Melting of tin using muffle furnace and microwave energy and its characterization

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Abstract: Conventional melting of metals consume significant amount of energy. Furthermore, there are possibilities of material and energy losses along with safety risks. To overcome these inherent disadvantages of conventional melting, a novel approach for melting of bulk tin using microwave energy is presented. In the present work, bulk Tin is melted using a conventional muffle furnace and a domestic multimode microwave oven. As received and as cast metals are characterised. X-Ray Diffraction (XRD) technique is used to analyse the phases present. The average tensile strength of the metal casted using muffle furnace and microwave oven is 44.1982 MPa and 50.2867 MPa respectively. Scanning Electron Microscope (SEM) is made use for the study of fractured surface of the tensile specimen, which reveals the areas of plastic deformation. Microwave processed specimen shows 10% higher tensile strength compared to that processed using muffle furnace. Radiography clearly shows cast specimen free from defects. The average hardness of as received tin is higher compared to casted specimens. However, the average hardness value of microwave processed specimen is 19.28% higher than the specimen processed using muffle furnace.

1. Introduction

Growth in the field of material processing led the industries to find new and improved methods for processing of wide range of materials. For sustainability, the developed methods should have characteristics of lower energy consumptions [1]. Microwave material processing is emerging as one of the promising sustainable process in manufacturing sector. Microwaves are electromagnetic radiations with frequency ranging 300MHz to 300 GHz. The unique characteristics of microwave processing like volumetric heating, reduced power consumption, environment green processing and yielding of enhanced quality products make it as one of the eminent processes [2]. When a material interacts with microwaves, heat is generated in the material and it is instantaneous with the irradiation of waves. Transfer of heat in materials by microwave processing is inverse in nature to conventional mode of heat transfer [3]. An additional benefit of using microwave applications is that the production facilities can be reduced to smaller sizes than needed for conventional technique, thus saving the capital investment, labour and floor space. Microwaves are being used for sintering of various metal powders and have also yielded near net shape products [4,5]. Later, microwaves were used for processing of bulk metals. Processing of metals using microwaves are used to successfully produce similar and dissimilar joints between bulk metals has been reported [6,7,8]. Furthermore, corrosion resistant clads have been developed on metallic substrate through microwave irradiation. Report shows that the hardness and resistance to wear was observed to be increased in the microwave cladded material [9,10]. Drilling of metal sheets is another area of microwave processing which has been reported [11]. Melting of various metals using multimode microwave applicator is reported, but the characterization of the same has been carried out [12]. This provides an opportunity to melt bulk tin alloy and to characterize the same.

2. Experimentation:

Melting of bulk metallic materials through microwave irradiation is difficult as they reflect microwaves at ambient temperature. Processes done in the present work are explained in this section.

2.1 Experimental setup and process:
**Metal melting using muffle furnace**

A domestic microwave oven with operating frequency of 2.45 GHz and power rating of 900W is used to melt tin. Crucible made of silica is used. The silica crucible is placed inside alumina caskets, which acts as a refractory shielding material. The casket is placed in the oven. Silicon Carbide, which is used as a susceptor is placed under the crucible. Thermocouple attached with temperature indicator is used to measure the temperature. Temperature limit is preset to 250 °C. Proportional-Integral-Derivative controller limits the temperature from exceeding the set temperature. The experiment is carried in the non-vacuum environment.

![Figure 1: Schematic diagram of casket and crucible setup](image)

1. Shielding material [(Alumina, 150x150x45)mm]
2. Crucible (silicon, 100 ml)
3. Susceptor [silicon carbide, (25x25x8)mm]
4. Molten metal

| Processing parameters     | Description                  |
|---------------------------|------------------------------|
| Microwave applicator      | Make: LG                     |
|                           | Model:MC2841SPS              |
| Woking frequency          | 2.45 GHz                     |
| Power rating              | 900 W                        |
| Exposure time             | 120 seconds                  |
| Work piece material       | Tin (60Sn40Pb)               |
| Susceptor material        | Silicon carbide              |

Schematic diagram of the experimental setup of system used is shown in Fig. 1. Parameters used in the present work are given in Table 1. In the initial stages, metal sample remains unaffected by microwaves. After a certain duration, heat developed by the susceptor inside the oven is sufficient for the microwaves to couple efficiently with the tin placed and hence the microwaves interact with the metal samples and uniform bulk heating takes place till complete melting of the sample. This is because of the fact that the microwaves couple with metallic materials at elevated temperature.

