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Semisolid heat treatment processing window of Pb-40% Sn alloy for feedstock in the 3D printing thixo-forming process

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Abstract

The 3D printing thixo-forming process is a method that utilizes semisolid thixotropic feedstock for layer extrusion by controlling feedstock viscosity below the complete melting temperature of metallic alloys to form a 3D structure. The process of producing metallic feedstock has a significant impact on alloy microstructure, which is critical for the material's printability. This study aims to propose a semisolid heat treatment processing window of Pb-40% Sn alloy for use as feedstock billets for a variety of extruder nozzles (sizes 1-3 mm) during the 3D printing thixo-forming process. Firstly, Pb-40% Sn alloys were cold deformed to reduce the specimens' diameters. Semisolid treatments for deformed specimens were performed at a variety of semisolid heating temperatures and three separate holding times (2, 5, and 10 minutes). The microstructure of semisolid feedstock specimens was then investigated. The temperature and time of the semisolid heating processing window of Pb- 40% Sn alloy were optimized to feedstock specimens and thixo-extruding processes with different printed output sizes. Therefore, the proposed semisolid heat treatment processing window was a promising approach to pave the way for the low-cost manufacturing of AM, which yields higher printing rates and improved scalability of printed lines. Additionally, this approach allows for a versatile manufacturing route for producing 3D metal parts with maximum density and intricate geometries as a result of the ability to tailor the microstructure.

Keywords: Semisolid; heat treatment; Pb-Sn; Alloy; Liquid-Solid; feedstock.

1. Introduction

In contrast to subtractive manufacturing processes such as machining [1-3] where the removal of the materials is required, the additive manufacturing (AM) process involves making an object from 3D model data by adding material layer by layer [4] to generate the final product shape. Several types of additive manufacturing technologies have been introduced over recent years. Among them are metal AM technologies. There has been a rapid increase in the use of metal-based additive manufacturing (AM) techniques in high-value industries requiring net shape manufacturing. However, the majority of metal-related work in the field of AM employs costly lasers or electron beam sources [5], which necessitate advanced and expensive AM methods to produce the final product. Employing semisolid metal (SSM) processing with the AM metal-base will address many of these challenges, offer greater advantages to the manufacturing process [6] and would achieve an efficient, affordable and widely accessible metallic AM process.

The 50th anniversary of the introduction of the Semi-Solid Metal (SSM) (also termed thixo-forming) process will fall in the early 2020s. SSM was discovered around fifty years ago by Spencer at the Massachusetts Institute of Technology (MIT) while he was investigating the hot tearing of steels during solidification [7]. SSM is a method for metal forming that is exploited commercially using non-ferrous [5-8] and ferrous [9-12] metals like magnesium, aluminum alloys, copper, steel and cast iron. SSM is equally applicable to any alloy with a sufficient melting range process window. In the SSM system, the metal alloy is treated when the metal is between a liquid and solid state before being yielded to a manufacturing process. A low working temperature, die filling capability without turbulence and less solidification shrinkage are all advantages of SSM processing in comparison with traditional forming methods such as ordinary casting techniques [13]. Moreover, the finer and more uniform microstructures with lower porosities produced by SSM result in enhanced mechanical properties of the part shaped through semisolid processing [14, 15]. SSM can introduce a near-net-shaped metal part in a semisolid state with a high degree of mechanical properties and functional performance due to the special structure of the thixoformed part [8]. The development of semisolid materials with a fine and non-dendritic grain structure is a crucial aspect of SSM. The controlled microstructure and the unique material property of the flow behaviour in semi-solid-slurry contribute to the successful formation of complex parts [16]. Generally speaking, there are two broad categories of SSM processing for the production of thixotropic feedstock with the desired structure: rheo-casting and thixo-forming. [17]

The former, ‘rheo-casting,’ refers to a process in which liquid alloy associated with thixotropic properties is immediately transferred into a die without an intermediate stage of solidification. The thixo-forming process, on the other hand, involves two distinct stages to fabricate SSM feedstock: reheating modified billets and the forming process [18, 19]. Therefore, the feedstock materials should be specially pre-alloyed using unique methods before reheating in order to achieve non-dendritic microstructures [20], after which they can be used for component creating.

