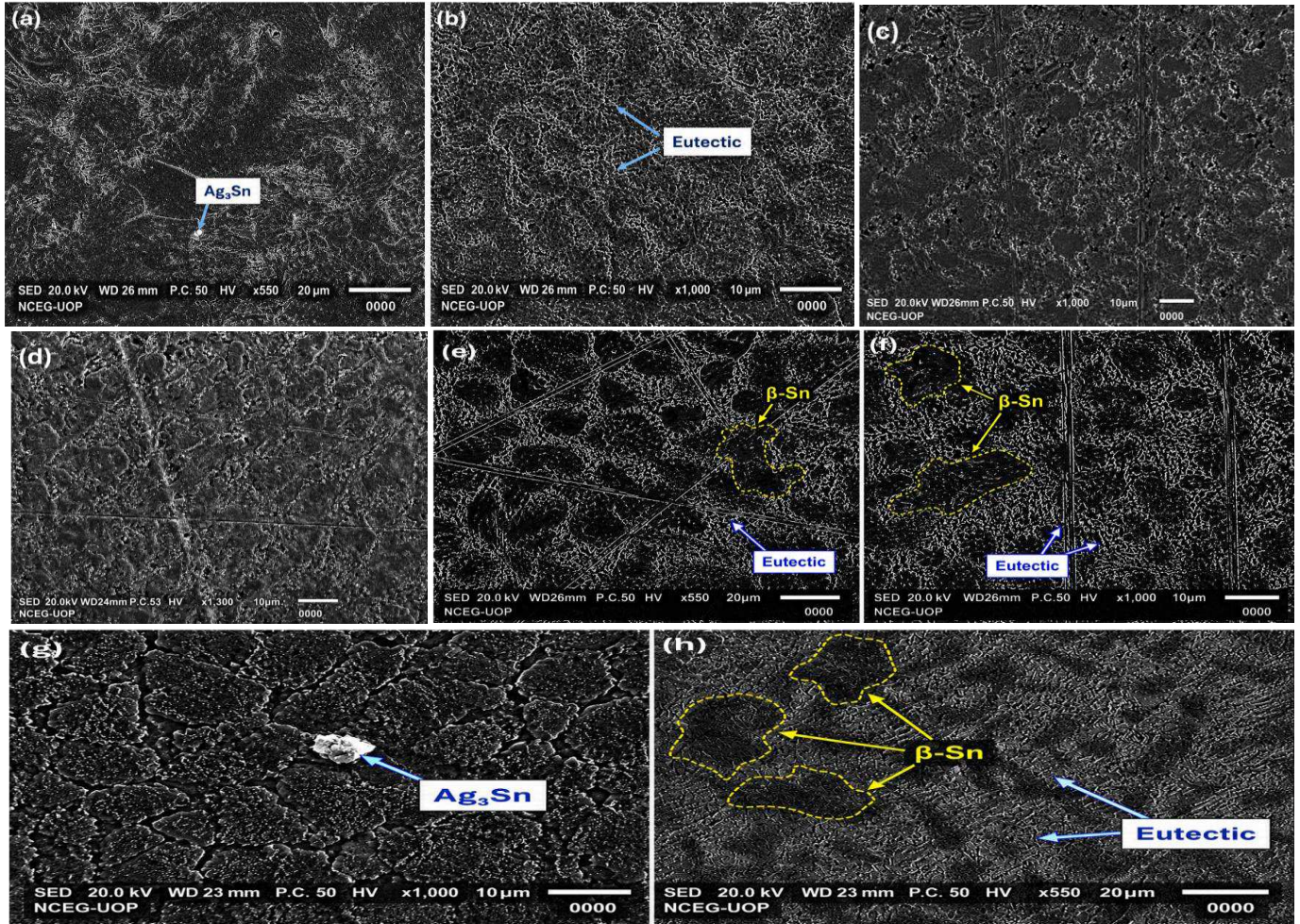


Figure 2. Microstructures of alloys (a,b) SAC305 (c,d) SAC305-0.2% Ce (e,f) SAC305-0.3% Ce and (g, h) SAC305-0.5% Ce

