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Study of the Impact of Cerium Addition on the Microstructure and Properties of Tin- Silver (Sn-3.5Ag) Solder Alloy

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***Abstract*—** Solders have a vast potential market and are involved in almost every manufacturing and engineering process, such as electronic circuit boards, automobile repair processes, and pipeline soldering. Lead-free solder alloy in the form of Tin-Silver Sn-3.5Ag is considered an excellent alternative to conventional Tin-Lead solder because of its good mechanical properties and less harmful environmental effects. However, some problems, like the formation of large intermetallic compounds associated with Sn-3.5Ag, need high attention. Hence, the growth of intermetallic compounds in the tin matrix is enhanced further at high temperatures; therefore, its effect on the mechanical properties becomes more substantial. Scanning electron microscopy was used to examine the microstructure of intermetallic compound particles. The elemental composition was confirmed using an energy-dispersive X-ray. The results were analyzed to study the effects of adding cerium in different compositions to Sn-3.5Ag, including its effect on making the microstructure more refined and coarser regarding IMCs existence and subsequent effects on mechanical properties. To overcome this problem, this study examines rare earth elements like cerium doping (0.1, 0.3, and 0.6) wt.% into Sn-3.5Ag to study the microstructure and subsequent mechanical properties. The study includes the examination of the microstructure and mechanical properties of novel alloys, namely Sn-3.5Ag, Sn-3.5Ag-0.1Ce, Sn-3.5Ag-0.3Ce, and Sn-3.5Ag-0.6Ce, to ensure the requirement for a green environment and make electronic materials, products, and processes as environmentally benign as possible.

***Keywords*—** Tin-Silver; cerium, microstructure, green environment, Intermettalic compounds, lead free solders.

I. INTRODUCTION

The demand for electronic devices is increasing in our daily lives. However, electronic devices are composed of various materials, many of which are hazardous and harmful to the environment. One such material is lead (Pb), which is extensively used in solders consumed in electronics. Recycling and disposal of such materials may cause significant risks to the environment. Hence, with the advancement in Technologies coupled with increased imports of refurbished computers and other electronic equipment, hazardous electronic waste has now been considered one of the most significant environmental risks [1].

Moving towards green electronics has been given the highest priority in recent years. The efforts to develop alternatives to Pb-based solders have increased significantly, where the main objective is to reduce the toxic environmental impact caused by Pb-based solders. Parliaments of different countries have also passed legislation to limit the use of Pb and other harmful materials while manufacturing electronic devices. Hence, green electronics have focused on eliminating harmful and toxic chemicals that have devastating effects on the environment. However, the most debatable and great challenge was banning Pb in solders, which are extensively used in printed circuit boards (PCBs). It is worth mentioning that lead solders have been used in almost every field of the electronics industry to join the components to PCBs [2].

Soldering is a process in which two metals are joined together by introducing a third metal or alloy with a relatively low melting point compared to the parent metal. The solder alloys are binary, ternary, and some are even quaternary alloys. During soldering, the solder is placed in the desired location and then heated with the help of a hot iron, which is the primary heat source. The heat from iron causes the solder to melt, which, after melting, is allowed to cool, thus creating a soldering joint [3].

Solder plays a vital role in making electronic connections. Unfortunately, conventional solders contain lead, which is harmful to humans and pollutes the environment [4]. The conventional solders used in the electronic industry contain 60% Tin and 40% lead. Hence, it is highly recommended to go towards lead-free solders and green electronics in such a way that they have minimum impact on the environment [5].

Based on all these adverse effects of Pb on the environment, much legislation has been passed to avoid Pb contents in solder alloys. Therefore, there was a great need to introduce Pb-free solder alloys to replace conventional ones.

Therefore, manufacturers and researchers must go towards lead-free solder alloys to make the environment safe. The development and investigation of various green solder alloys are in progress to determine the mechanical, metallurgical, and physical properties and investigate their toxicity on living things and environmental impacts [2]. Various research has developed lead-free solders in which Tin-Silver with Copper (Sn-Ag-Cu) and Tin-Silver (Sn-Ag) alloys are the most extensively accepted alloys to replace Sn-Pb solders. These lead-free solders are widely used in the microelectronic packaging industry due to their excellent properties. However, selecting the best alternative Pb-free solder alloys with all the desirable mechanical and physical properties is still challenging. This is because Pb-free solder should satisfy engineering and other properties, including environmentally friendly, easily accessible, reliable, good mechanical properties, etc [5].