A number of thixo-forming methods have been introduced within the industry for producing semisolid billets with spherical microstructures. Owing to its low cost and simplicity, one of the most efficient and commercially accessible solid-state routes to thixotropic feedstock is the Recrystallization and Partial Melting (RAP) method developed by Kirkwood et al. [21]. This process involves two essential steps of deformation followed by isothermal heat treatment. Nevertheless, prior to the RAP process, a dendritic cast microstructure is prepared in suitable forms through a conventional casting process. Subsequently, during the RAP procedure, the material is exposed to deformation by cold or warm temperatures falling below the recrystallization temperature. Then, the deformed material is subjected to a partial re-melting to a semisolid state and isothermally held for a period of time to introduce the thixoformed billet [22]. The deformation stage may be completed using a variety of different techniques, including rolling, forging, upsetting, and extrusion [23].

Semisolid heat treatment is a critical factor in managing semisolid microstructure in the thixo-forming process and is usually accomplished using an induction heating system to ensure an accurate and quick heating rate. During isothermal heat treatment, the slurry is heated to a semisolid temperature at which the solid and liquid phases coexist in equilibrium to ensure that the solid phase is transformed into a globular morphology with fine particle size, as well as to obtain a desirable nominal liquid fraction [24-27].

Semisolid feedstock is comprised of several special characteristics, and using these unique properties for additively-produced components opens up the prospect of advanced technology. Due to the excellent mechanical and rheological properties of semisolid billets, it is expected that completely dense 3D printed components can be formed [6]. As a result, they can be used in functional applications. Furthermore, optimal feedstock viscosity results in successful extrusion and deposition of semisolid slurries and the formation of 3D structures [16, 28].

The preparation of a solid filament and development of a thixo-extrusion process of Pb-40 Sn alloy for additive manufacturing of metals in semisolid state have been investigated [29-31] in previous studies. It was reported that wire pre-treatment process variables have a strong effect on the metallic filament microstructure and an important role in the alloy’s thixo-extrudability. Although previous studies [29-31] reported that the proper design of the thixo-extruder and solid fraction of Pb-Sn alloy was the most influential factor in the thixo-extrusion process, the optimum semisolid heating temperature (fraction of solid) and isothermal holding time for Pb-40Sn as feedstock in the 3D printing for a wide extruder nozzle size (1-3 mm) remain ambiguous. This research focuses on the optimization of the semisolid heat treatment of Pb-Sn alloy for use as feedstock billets for a wide range of extruder nozzle sizes. Consequently, a semisolid heat treatment processing window of Pb-40% Sn alloy for use as feedstock in the 3D printing thixo-forming process was proposed. The initial outcomes of printed samples are offered in this study.

2. Experimental Procedures

Ingot alloys with a chemical composition of 60%Pb-40%Sn were supplied by Solderking Assembly Materials Ltd. UK. Scanning calorimetry (DSC) examinations were conducted using an SDT Q600 V20.9 Build 20 instrument to perform thermal analysis of metal alloy to evaluate the solidus and liquidus temperatures and liquid fraction and determine the optimum working temperatures window. The Pb-40 Sn specimen of 20 mg was heated to 250 ºC with a heating rate of 10 °C/min on a nitrogen gas atmosphere with a flow rate of 100 ml/min in order to mitigate the impact of oxidization. The solidus and liquidus temperatures were calculated, and the curve of the liquid fraction versus the heating temperature was obtained by integrating the DSC curve.

The as-cast ingots were machined into cylindrical bars with a diameter of 20 mm and a length of 25mm. An extrusion die set was designed to reduce the diameter of the starting materials and prepare billets for heat-treatment processes. The extrusion process was carried out at room temperature using a hydraulic press with a maximum force of 250KN, as shown in Fig. 1.