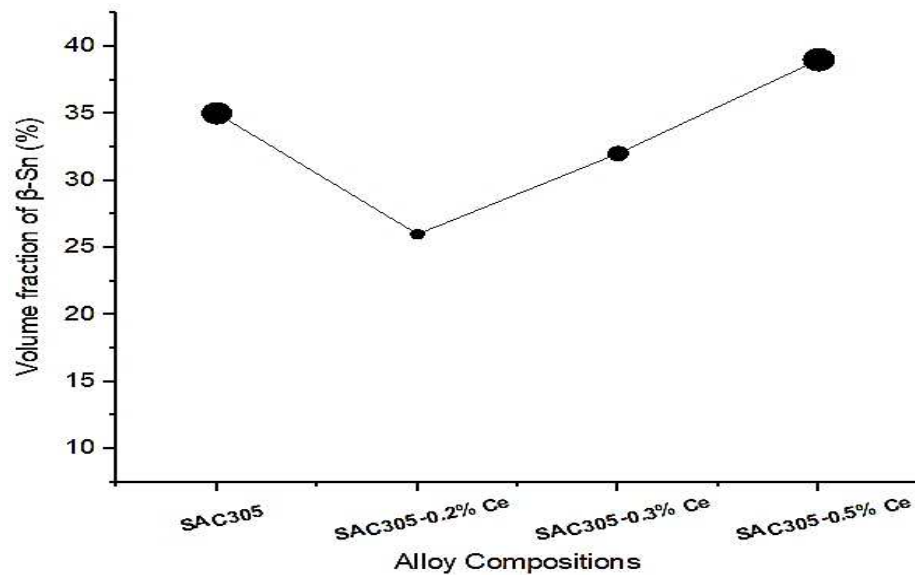


Figure 3. Variation of  $\beta$ -Sn volume fraction with alloy composition; marker size is proportional to the average  $\beta$ -Sn grain size.

Table 3. Quantitative  $\beta$ -Sn volume fraction and average  $\beta$ -Sn grain size of SAC305 and Ce-modified SAC305 solder alloys. Values represent mean  $\pm$  standard deviation obtained from quantitative image analysis of five SEM micrographs from different regions of each alloy.

| Composition  | $\beta$ -Sn Volume Fraction (%) | Avg Grain Size ( $\mu\text{m}$ ) |
|--------------|---------------------------------|----------------------------------|
| SAC305       | $35 \pm 3$                      | $20 \pm 2$                       |
| SAC305–0.2Ce | $26 \pm 2$                      | $10 \pm 2$                       |
| SAC305–0.3Ce | $32 \pm 2$                      | $14 \pm 2$                       |
| SAC305–0.5Ce | $39 \pm 3$                      | $21 \pm 3$                       |

Table 4. Local EDX compositional analysis of selected microstructural regions in SAC305 and Ce-modified SAC305 alloys

| Samples Alloy | Atomic concentration in % |      |      |      |
|---------------|---------------------------|------|------|------|
|               | Sn                        | C    | O    | Ce   |
| SAC305        | 94.60                     | 5.05 | 0.35 | 0.00 |
| 02%Ce- SAC305 | 92.60                     | 6.59 | 0.82 | 0.00 |
| 03%Ce- SAC305 | 92.40                     | 5.30 | 0.90 | 1.40 |
| 05%Ce- SAC305 | 92                        | 5    | 0.80 | 2.20 |

Note: Detected carbon is assumed to be from sample preparation and handling processes, not from the solder alloy and thus excluded from compositional interpretation.

To complement the local EDS point analyses, EDS spectrum acquisition and elemental mapping were performed on the SAC305–0.5 wt.% Ce alloy to evaluate the overall elemental distribution (Figure 4). The EDS spectrum confirms the presence of the principal constituent elements, with prominent Sn peaks accompanied by characteristic Ag, Cu, and Ce peaks, confirming the presence of Ce in the SAC305–0.5 wt.% Ce alloy. No additional elemental peaks attributable to impurities were detected within the detection limits of the EDS analysis. The corresponding elemental maps indicate that Sn, Ag, Cu, and Ce are detected throughout the analyzed region without obvious large-scale elemental agglomeration. Sn exhibits the highest signal intensity, consistent with its role as the matrix phase, whereas Ag and Cu are uniformly distributed throughout the microstructure in accordance with their lower concentrations. The Ce elemental map likewise shows a detectable signal across the analyzed region, suggesting that the added Ce is present within the investigated microstructure rather than existing as isolated macroscopic accumulations. However, owing to the limited spatial resolution and analytical capability of SEM–EDS, the elemental maps cannot distinguish whether Ce is dissolved within the  $\beta$ -Sn matrix, associated with intermetallic compounds, or present as nanoscale Ce-containing phases.

The quantitative EDS analysis summarized in Table 5 shows that the mapped region contains 95.7 wt.% Sn, 2.8 wt.% Ag, 0.45 wt.% Cu, and 0.4 wt.% Ce, which agrees well with the nominal composition of the SAC305–0.5 wt.% Ce alloy. The slight difference between the measured and nominal Ce contents (0.5 wt.%) is within the typical uncertainty of SEM–EDS quantitative analysis, particularly for minor alloying additions. A small carbon content (0.65 wt.%) was also

detected, which is attributed to the carbon mounting tape and/or minor surface contamination introduced during SEM sample preparation rather than the alloy itself.