Considering the above criteria, the development of different Pb-free solder alloys is in progress to replace the 63Sn-37Pb solder, yet it is not an easy to get a suitable candidate. Therefore, various research studies were conducted to get firsthand knowledge and an in-depth idea on the development of different Pb-free solders to move towards green applications and different companies organized several projects. Hence, it was concluded that one of the most suitable and widespread is the Tin-Silver Copper (Sn-Ag-Cu) and Tin and Silver (Tin-Silver). Research also shows that these alloy exhibits good thermal fatigue properties and joint strength is considerably more than lead bearing solders; however, the existence of coarse microstructure along with hard and brittle inter-metallic compounds (IMCs) has brought the restriction to the use of these in high-temperature applications [6]. Therefore, lots of additives were considered to refine the microstructure and enhance the mechanical properties of Pb-free solders. In this regard, researchers have tried to improve solder's material properties by introducing rare elements (RE) known as the vitamins for the metals and be effective [7]. The three significant benefits of RE on lead-free solder materials are enhancing mechanical properties, improving wettability, and refining microstructure by reducing the IMCs growth.

Previous studies on the inclusions of RE elements in Sn-Ag include lanthanum (0.1, 0.5, and 1.0) wt.% [8], lanthanum (0.05, 0.1, and 0.25) wt.% [9, 10], lanthanum 0.5 wt.% [11], holmium (0.1, 0.2, 0.3, 0.4 and 0.5) wt.% [12], whereas for Sn-Ag-Cu the doping of RE elements includes cerium (0.006, 0.019, 0.171, 0.011, 0.024, 0.152, 0.005, 0.021, and 0.162) wt.% [13], cerium (0.02, 0.15, and 0.5) wt.% [14], lanthanum 0.4 wt.% [6, 7, 15], cerium (0.15, 0.35, 0.55, 0.75, and 0.95) wt.% [16], lanthanum (0.05, 0.25, and 0.5) wt.% [17], lanthanum (0.1, 0.3, and 0.5) wt.% [18], lanthanum (0.3 and 0.6) wt.% [19], Erbium (0.025, 0.05, 0.1, 0.25, 0.5, and 1.0) wt.% [20], Neodymium (0.025 0.05 0.1 0.25 0.5) [21], praseodymium (0.025 0.05 0.1 0.25 0.5) [22] and Ytterbium (0.025 0.04 0.05 0.06 0.075 0.1 0.2 0.5) [23]. This indicates most of the studies in the development and characterization of Pb-free solders with the inclusions of RE elements has been done on Sn-Ag-Cu compared to Sn-Ag. Additionally, there is a need to investigate different compositions apart from those considered in the literature. Furthermore, literature has also acknowledged that among all the RE elements, Cerium (Ce) is considered the best because of its lower cost, widespread availability and low melting point [14]. Therefore, in this work, doping cerium with different compositions has been used with Sn-Ag as the base alloy. It is worth mentioning that a good doping of certain RE elements is necessary for improving mechanical properties, refining microstructure, and reducing intermetallic growth. This research work aims to assist the industry in selecting the best alloy from the many available alternatives and examine the contribution of lead-free soldering to green electronics. The main objective of this research is to develop a new Pb-free solder alloy with the inclusion of different compositions of cerium in different weight ratios of 0.1%, 0.3%, and 0.6% to ensure the requirement for a green environment and making electronic materials, products, and processes as environmentally benign as possible. The study is also based on examining the microstructure of these novel alloys in terms of IMCs to ensure the reliability of solder joints. Further investigations include the evaluation of mechanical properties by performing tensile tests.

II. MATERIALS AND METHODS

In this study, the alloys prepared were Sn-3.5Ag, Sn-3.5Ag-0.1Ce, Sn-3.5Ag-0.3Ce, and Sn-3.5Ag-0.6Ce. These alloys include Sn-3.5Ag as a base material, and then inclusions of 0.1, 0.3, and 0.6 (wt.%) were made to the base alloy. All these alloys were made from 99% purity from Sn, Ag, and Ce powders. Table I shows the alloy composition in this study.