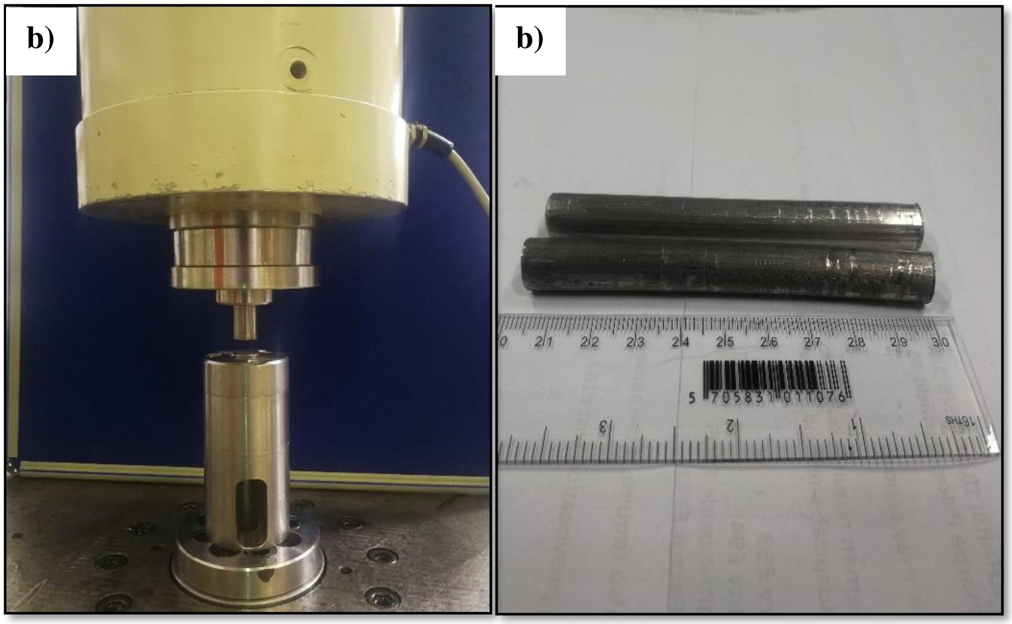


Fig. 1. a) Experimental setup of extrusion using a hydraulic machine and b) Specimen rods deformed by extrusion

An electric furnace (Carbolite Furnaces model STF 16/180) was used for isothermal semisolid heat treatment of feedstock Pb-40%Sn specimens. The specimens were heated to a range of isothermal temperatures and held at three separate time intervals (2, 5, 10 min) to investigate the microstructure development of grain growth and spheroidisation in the semisolid region. After each isothermal treatment, the specimens were immediately quenched in water at room temperature. To assess the viability of employing thixo-forming processes in the metal additive manufacturing technology of semisolid metals to create 3D structures, experiments were performed through a 3D printing thixo forming process using a number of extrusion nozzle sizes. The 3D printer was designed and manufactured in-house. Its design is shown in Fig. 2.

To perform microstructure investigations, the specimens were cold mounted in an epoxy resin material and then cured at ambient temperature for 12 hours. The specimens were ground using sandpapers (P320, P600, P1200 and P2500-grit silicon carbide), mechanically polished with different grades of diamond suspension (3µm, 1µm and 0.05µm) and etched with natal (2%) solution. An optical microscope (Nikon Eclipse LV150) was used for metallurgical observations and microstructure characterisations. The quantitative image measurements of the examined samples were calculated using the image software analyzer, and the size and sphericity (shape factor) measurements of the primary solid grains are defined. The average diameter of grain size (D) and the shape factor (FS) were calculated based on the following equations [32]:

$$SF = 4\pi A/P^2 \qquad \text{Equation 1}$$

$$D = 2\sqrt{(A/\pi)} \qquad \text{Equation 2}$$

Where A is the examined area of the grain and P is the perimeter of the primary grain.