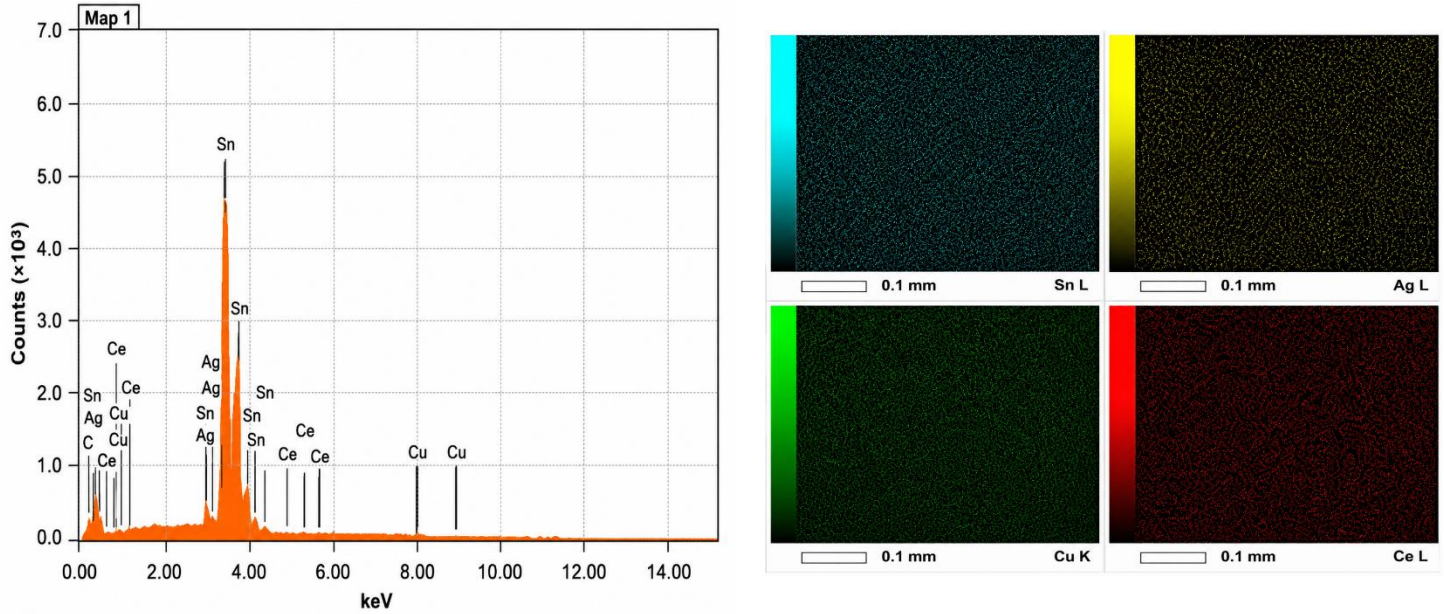


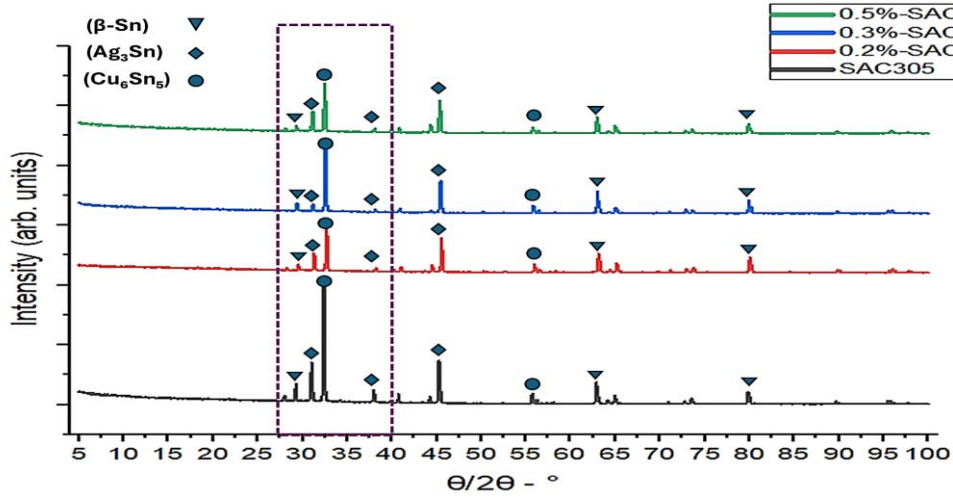
Figure 4. EDS spectrum and corresponding SEM–EDS elemental maps of Ag, Cu, Ce, and Sn for the SAC305–0.5 wt.% Ce alloy.