Table I. The alloy composition of this study

Solder alloy	Elements (wt.%)		
	Sn (%)	Ag (%)	Ce (%)
Sn-3.5Ag	96.5	3.5	0
Sn-3.5Ag-0.1Ce	96.4	3.5	0.1
Sn-3.5Ag-0.3Ce	96.2	3.5	0.3
Sn-3.5Ag-0.6Ce	95.9	3.5	0.6

Good alloy composition is essential in getting the required results like elemental composition, intermetallic compound size, particle distribution, grain boundaries and grain size, and most importantly, mechanical properties. Hence, a special Die was designed and used in this study. The upper and lower sections of the Die were made from aluminium, and the center plate was made from steel, as shown in Figure 3.2. It is worth mentioning that the Die can make five tensile specimens and provide a high colling rate. First, the required quantity of the target alloys in powder form was put in the crucible, and then the crucible was placed inside the electric furnace. A temperature of 1000 °C was set for 45 minutes to make the molten metal, which was then poured into the Die and left for 5 hours of air cooling. Figure 3 shows the samples produced for further investigations. Half of these samples were exposed to thermal aging at 200°C for 60 hours, while the remaining samples were left as-casted. It is worth noting that thermal aging analyses the effects on material due to prolonged exposure to high temperatures. Such conditions are generally responsible for the reduction in strength due to changes in microstructure after prolonged contact with the material at high temperatures. Thermal aging was done at a temperature of 200 °C for 60 hours using the oven (Thomas Scientific Model 605 with a maximum drying temperature of 300 °C)

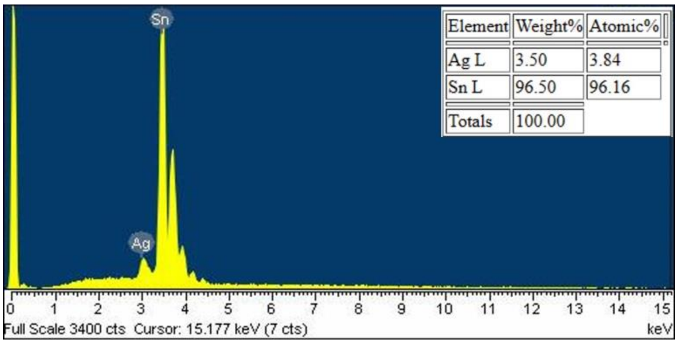
The tensile tests were performed on the universal testing machine for the as-casted and thermally aged samples, and the ultimate tensile strength was measured. A comparison was first made between the undoped and Ce-doped Sn-3.5Ag alloys for both the as-casted and thermally aged samples.

For the microstructural analysis, the as-casted and thermally aged samples were cut and mounted in Bakelite. Afterwards, grinding of the samples was done with silicone carbide with a grit size of 350, 600, 800, and 1200. Tap water was used as a lubricant to remove heat generation during this process. The samples were then polished with polycrystalline diamond suspension as the abrasive particle size of 6 µm, 1 µm and 0.25 µm on cotton silk paper. After polishing, the samples were cleaned with distilled water and etched for 12 seconds with 5% hydrochloric acid and 95% ethanol. Each sample was then coated with gold using the coater machine, and finally, SEM images were taken using scanning electron microscopy. EDX analysis was done to identify the elemental composition of samples, which is coupled with scanning electron microscopy. Furthermore, the SEM images are digitally processed using ImageJ software to analyze the intermetallic compound particle size microstructure.