**Metal melting using muffle furnace**

A muffle furnace is a heating aid with an externally heated chamber, the walls of which radiantly heat the contents of the chamber, so that the material being heated has no direct contact with the source. A conventional muffle furnace of power 900 W is used to melt the metal. Silica crucible is used for conventional melting too. The silica crucible containing bulk metallic sample is placed inside the furnace. Temperature is set for 250 °C. Numbers of trails are carried out to know the time for complete melting of metal and found to be 59 minutes.
2.2 Characterization:

As received and cast samples are tested for their mechanical and metallurgical properties viz. tensile, hardness, XRD. Cu-Kα radiations from Rigaku Smartlab X-Ray Diffractometer at the scanning rate of 1° min⁻¹ within a range of 10° - 90° is used to find the phases formed. Specimens for tensile test are prepared according to ASTM E8 standard, a schematic of which is shown in Fig. 2. Tensile test is performed at a uniform strain rate of 1 × 10⁻² mm/s (Mecmesin Multitest 10-i micro universal testing machine). Fractographs of the specimens are captured by Hitachi S300N equipment for microstructure study. Highwood Micro Vickers Hardness Tester is utilized to measure the hardness of the samples by applying a load of 30 g with a dwell time of 10 s. Hardness values are obtained at 6 different positions and the average value is considered.

Radiography using gamma radiations (Ir-192) of strength 8Ci for 5 minutes of exposure time is carried out on as cast specimen through Single Wall Single Image (SWSI) technique agreeing to ASTM E 155 standard.

3. Result and discussion:

3.1 XRD study:

XRD spectrum of the as received, muffle furnace and microwave processed bulk tin is shown in Figure 3(a), (b) & (c) respectively.

![Figure 2: Schematic diagram of tensile specimen](image)

![Figure 3(a). XRD graph of 60Sn40Pb for as received material](image)
Table 2(a). Peaks and phases identified in as received specimen

| Peak | Phases Detected                |
|------|--------------------------------|
| a    | Lead Iron Antimony Tin Sulfide |
| b    | Copper Lead Bismuth Tin Sulfide|
| c    | Lead Tin Antimony Sulfide      |
| d    | Lead Iron Tin Antimony Sulfide |
| e    | Lead Antimony Tin Sulfide      |

XRD spectrum of as received material has fifteen noticeable peaks. Lead Iron Antimony Tin Sulfide has highest intensity peak at 2 theta value 30.699°. Other phases which show their presence are Lead Tin Antimony Sulfide, Lead Iron Tin Antimony Sulfide and Lead Antimony Tin Sulfide.

Plot showing XRD pattern of muffle furnace processed specimen is presented below in Fig. 3 (b). Muffle furnace processed specimen shows fourteen significant peaks of lead and tin.

Table 2(b). Peaks and phases detected in muffle furnace processed specimen

| Peak | Phases Detected                |
|------|--------------------------------|
| a    | Lead Tin Antimony Sulphide     |
| b    | Lead Antimony Tin Sulfide      |
| c    | Lead Tin Antimony Oxide        |

Further, muffle furnace processed specimen shows the presence of other phases, which are listed in the table 2 (b). Lead Tin Antimony Sulphide has the highest intensity peak at 2 theta value of 32.345°, which is found to be dominant. Formation of oxides could be due to the processing of the candidate material in atmospheric conditions.

Figure 3 (c) shows the XRD spectrum of the specimen processed by using microwave oven. Thirteen high intensity peaks can be identified from the XRD pattern. In microwave processed specimen, Lead Tin Antimony Oxide shows dominance with highest intensity peak at 2 theta value of 31.227°. Other phases found to show their presence are tabulated in table 2 (c).
Table 2(c). Peaks and Phases for microwave oven processed metal

| Peak | Phases Detected                        |
|------|----------------------------------------|
| A    | Lead Tin Antimony Oxide                |
| B    | Lead Iron Tin Antimony Sulfide         |
| C    | Lead Tin Antimony Sulfide              |
| d    | Lead Antimony Tin Sulfide              |

3.2. Tensile Test:

Tensile test is carried on the cast specimens in order to obtain the tensile properties. Strain rate of $1 \times 10^{-2}$ mm/s is uniformly applied on the specimen. Photograph (Fig 4(a)) shows the before the tensile test and Fig. 4(b) shows the fractured specimen after the tensile test.
The strain–stress curve for muffle furnace and microwave processed specimens are shown in Fig 5. Elastic and plastic regions of the stress-strain curve are indicated. Salient points such as Yield point (A), Ultimate tensile strength (B) and Fracture point (C) are indicated on the curve. It can be seen that the average tensile strength of muffle furnace and microwave processed specimens are 40.1982 MPa and 50.2867 MPa respectively. The tensile strength of microwave processed specimens is nearly 20% higher than the muffle furnace processed specimen. This improvement could be due to the uniform heating that takes place in microwave processing which leads to proper grain growth and in turn results in increasing the strength of the processed specimen. Average elongation of the muffle furnace processed specimen is found to be 13.12% while that of microwave processed specimen is 22.6%.