Table 5. Quantitative EDS elemental composition of the SAC305–0.5 wt.% Ce alloy obtained from the mapped surface

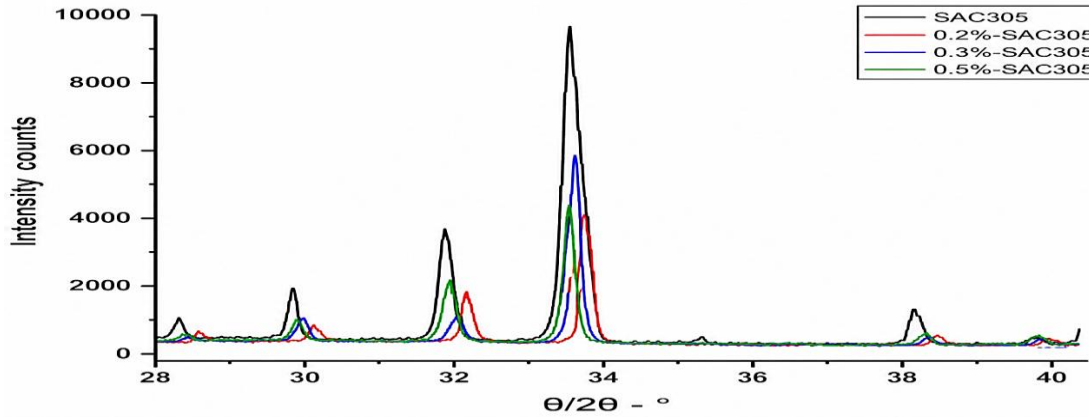
| Sample                 | Composition (wt%) |     |      |     |      |
|------------------------|-------------------|-----|------|-----|------|
|                        | Sn                | Ag  | Cu   | Ce  | C    |
| <b>0.5% Ce-SAC-305</b> | 95.7              | 2.8 | 0.45 | 0.4 | 0.65 |

The XRD patterns of the SAC305 and Ce-modified SAC305 solder alloys are presented in Figure 5(a), while the enlarged diffraction region between  $28^\circ$  and  $40^\circ$  ( $2\theta$ ) in Figure 5(b) provides a clearer view of the principal reflections. All alloys exhibit characteristic diffraction peaks corresponding to  $\beta$ -Sn,  $\text{Ag}_3\text{Sn}$ , and  $\text{Cu}_6\text{Sn}_5$ , indicating that Ce microalloying does not alter the primary phase constituents of the SAC305 alloy. No additional diffraction peaks associated with Ce-containing compounds were detected. This is likely due to the relatively low Ce additions together with the limited detection sensitivity of conventional XRD for phases present in very small volume fractions. The most significant change following Ce addition is observed in the relative intensity of the dominant  $\beta$ -Sn diffraction peak located at approximately  $33.7^\circ$  ( $2\theta$ ), as shown in the overall XRD patterns (Figure 5a) and more clearly in the enlarged diffraction region (Figure 5b). Compared with the unmodified SAC305 alloy, the SAC305–0.2 wt.% Ce alloy exhibits a noticeably lower  $\beta$ -Sn peak intensity. Although diffraction intensity may also be influenced by crystallographic preferred orientation and other experimental factors, the observed reduction is consistent with the previously discussed SEM observations and quantitative image analysis, which indicate reduced  $\beta$ -Sn grain size and volume fraction in the SAC305–0.2 wt.% Ce alloy. Collectively, these observations are consistent with the development of a finer and more

homogeneous microstructure in the SAC305–0.2 wt.% Ce alloy. As the Ce content increased to 0.3 wt.% and 0.5 wt.%, the  $\beta$ -Sn peak intensity increased relative to the SAC305–0.2 wt.% Ce alloy, in agreement with the reappearance of larger  $\beta$ -Sn regions and the corresponding increase in  $\beta$ -Sn volume fraction and grain size. This reduced refinement is consistent with the local EDS analyses, which detected higher Ce concentrations in selected regions of the higher-Ce alloys, suggesting localized Ce enrichment at higher Ce additions, which may contribute to the observed reduction in refinement efficiency. Furthermore, no measurable shifts in the  $\beta$ -Sn diffraction peak positions were observed with increasing Ce addition (Figure 5b), suggesting that any lattice distortion or solid solubility of Ce in  $\beta$ -Sn is below the detection capability of the present XRD measurements. Accordingly, the XRD results suggest that the principal effect of Ce under the present experimental conditions is associated with microstructural evolution during solidification rather than detectable changes in the crystalline phases present in the alloy.



(a)



(b)

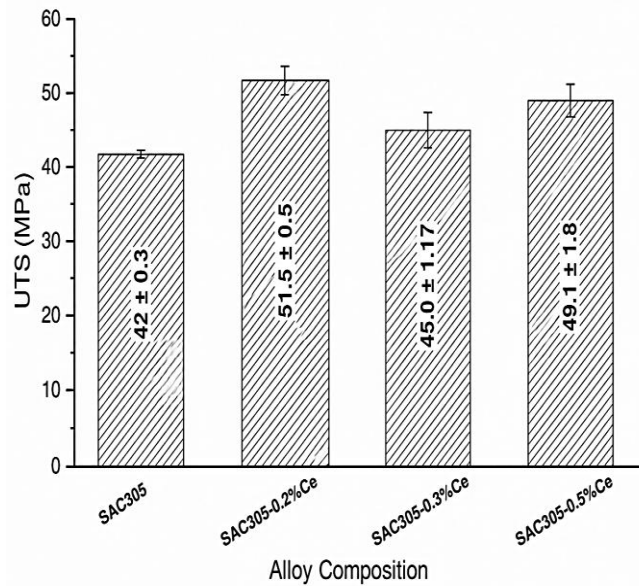
Figure 5. (a) X-ray diffraction patterns corresponding to cerium addition (0.2% to 0.5%) in SAC305 solder alloy (b) Enlarged view of the XRD patterns in the  $2\theta$  range of  $28^\circ$ – $40^\circ$ , showing the variation in  $\beta$ -Sn diffraction peak intensities with increasing Ce content.