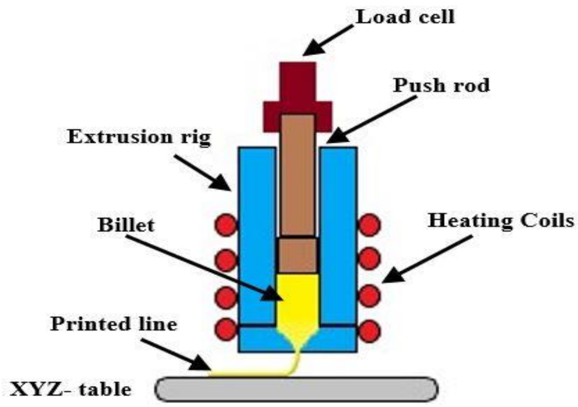


Fig. 2. Schematic of the 3D printing process

3. Results and Discussion

The main aspect of semisolid processes is the preparation of semisolid materials with a thixotropic microstructure. The morphology of this phase is a spheroidal structure with a fine grain size [33]. One advantage of this unique microstructure is its strong positive influence on the flow behavior of semisolid slurry, which allows for parts to be prepared efficiently without typical defects that can be found during the conventional casting process. Furthermore, the produced components which present fine and non-dendritic structures benefit from better mechanical properties compared with other conventional manufacturing processes [34]. In this study, the RAP method was adopted to obtain thixotropic feedstock. It is proposed that the initial microstructural morphology of materials will be achieved by a deformation using an extrusion-die at room temperature with subsequent partial melting in a furnace. Then, the pre-alloyed feedstock will be fed into the 3D printing thixo-forming process, allowing successful extrusion of semisolid slurries and the creation of 3D metallic structures.

3.1 Thermal properties of the as-received Pb-40%Sn alloy

Fig. 3 shows the Pb-Sn phase diagram with the vertical line of the current Pb-40% Sn study alloy. It is clear that the Pb-40% Sn has a reasonable range of solidification that facilities its semisolid heat treatment processing. The solidification of Pb-40% Sn started with the primary Pb phase, and, through cooling Pb + liquid, transformed to Pb + eutectic (Pb and Sn). Liquid fraction vs. heating temperature of Pb-40% Sn alloy is shown in Fig. 4. It is clear that the fraction of liquid changed smoothly from high temperature by cooling up to approximately 0.3 liquid fraction. Fig. 5 shows the DSC curve and liquid fraction profile. At 185 ºC, a high heat flow associated with the sudden drop in fraction of liquid is observed. This sudden drop in fraction of liquid confirms the eutectic transformation of Pb-40% Sn alloy.

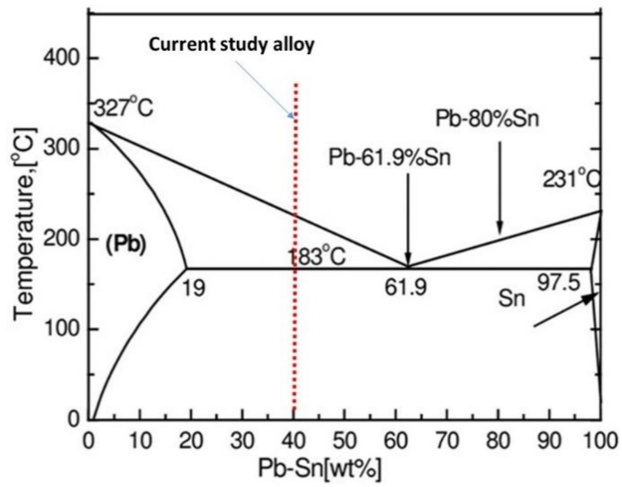


Fig. 3. Pb-Sn Phase diagram showing the vertical line of current study alloy [35].

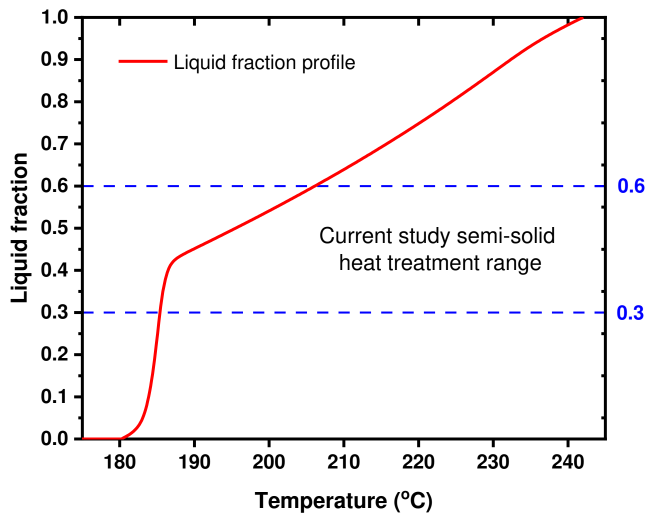


Fig. 4. Liquid fraction vs. heating temperature.