Scanning Electron Microscope is made use for the study of fractured surfaces of the tensile specimen. SEM images of the fractured surface of tensile specimens are shown in Fig 6. Investigation of the fractured surface show the areas of plastic deformation. The specimen prepared from the sample processed using muffle furnace leads to early failure in tensile test due. Excessive deformation is clearly visible in microwave processed specimen.
3.3 Porosity test (Radiography):

Radiography of the cast specimen is done by exposing the specimen to the radiations emitted by Ir-192 as shown in Fig. 7 casted metal processed in (a) muffle furnace and (b) microwave oven, to observe the presence of any casting defects. Iridium isotope source emits γ-radiations, which are passed through the specimens to be tested. A film is placed next to the test specimen. Radiations coming from the source pass through the specimen and forms dark spots if any defects are present. It can be clearly seen from the radiographs that there is no significant defect. This results in increased strength, ductility and hardness of the cast metal.

![Radiographs of (a) Muffle furnace processed specimen (b) Microwave processed specimen](image)

Figure 7: Radiographs of (a) Muffle furnace processed specimen (b) Microwave processed specimen

3.4 Microhardness Test

Microhardness test of as received sample, muffle furnace and microwave processed specimen is conducted by using a Vickers microhardness tester. Indentations were made at six different points on the sectioned surface from bottom to top of the cast metal.

The average hardness of as received metal, muffle furnace and microwave processed specimen was found to be 11.33 Hv10, 8.66 Hv10 and 10.33 Hv10 respectively.

Table 3: Parameters for Vickers microhardness number calculation

| Parameters   | As received material | Muffle furnace | Microwave oven |
|--------------|----------------------|----------------|----------------|
| Dwell time (s) | 10                   | 10             | 10             |
| Load (g)     | 30                   | 30             | 30             |
| Average hardness (Hv) | 11.33   | 8.66          | 10.33          |

3.5 Power consumption

Power consumption for melting is one of the important parameters to be studied. In this regard, power consumed by muffle furnace and microwave oven for melting of 462.976 g of Tin (60Sn40Pb) is calculated from equation (1).

\[
\text{Power consumption} = \text{Time required for melting (Hours)} \times \text{power rating (Kilo Watts)}
\]

\[
\text{Power consumption} = 0.025 \times 0.9 = 0.0225 \text{ KWh}
\]

\[
\text{Power consumed by Muffle furnace} = 0.9833 \times 0.9 = 0.885 \text{ KWh}
\]
| Parameters                        | Microwave oven | Muffle furnace |
|----------------------------------|----------------|----------------|
| Material                         | Tin (60Sn40Pb) | Tin (60Sn40Pb) |
| Quantity (g)                     | 462.976        | 462.976        |
| Time taken for melting (min.)    | 1.5            | 59             |
| Power rating (kW)                | 0.9            | 0.9            |
| Power consumption (kWh)          | 0.0225         | 0.885          |

By using **muffle furnace**, the power consumed in melting completely, the specified quantity of sample is calculated and found to be **0.885 kWh** and the time taken is 59 minutes.

By using **microwave oven**, the power consumed in melting completely, the specified quantity of sample is calculated and found to be **0.0225 kWh** and the time taken is 1.5 minutes. From the above calculation, it is clear that the power consumed in muffle furnace is around 39 times more when compared to microwave oven.

4. **Conclusion:**

i. Around 460 g of bulk tin (60Sn40Pb) is melted successfully using a microwave oven and a conventional muffle furnace.

ii. Oxides of tin and lead are found to be dominant in the as received, muffle furnace and microwave processed specimen. Formation of oxide could be due to conducting the experiment in atmospheric conditions.

iii. Tensile strength of the microwave processed specimen is found to be higher than muffle furnace processed specimen. Clear indication of plastic deformation in microwave processed specimen is observed, leading to increased strength.

iv. Hardness of the received tin is higher than the casted 60Sn40Pb. However, hardness of microwave processed specimen is around 20% higher than the specimen processed using muffle furnace.

5. **Reference:**

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