### 3.2 Mechanical properties analysis

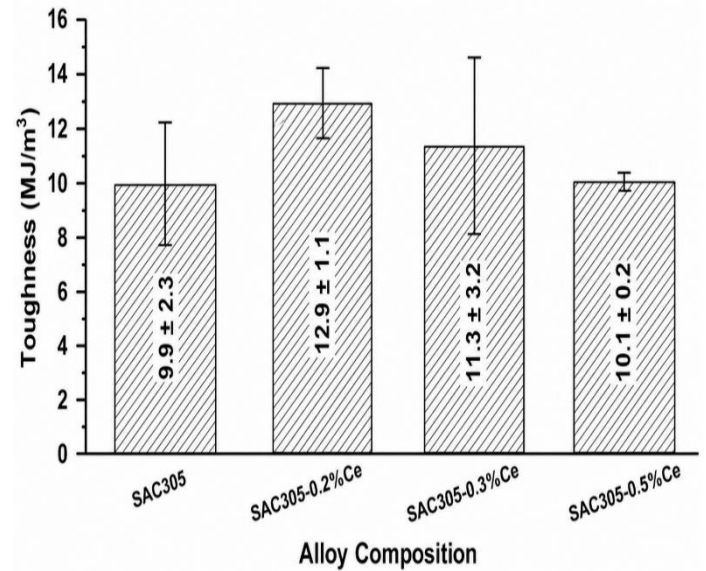
The effect of Ce addition on the mechanical properties of SAC305 solder alloys is summarized in Figure 6, which presents the variations in ultimate tensile strength (UTS), toughness, ductility, and Vickers hardness. The mechanical response closely follows the microstructural evolution observed by SEM and is consistent with the XRD results, suggesting that the variations in mechanical properties are primarily associated with changes in microstructure rather than the formation of new crystalline phases. The SAC305–0.2 wt.% Ce alloy exhibited the best overall mechanical performance, with the UTS increasing to  $51.5 \pm 0.5$  MPa, representing an improvement of approximately 22.6% compared with the base SAC305 alloy. The toughness increased from  $9.9 \pm 0.8$  to  $12.9 \pm 1.1$  MJ m<sup>-3</sup>, while the ductility remained above 52%, indicating that the increase in strength was achieved without a significant loss of plastic deformation capability. The hardness also increased from  $13.8 \pm 0.5$  to  $18.5 \pm 0.7$  HV, corresponding to an improvement of approximately 34%. The improved mechanical performance of the SAC305–0.2 wt.% Ce alloy is consistent with the refined microstructure observed by SEM, the quantitative image analysis, and the XRD results. These characterization techniques collectively showed reductions in both the volume fraction and grain size of coarse primary  $\beta$ -Sn regions together with a lower intensity of the dominant  $\beta$ -Sn diffraction peak. Such microstructural refinement is expected to increase the density of phase boundaries that impede dislocation motion while promoting a more uniform distribution of strain during tensile deformation. Consequently, localized deformation is reduced, contributing to the observed improvements in tensile strength, toughness, and hardness while maintaining good ductility. As the Ce content increased beyond 0.2 wt.%, the mechanical properties gradually deteriorated. The UTS decreased to  $45.0 \pm 1.17$  MPa for the SAC305–0.3 wt.% Ce alloy before increasing slightly to  $49.1 \pm 1.8$  MPa for the SAC305–0.5 wt.% Ce alloy, whereas the ductility decreased continuously to  $40.3 \pm 0.7\%$ . The hardness followed a similar trend, decreasing to  $15.8 \pm 0.6$  HV at 0.3 wt.% Ce before increasing slightly to  $17.2 \pm 0.6$  HV at 0.5 wt.% Ce. The reduction in mechanical performance at higher Ce contents is consistent with the coarsening of the  $\beta$ -Sn microstructure observed by SEM, the increased  $\beta$ -Sn diffraction intensity observed by XRD, and the local EDS analyses, which detected higher Ce concentrations in selected regions of the SAC305–0.3 wt.% Ce and SAC305–0.5 wt.% Ce alloys. Collectively, these observations suggest that increasing Ce additions reduce the degree of microstructural refinement achieved at 0.2 wt.% Ce, resulting in larger  $\beta$ -Sn regions and increased microstructural heterogeneity. These microstructural changes are expected to promote localized plastic deformation and stress concentration, thereby contributing to the observed reductions in tensile strength and ductility.