III. RESULTS AND DISCUSSION

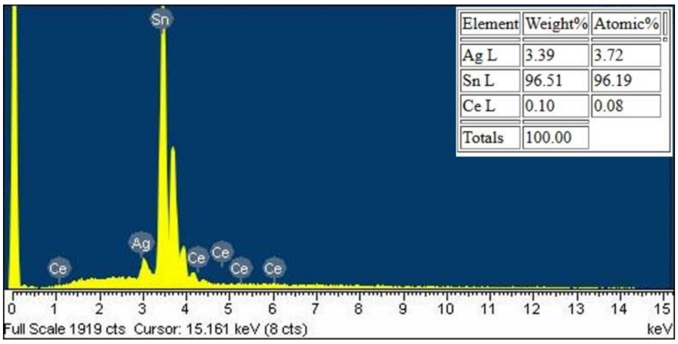
A. Energy-dispersive X-ray spectroscopy

In this study, the elemental composition of the undoped Ce-doped Sn-3.5Ag was done using energy-dispersive X-ray spectroscopy (EDS). This is one of the most used techniques for the elemental analysis or chemical characterization of the samples to confirm the composition of the prepared alloys. Figure 1 shows the EDS analysis of Sn-3.5Ag alloy, confirming Sn and Ag presence in wt.%. The confirmation of cerium addition into Sn-3.5Ag with 0.1, 0.3 and 0.6 wt. % are given in Figure 1 – 4.



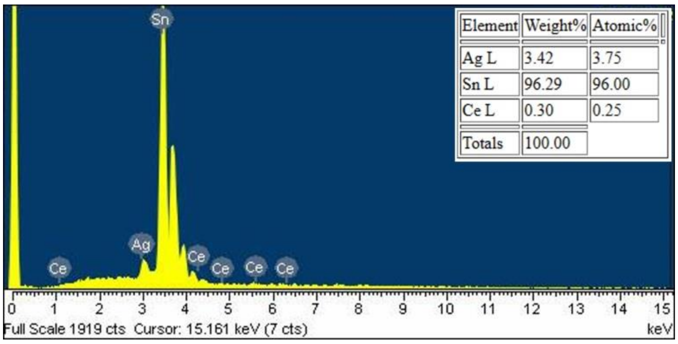
Element	Weight%	Atomic%
Ag L	3.50	3.84
Sn L	96.50	96.16
Totals	100.00	

Figure 1. EDX of Sn-3.5Ag



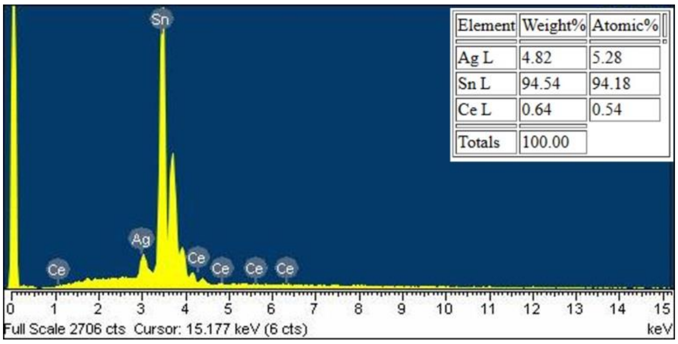
Element	Weight%	Atomic%
Ag L	3.39	3.72
Sn L	96.51	96.19
Ce L	0.10	0.08
Totals	100.00	

Figure 2. EDX of Sn-3.5Ag-0.1Ce



Element	Weight%	Atomic%
Ag L	3.42	3.75
Sn L	96.29	96.00
Ce L	0.30	0.25
Totals	100.00	

Figure 3. EDX of Sn-3.5Ag-0.3Ce



Element	Weight%	Atomic%
Ag L	4.82	5.28
Sn L	94.54	94.18
Ce L	0.64	0.54
Totals	100.00	

Figure 4. EDX of Sn-3.5Ag-0.6Ce

B. Impact of cerium addition and thermal aging on Sn-3.5Ag

- **Mechanical properties:** In this study, the as-casted and thermally aged samples of undoped and Ce-doped Sn-3.5Ag alloys are tested under tensile testing to investigate the ultimate tensile strength of each alloy, as shown in Figure 5.

First, a comparison was made between the undoped and Ce-doped Sn-3.5Ag, and then the same composition alloys were compared for the thermally aging process. Hence, Figure 1 illustrates the ultimate tensile strength of Sn-3.5Ag after adding Ce addition and the thermally aged samples at 200°C for 60 hours. The results showed increased UTS after adding Ce into Sn-3.5Ag alloy. The UTS of the Sn-3.5Ag increased with the addition of 0.1 wt.% and 0.3 wt.% Ce addition and decreased with 0.6 wt.% Ce inclusions, regardless of the thermal aging.