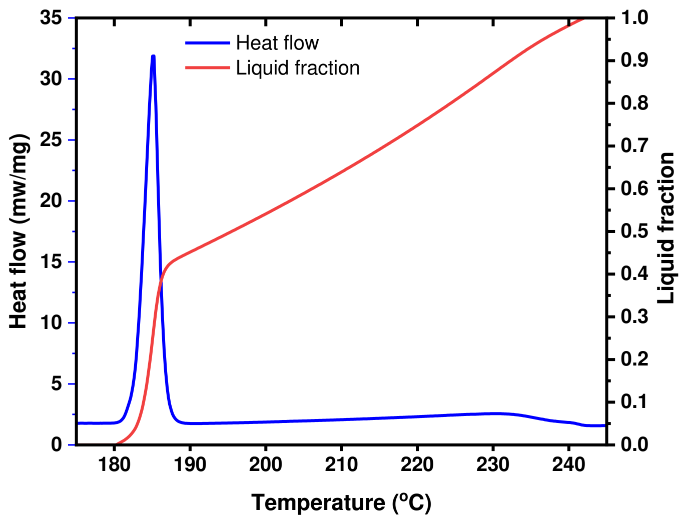


Fig. 5. DSC curve and liquid fraction profile

A relation between liquid fraction (Lf) and isothermal holding temperature is derived from the thermal analysis of Pb-40% Sn alloy, (Table 1). Liquid fraction vs. isothermal holding temperature and proposed semisolid isothermal holding time ranging from eutectic temperature 185 °C (Lf=0.3) to 206 °C (Lf= 0.6) are presented in Table 1. To successfully fabricate layers, the material is added layer-by-layer to build a product that presents good geometric flexibility as well as great potential savings in time and costs [36]. The maximum liquid fraction of 0.6 (206 °C) was selected for control of the final dimensions of the fabricated layer. Three semisolid isothermal time intervals of 2, 5 and 10 minutes for each isothermal temperature are proposed to observe microstructure change, especially in grain size and particle sphericity.

Table 1. Liquid fraction vs. isothermal holding temperature and proposed isothermal holding time.

Liquid fraction %	Isothermal holding temperature (°C)	Isothermal holding time (min.)	Isothermal holding time (min.)	Isothermal holding time (min.)
0.3	185	2	5	10
0.4	186.5	2	5	10
0.45	190	2	5	10
0.5	195	2	5	10
0.55	201	2	5	10
0.6	206	2	5	10

3.2 Microstructure evolution during the semisolid isothermal heat treatment process

The microstructure of as-cast and as-extruded Pb-40% Sn alloy are presented in Fig. 6. A dendritic grain structure of the primary phase (Pb) is clearly observed in the as-cast alloy (Fig. 6 a & b). On the other hand, elongated columnar dendrites are observed in extruded Pb-40% specimens (Fig. 6 c & d). The microstructures of as-received and as-extruded Pb-40% Sn specimen are ordinarily founded in casting and extrusion processes. Those kinds of dendritic and elongated grain structures are highly affected by the homogeneity and mechanical properties of the final fabricated products.