The slight recovery in tensile strength and hardness observed for the SAC305–0.5 wt.% Ce alloy may be associated with the presence of Ce-enriched regions detected by the local EDS analyses. However, these regions do not appear to compensate for the accompanying coarsening of the  $\beta$ -Sn

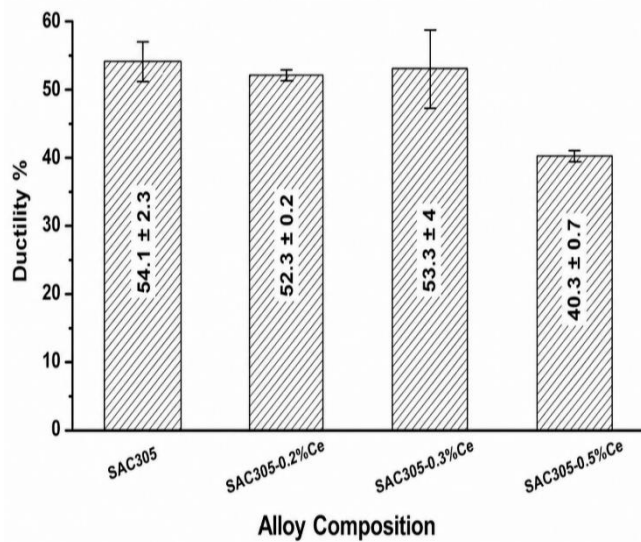
microstructure, as reflected by the lower ductility and the overall mechanical performance remaining inferior to that of the SAC305–0.2 wt.% Ce alloy. Among the investigated compositions, the SAC305–0.2 wt.% Ce alloy exhibited the most favorable combination of tensile strength, toughness, ductility, hardness, and microstructural refinement. These results indicate that 0.2 wt.% Ce provides the optimum balance of mechanical performance for the present alloy system. Although the refined microstructure is expected to be beneficial for solder joint performance, the implications for long-term reliability under service conditions such as thermal cycling and mechanical fatigue require dedicated reliability testing and therefore remain beyond the scope of the present study.



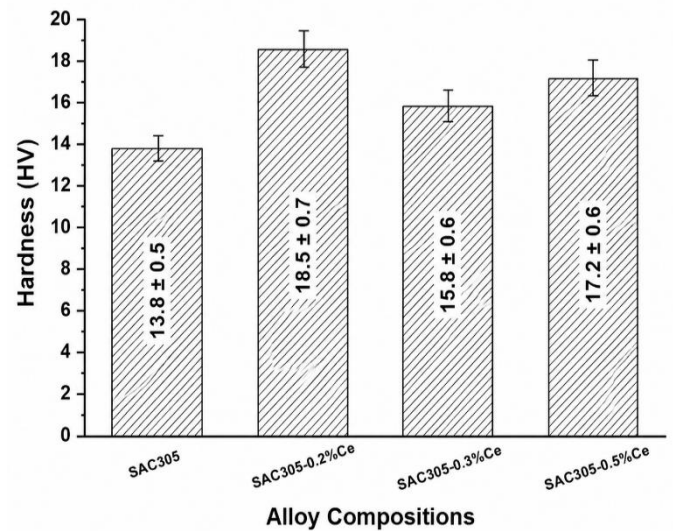
(a)



(b)



(c)



(d)

Figure 6. Effect of Ce addition on the mechanical properties of SAC305 solder alloys: (a) ultimate tensile strength (UTS), (b) toughness, (c) ductility, and (d) Vickers hardness. Error bars represent the standard deviation obtained from repeated measurements.