Furthermore, the UTS decreased when the samples were tested after thermal aging, irrespective of the Ce-addition into Sn-3.5Ag. This shows that thermal aging has a significant impact on the reliability of the alloys. However, despite the thermal aging effect, the highest UTS was obtained at the inclusion of 0.3wt% Ce into Sn-3.5Ag. Hence, for a reliable solder joint, the optimal concentration of Ce into Sn-3.5Ag was 0.3 wt.%, as selected in this study. A likely explanation for this, as reported in the literature, is that an appropriate composition should be selected as inclusion into lead-free solder alloys because there are chances that higher concentrations might affect the microstructure by increasing the size of the intermetallic compounds. It is also worth mentioning that these IMCs play an essential role in the coarsening or refining of the microstructure, which ultimately affects the strength of the alloys. To confirm this, the samples after Ce addition and thermally aged were examined for microstructure analysis, as discussed in the preceding sections.

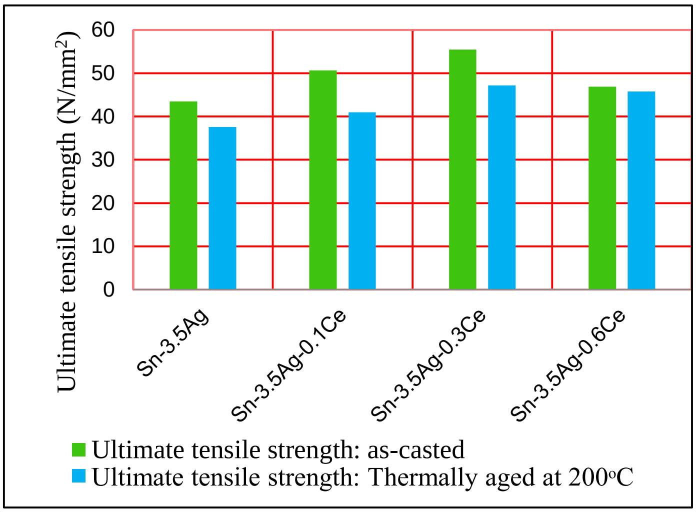


Figure 5. Ultimate tensile strength

- **Microstructure examination:** This study used scanning electron microscopy to examine the undoped and Ce-doped Sn-3.5Ag microstructure. The microstructure of Sn-Ag is composed of large needle-or rod-like Ag3Sn IMCs that are randomly distributed in the Sn-matrix [24]. The dark zone represents the Sn-matrix whereas the Ag3Sn IMCs are represents the bright area [12]. The addition of minor amounts of the Ce as a rare earth element into the Sn-3.5'Ag led to the refinement of the microstructure. As a surface-active element, RE elements have been widely acknowledged to play a vital role in the refinement of the microstructure, enhancing the mechanical properties and alloying and purification of materials [24]. This is attributed by the additional RE particles in the Pb-free solders, for example, CeSn3 in the case of Ce inclusions to Pb-free solders [13]. Hence, if the optimal level of RE addition is exceeded, these particles grow in number and size, which resulted in coarsening the microstructure and decreasing the mechanical properties [13].

Figures 6 show the SEM micrographs of the Sn-3.5Ag, Sn-3.5Ag-0.1Ce, Sn-3.5Ag-0.3Ce and Sn-3.5Ag-0.6Ce. The results showed a finer microstructure of the Sn-3.5Ag after including cerium. The IMCs of Sn-3.5Ag-0.1Ce seem more controlled than Sn-3.5Ag. This controlled formation of IMCs further enhanced the microstructure of Sn-3.5Ag when the Ce addition was increased to 0.3 wt.%. Literature has shown that rare earth elements, including cerium, act as the surface-active element that increases the activation energy for the diffusion of the grain boundaries, thus restricting the grain boundaries from moving. Furthermore, stronger bonds occurred at the grain boundaries by providing a pinning effect from the Ce atoms. In addition, due to the large atomic radius, RE elements tend to be insoluble in many metals, including Tin and Silver, and accumulate at the phase boundaries. This accumulation provides a barrier to the further growth of IMCs. Hence, microstructure refinement is expected to result in improved mechanical properties. However, a further increase of Ce addition with 0.6 wt.% in Sn-3.5Ag coarsens the microstructure, showing large IMCs. Hence, the decrease in the UTS. Thus, it is concluded in this study that the doping concentration of Ce in Sn-3.5Ag should be limited to 0.3 wt. %.