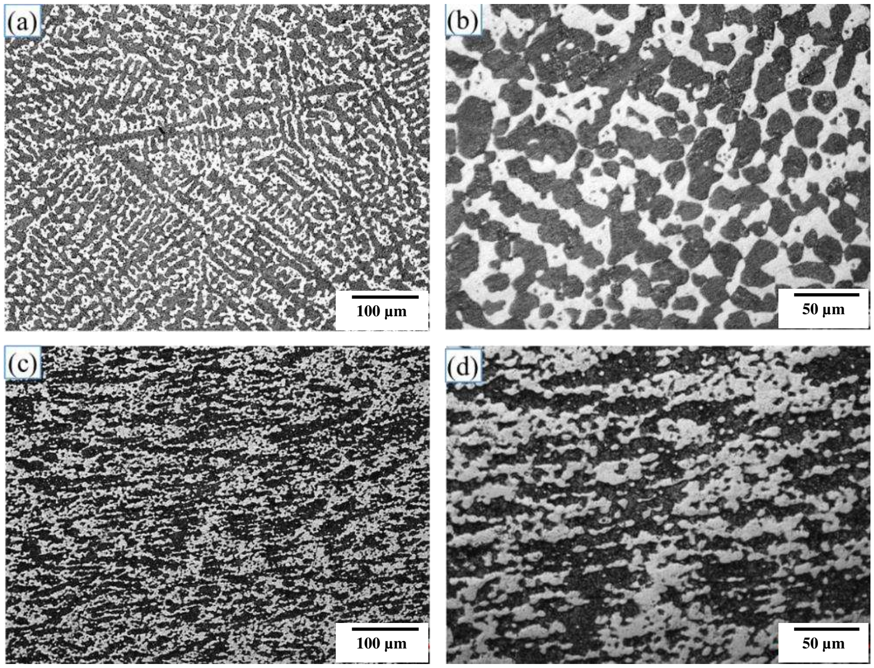


Fig. 6. Microstructure of as-cast (a) & (b) at lower and higher magnification and as-extruded (c) &(d) at lower and higher magnification, respectively.

In contrast with as-cast and as-extruded microstructures, microstructures of semisolid heat-treated specimens at different temperatures for different heating times show more globular and homogeneous results (Fig. 7). As a general observation among all semisolid heating temperature ranges and all three holding times, the heat treatment improved both grain size and grain sphericity compared with the as-cast and extruded specimens. The fraction of liquid increases as the heating temperature and time increase in the semisolid state range.

The partial re-melting of semisolid Pb-40% Sn specimens is a critical procedure in the thixo-forming process, whereas obtaining a desirable optimum liquid fraction is not only the aim but also the manner of ensuring the transformation of the primary solid phase fraction to a spheroidal morphology with fine grain size. During semisolid heat treatment, the Pb-40% Sn alloy was heated up to a temperature at which the primary solid and liquid phases coexist in equilibrium and the desirable liquid fractions are achieved through the control of the heating temperature as well as the holding time. However, the coarsening of primary grains could be observed after a long holding period [33, 37]. Within the current study, the rate of microstructural coarsening of the primary phase is not significantly observed for a holding time of up to 10 minutes (Fig. 7).

The primary solid shape factor and grain size of Pb-40 % Sn alloy at different heating temperatures and three different holding times are shown in Fig. 8. The quantitative measurements of solid shape factor and grain size of Pb-40% Sn alloy show that heating the alloy to 190 ºC (Liquid fraction of 0.45) for 2, 5 and 10-minute intervals shows the optimum microstructure considering a compromise of both shape factor and primary phase. The current and the previous study [29-31] reveal an agreement between the same mentioned result. Although a heating temperature of 190 ºC appears to result in the best globular and fine structure, the semisolid heating of Pb-40% Sn alloy at this temperature could not produce the optimum heating condition for a feedstock, nor during the 3D printing thixo-forming process. For semisolid feedstock heating, the optimum shape factor and grain size conditions were produced at a temperature of 186.5 ºC (Lf = 0.4) and a 5 min interval, considering the specimen's shape during semisolid heat treatment. The fraction of solid material should be enough to attain stable shape and dimensions among Pb-Sn specimens during semisolid heat treatment.