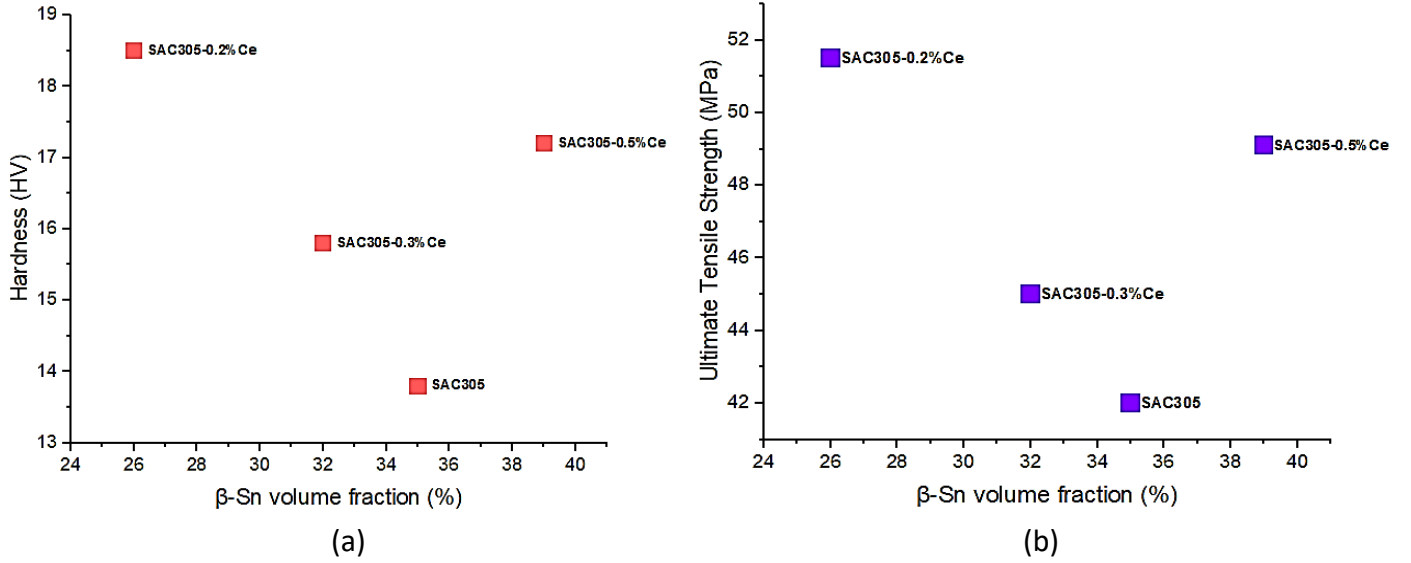


Figure 7. Relationship between  $\beta$ -Sn volume fraction and the mechanical properties of SAC305 and Ce-modified SAC305 solder alloys: (a) Vickers hardness and (b) ultimate tensile strength (UTS).

Figure 7(a,b) further illustrates the relationship between the measured volume fraction of coarse primary  $\beta$ -Sn regions and the corresponding mechanical properties of the SAC305 and Ce-modified SAC305 alloys. The reduction in the  $\beta$ -Sn volume fraction from 35% in the base SAC305 alloy to 26% following the addition of 0.2 wt.% Ce was accompanied by an increase in hardness from 13.8 to 18.5 HV and an increase in ultimate tensile strength from 42.0 to 51.5 MPa. As the Ce content increased to 0.3 wt.% and 0.5 wt.%, the measured  $\beta$ -Sn volume fraction increased again, accompanied by corresponding decreases in both hardness and tensile strength. This trend is consistent with the SEM, EDX, and XRD results, indicating that suppression of coarse primary  $\beta$ -Sn and the resulting microstructural refinement contribute significantly to the observed improvement in mechanical properties. Therefore, Figure 6 provides additional quantitative support for the structure–property relationship established in this study.

### 3.3 Thermal property analysis:

Figure 8 presents the DSC heating curves of the SAC305 and Ce-microalloyed SAC305 solder alloys, while the corresponding thermal parameters are summarized in Table 6. All alloys exhibited a single endothermic melting peak, indicating that Ce microalloying did not alter the fundamental melting behavior of SAC305. The peak melting temperature ( $T_p$ ) varied only between 222.07 and 223.40 °C, corresponding to a maximum difference of 1.33 °C among all compositions. Similarly, the onset and endset melting temperatures remained nearly unchanged, indicating that Ce addition has a negligible influence on the melting characteristics of SAC305. These results suggest that Ce microalloying is unlikely to require modifications to conventional reflow soldering profiles.

Although the characteristic melting temperatures remained essentially unchanged, Ce addition influenced the shape and intensity of the DSC endothermic peak. As shown in Figure 8, the unmodified SAC305 alloy exhibited the highest peak heat-flow intensity ( $71 \text{ mW mg}^{-1}$ ), whereas the Ce-containing alloys showed lower peak intensities ranging from 35 to 39  $\text{mW mg}^{-1}$ . In addition, the melting interval decreased slightly from  $15.8^\circ\text{C}$  for SAC305 to approximately  $14.2\text{--}14.3^\circ\text{C}$  for the Ce-containing alloys. Because DSC peak intensity may also be affected by factors such as sample mass, thermal contact, and baseline correction, these changes should be interpreted cautiously. Nevertheless, the observed trend is consistent with the refined and more homogeneous microstructure identified by the SEM analysis, which may promote a more uniform melting response.

The estimated enthalpy of fusion ( $\Delta H$ ) remained nearly constant, ranging from  $58.9$  to  $60.8 \text{ J g}^{-1}$ , indicating that Ce addition does not significantly affect the total energy required for melting. Overall, the DSC results demonstrate that Ce microalloying produces only minor changes in the thermal response while preserving the characteristic melting behavior of SAC305. Combined with the SEM, EDS, and XRD results, these findings suggest that the principal effect of Ce is associated with refinement of the solidification microstructure rather than modification of the intrinsic thermal characteristics of the alloy. Consequently, the improved microstructural and mechanical properties are achieved without compromising compatibility with conventional reflow soldering processes.