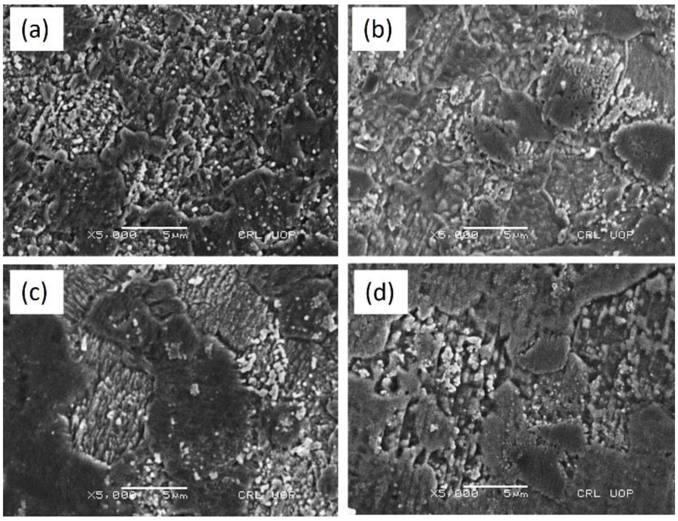


Figure 6. As casted: (a) Sn-3.5Ag (b) Sn-3.5Ag-0.1Ce (c) Sn-3.5Ag-0.3Ce and (d) Sn-3.5Ag-0.6Ce

Further investigation of IMCs with the thermal aging of all the samples selected in this study also showed a coarse microstructure, as given in Figure 7. For the Sn-3.5Ag, the formation of brittle Ag3Sn IMCs was the main reason for the increase in IMCs size after thermal aging at 200 °C for 60 hours. This also led to weaken the mechanical properties [25]. However, the growth of IMCs after the inclusion of Ce into the Sn-3.5Ag was found to be minimum even after thermal aging. The least impact of thermal aging was found on the Sn-3.5Ag alloy with 0.3 wt. % Ce concentration. In the case of Sn-3.5Ag-0.6Ce, it is likely the Large CeSn3 particles cause a significant decrease in UTS [26]. The digitally processed SEM images from ImageJ are shown in Figure 7 whereas the IMCs size for the as-casted and thermally aged alloys selected in this study is given in Figure 8.1 and 8-2. Moreove, comparison of Intermellatic compounds paritical size of “as casted” and “thermally aged” are shown in Figure 9.

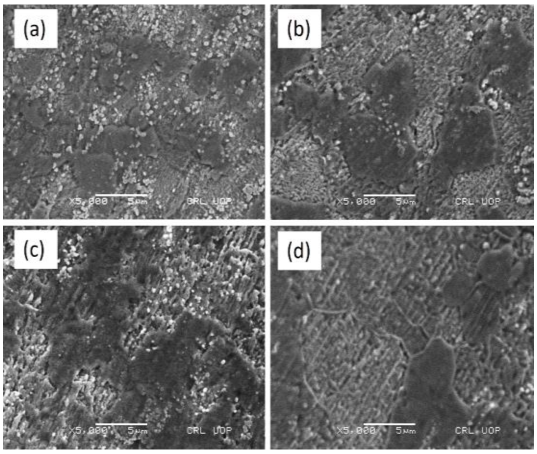
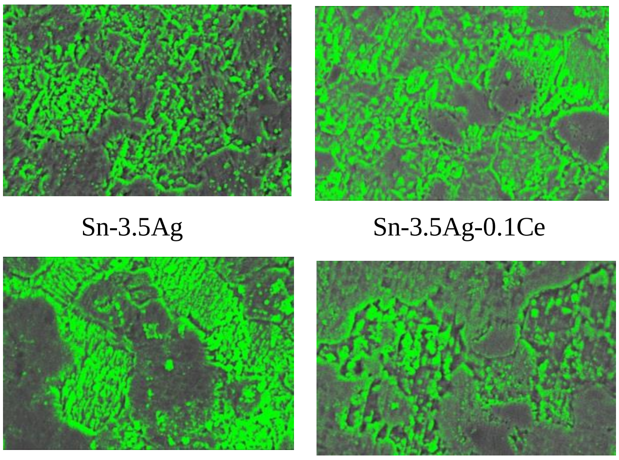