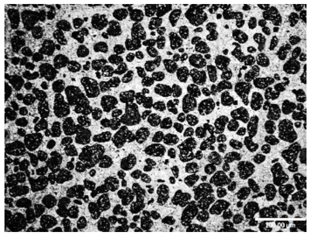
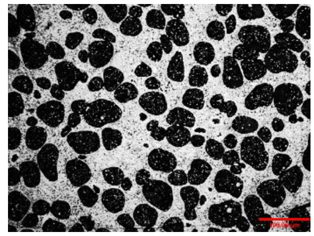
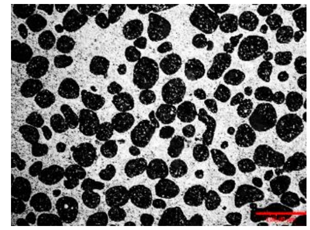
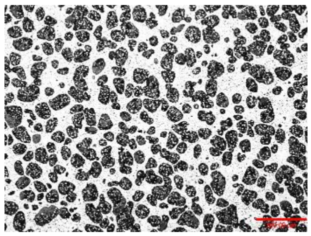
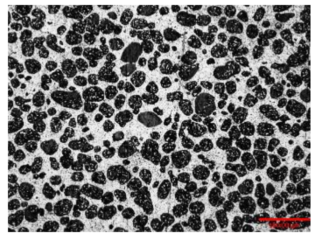
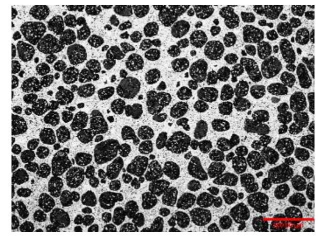
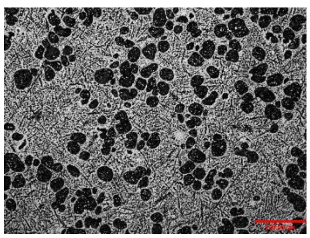
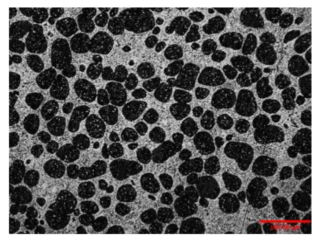
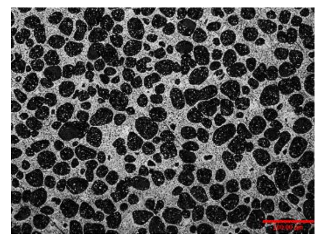
Semi-solid temperature/ Time	2 min	5 min	10 min
185 ºC **Liquid fraction= 0.3**			
190 ºC **Liquid fraction= 0.45**			
206 ºC **Liquid fraction= 0.6**			

Fig. 7. Microstructure of semisolid heat-treated specimens at different temperatures for different heating times.

The optimum temperature and time conditions for heating Pb- 40% Sn during thixo-extruding processes are primarily dependent on the design of the extruder, particularly the output orifice. For a narrow extruder orifice of 1-1.5 mm, 195 ºC ($L_f = 0.5$) during a 2-minute period results in the optimum shape factor and grain size conditions. On the other hand, for a wider extruder orifice of 2.5-3 mm, a 186.5 º C ($L_f = 0.4$) temperature for 5 minutes results in the optimum shape factor and grain size conditions. For a medium-size extruder offset of 1.5-2.5 mm, a temperature of 190 º C ($L_f = 0.45$) for 2 min results in the optimum shape factor and grain size conditions.

To facilitate AM of metals and alloys for rapid printing rates, scalable filament sizes as well as a high thermal efficiency should be considered. This semisolid heat treatment approach, termed semisolid heat treatment processing window (SSHPW), takes advantage of inexpensive, well-structured preparing feedstock, allows for a controlled printing process and minimizes device complexity and cost. An example of a 3D deposited layer is illustrated in Fig. 9a. Fig. 9b shows an image of the resulting microstructure, exhibiting globular microstructures of the printed feature. The feedstock billet was printed at 186.5 ºC, with an extrusion speed of 5.0 mm/s and an orifice diameter of 3 mm. The velocity of the substrate was held constant at 20 mm/s.