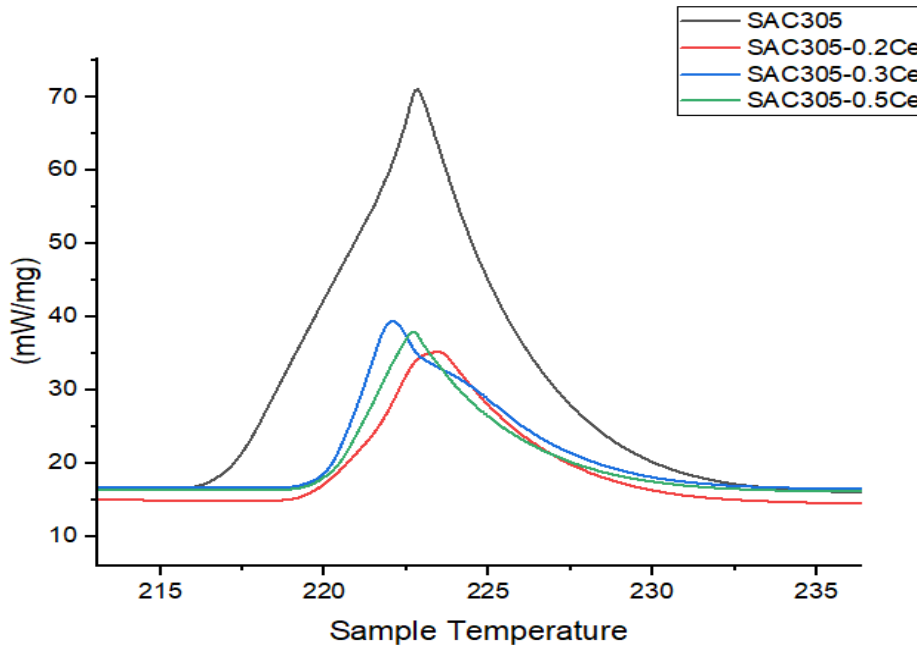


Figure 8- Differential scanning calorimetry (DSC) heating curves of the as-cast SAC305 and Ce-microalloyed SAC305 solder alloys containing 0.2, 0.3, 0.4, and 0.5 wt.% Ce

Table 6. DSC thermal parameters of the as-cast SAC305 and Ce-microalloyed SAC305 solder alloys

| Sample       | Tonset (°C) | Peak Temperature, T <sub>p</sub> (°C) | Tendset (°C) | Melting Range (°C) | (mW/mg) | ΔH (J/g) |
|--------------|-------------|---------------------------------------|--------------|--------------------|---------|----------|
| SAC305       | 217.6       | 222.83                                | 233.4        | 15.8               | 71      | 60.8     |
| SAC305-0.2Ce | 219.0       | 223.4                                 | 233.2        | 14.2               | 35      | 60.1     |
| SAC305-0.3Ce | 219.2       | 222.07                                | 233.4        | 14.2               | 39      | 59.5     |
| SAC305-0.5Ce | 219.0       | 222.8                                 | 233.3        | 14.3               | 38      | 58.9     |

#### 4. Conclusions:

- Cerium microalloying significantly influenced the microstructural evolution of SAC305 solder. Among the investigated compositions, the addition of 0.2 wt.% Ce produced the greatest microstructural refinement, resulting in a finer and more homogeneous eutectic structure with a reduced fraction of coarse primary  $\beta$ -Sn regions.
- Quantitative image analysis confirmed that the SAC305–0.2 wt.% Ce alloy exhibited the lowest  $\beta$ -Sn volume fraction ( $26 \pm 2\%$ ) and the smallest average  $\beta$ -Sn grain size ( $10 \pm 2 \mu\text{m}$ ), whereas higher Ce additions reduced the grain-refining efficiency.
- XRD analysis identified  $\beta$ -Sn,  $\text{Ag}_3\text{Sn}$ , and  $\text{Cu}_6\text{Sn}_5$  as the principal phases in all investigated alloys, with no detectable Ce-containing phases under the present experimental conditions. The diffraction results indicate that Ce primarily influences the solidification behavior through microstructural refinement rather than by forming detectable new crystalline phases.
- The refined microstructure achieved with 0.2 wt.% Ce resulted in the best overall mechanical performance, yielding the highest ultimate tensile strength ( $51.5 \pm 0.5 \text{ MPa}$ ), hardness ( $18.5 \pm 0.7 \text{ HV}$ ), and toughness ( $12.9 \pm 1.1 \text{ MJ m}^{-3}$ ) while maintaining good ductility.
- Differential scanning calorimetry (DSC) demonstrated that Ce microalloying produced only negligible changes in the melting characteristics of SAC305, indicating that the enhanced microstructural and mechanical properties were achieved without compromising compatibility with conventional reflow soldering processes.
- Overall, 0.2 wt.% Ce was identified as the optimum microalloying concentration for SAC305 under the present experimental conditions, providing the best combination of microstructural refinement, mechanical strengthening, and thermal stability.

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#### Conflict of interest declaration:f

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

### Authors' Contributions:

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