Figure 7. Thermally aged: (a) Sn-3.5Ag (b) Sn-3.5Ag-0.1Ce (c) Sn-3.5Ag-0.3Ce and (d) Sn-3.5Ag-0.6Ce

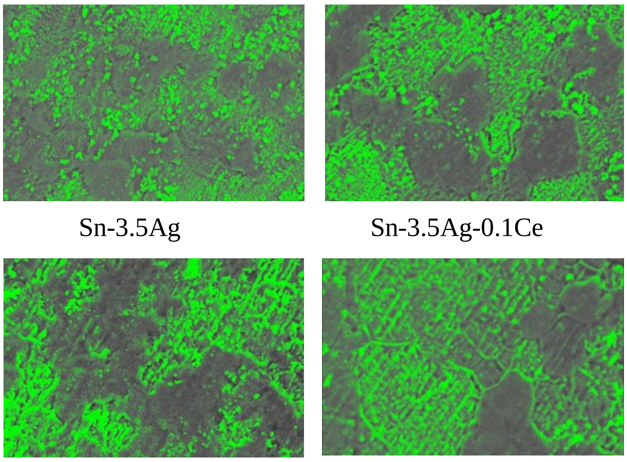


Sn-3.5Ag Sn-3.5Ag-0.1Ce

Sn-3.5Ag-0.3 Ce Sn-3.5Ag-0.6Ce

Figure 8.1 Digitally processed images from ImageJ

(As casted)



Sn-3.5Ag Sn-3.5Ag-0.1Ce

Sn-3.5Ag-0.3 Ce Sn-3.5Ag-0.6Ce

Figure 8.2 Digitally processed images from ImageJ

(Thermally aged)

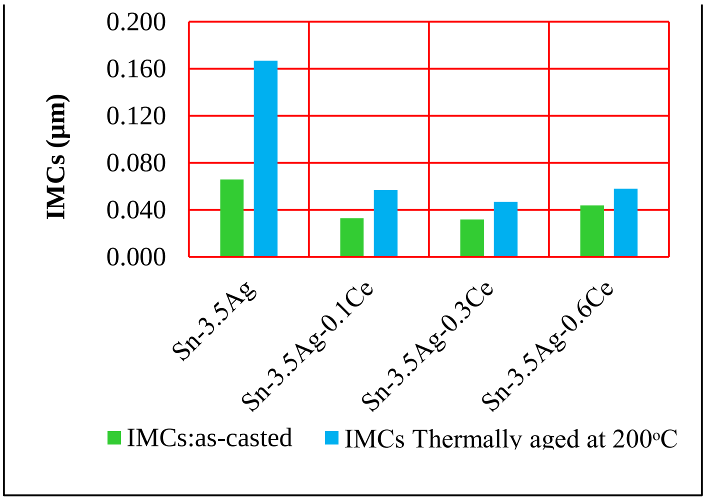


Figure 9. Intermetallic Compound particle size

CONCUSLION

In this study, different compositions of cerium, including 0.1, 0.3 and 0.6 wt.% were added to the Sn-3.5Ag binary solder alloy. All the samples were examined under scanning electron microscopy for the microstructure examination and their mechanical properties were evaluated. Further evaluation of all the samples were done using thermal aging at high temperature i.e., 200 °C for the reliability of the solder joint. The study concluded that the addition of cerium into the Sn-3.5Ag refined the microstructure by reducing the intermetallic compound particle size, which also resulted in an increase in the ultimate tensile strength. However, after the samples were treated under thermal aging at high temperature, the growth of intermetallic compounds increased hence, reducing the strength of all the solder alloys studied in this study. The least impact of thermal aging was found with the 0.3 wt. % cerium concentration into Sn-3.5Ag. Hence, the optimal content of cerium in this study was found to be 0.3 wt.% with the most refined microstructure and highest ultimate tensile strength.

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