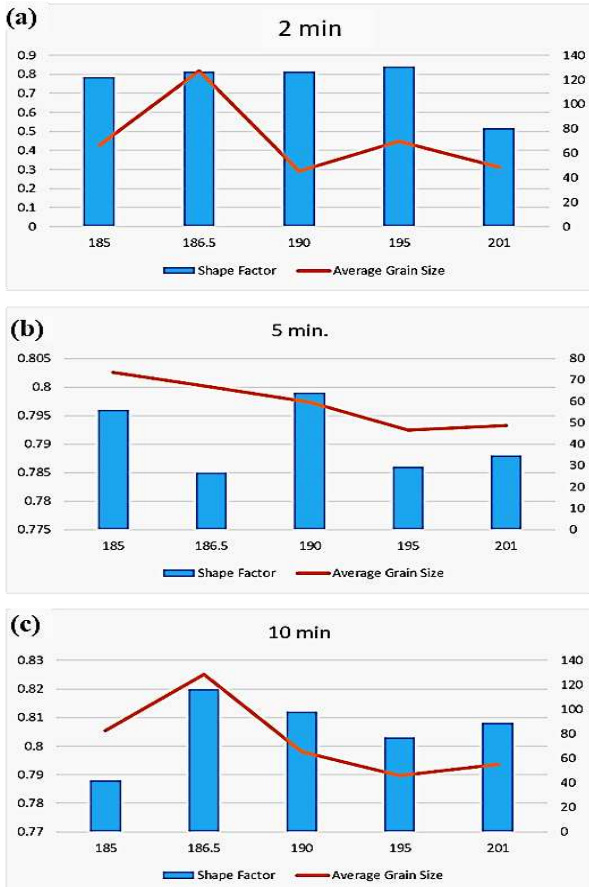


Fig. 8. Primary solid shape factor and grain size of Pb-Sn alloy at different heating temperatures and times.

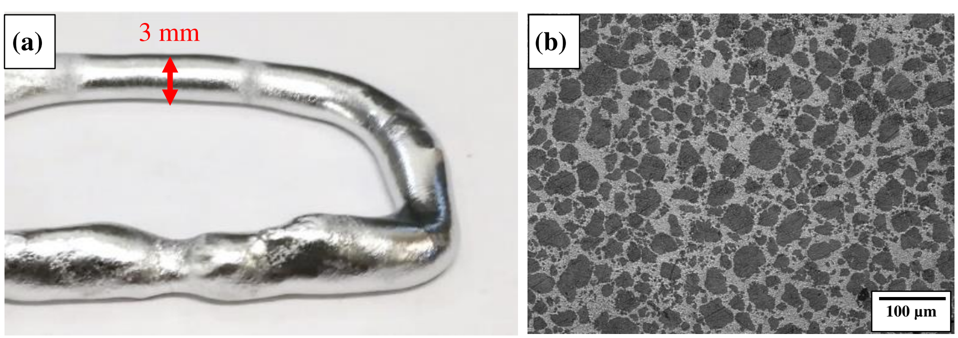


Fig. 9. (a) Printed sample. (b) Typical microstructure of a Pb-40% Sn alloy extruded by 3D printing thixo-forming process

4. Conclusion

This research examined a semisolid heat treatment processing window (SSHPW) of Pb-40% Sn alloy for use as feedstock in the 3D printing thixo-forming process. Microstructural analysis revealed that the suggested semisolid heat treatments improve the primary grain to be globular and finer for optimum feedstock billets structure as well as thixo-extruding conditions. Key findings of the present work are as follows:

- The proposed semisolid heat treatment processing window is a promising approach to pave the way for low-cost manufacturing of AM and is associated with high printing rates and improved scalability of printed lines.

- The optimum temperature and time heating conditions of Pb- 40% Sn during thixo-extruding are primarily dependent on the design of the extruder, especially the output orifice.

- For feedstock semisolid heating, a heating temperature of 186.5 oC (Lf = 0.4) at a 5-minute interval results in the optimum shape factor and grain size conditions considering specimen shape during semisolid heat treatment.

- The current approach allows for a versatile manufacturing route for producing 3D metal parts with maximum density and intricate geometries as a result of the ability to tailor the microstructure.

- This promising approach was introduced to address the limitations and drawbacks mentioned in the literature.

- The process and results of the SSHPW presented in the study are applicable to a wide variety of semisolid materials to prepare thixotropic feedstock for optimized AM methods to develop high-performance 3D structures.

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