Extrusion-Based 3D Printing of CuSn10 Bronze Parts: Production and Characterization

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Abstract: The interest in producing cost-effective 3D printed metallic materials is increasing day by day. One of these methods, which has gained much attention recently, is the fused deposition modelling (FDM) method. The parameters used in the FDM method have significant effects on the printed part properties. In this study, CuSn10 bronze alloy was successfully produced. The printing speed and layer thickness were investigated as the printing process parameters, and their effect on morphological properties was characterized by using SEM. As a result, it was observed that the formation of printing-induced voids was prevented by applying a layer thickness of 0.2 mm. Additionally, by increasing printing speed, a slight decrease in product density was observed. Following determination of 3D printing parameters which give the highest printed part density, the parts were debound in hexane solution via solvent debinding. Finally, the parts were sintered at 850, 875 and 900 °C for 5 h to examine effect of sintering temperature on density, porosity, shape deformation and mechanical properties. Although partial slumping started to form over 875 °C, the highest density (94.19% of theoretical density) and strength (212 ± 17.72 MPa) were obtained by using 900 °C as the sintering temperature.

Keywords: 3D printing; sintering; CuSn10; mechanical properties; characterization

1. Introduction

The 3D printing process, known as additive manufacturing (AM) method, is the production of parts layer by layer with a device that uses computer-aided design (CAD). This process does not require molding, cutting, drilling, or machine processes [1,2]. Three-dimensional printing has many advantages and allows producing sophisticated tools without material and energy waste. In addition, this method provides low-cost and simple printing at home and in laboratories [3].

Different types of 3D printing techniques can be used for the production of metallic materials [4,5]. The selective laser melting (SLM) method is commonly preferred to print metallic materials among these techniques. However, the production costs of metallic materials are high for the manufacturer since the SLM method depends on high-energy laser sources. Cost-efficient alternatives to the SLM have been developed for the 3D printing of metallic materials. One of the relatively novel alternative methods is fused deposition modeling (FDM). In this technique, the feedstock contains metallic powders and polymeric binders. Binders provide the required viscosity for extrusion of metallic powders while printing and keep powders in a desired shape during sintering. After printing, the green sample undergoes debinding which enables partially removing of polymeric constituents, and the brown sample (debound sample) is sintered to obtain bulk metallic part. However, the FDM process parameters have significant effects on the printed part. One of the important points in the production of metallic parts with the FDM method is the production of parts with the highest possible density after printing.
Additionally, Viswanath et al. stated that large voids caused by printing could not be removed after sintering [6].

There is increasing interest in the production of metals and alloys by this relatively new method. Various steel alloys, copper, magnesium alloys, titanium alloys were produced by the FDM method [6–12]. Due to their good thermal and electrical properties, Cu and Cu-alloys are some of the most widely utilized non-ferrous metals in the industry. Additionally, the high corrosion resistance, wear resistance, and strength of these alloys make them suitable for different applications. Among Cu-based alloys, CuSn10 alloys are widely used especially as bearing materials due to their good mechanical properties and high wear resistance. Traditional methods for fabricating Cu-Sn alloy parts, such as casting and mechanical alloying, have been extensively studied [13]. However, printability, microstructure, phases, and mechanical properties of CuSn10 alloy parts made by the FDM method are not present in the literature. In light of this trend, in this study, the FDM process of CuSn10 alloy is studied. The study covers the printing and sintering of feedstock at selected conditions. The printing parameters giving the highest density were determined and the effect of sintering temperature on density, porosity, shape distortion, and mechanical properties was investigated.

2. Materials and Methods

2.1. Materials

In the present study, pre-alloyed, gas atomized CuSn10 bronze alloy powder (Molchem Chemical Technologies, London, UK) was used for the preparation of feedstock. Polyethylene-wax (PE-W) and polypropylene (PP) were used as primary and backbone polymers, respectively, and for the dispersion of metal powders, stearic acid was used as the surfactant.

2.2. Production

Bronze powder content of feedstock was fixed at 55 vol.% PE-W, PP, and SA content of binder mixture were fixed at 65 wt.%, 30 wt.%, and 5 wt.% respectively. Binders and metal powder were melt-mixed at 190 °C, for 30 min. After mixing, the feedstock was ground manually and sieved to a size between 1.7 mm and 1 mm (Retsch AS 200 Basic, Retsch GmbH, Düsseldorf, Germany). Subsequently, these pellets were fed into the extruder part of the 3D printer.

Printing trials were performed on Anet A8 (Shenzhen Anet Technology Co., Ltd., Shenzhen, China) 3D printer. Anet A8 is a cartesian type desktop printer with a printing size of 220 × 220 × 240 mm (x × y × z). The filament extruder part of the 3D printer was replaced by a system with a screw to enable pellet printing. A custom-designed pellet extruder contains a screw with a diameter of 8 mm and a channel depth of 2 mm. A cross-sectional image of the pellet extruder is shown in Figure 1.

The software Cura v3.6.0 (Ultimaker B.V., Utrecht, The Netherland) was used to prepare the G-code for printing. In this software, the following parameters were kept constant: the nozzle temperature of 210 °C; infill density of 100%, rectilinear fill pattern for all layers, a fill angle of 45° and the cooling fan speed was set to 0% for the first layer, and 100% for upper layers. In the study, a 0.6 mm diameter steel nozzle was used to prevent wear during the printing process. The layer thickness of the prints varied between 0.2 and 0.6 mm and also the printing speed varied between 20 and 60 mm/s. Square prism samples with dimensions of 10 mm × 10 mm × 5 mm (x × y × z) were printed for density determination and microstructural investigation. The experimental program of study is given in Table 1.
Figure 1. Schematic representation of pellet extruder.

Table 1. The experimental program of study.

| Number | Layer Thickness (mm) | Printing Speed (mm/s) | Nozzle Temperature (°C) | Sintering Temperature (°C) |
|--------|----------------------|-----------------------|-------------------------|---------------------------|
| 1      | 0.2                  | 20                    | 210                     | 850 (*)                   |
| 2      | 0.2                  | 20                    | 210                     | 875 (*)                   |
| 3      | 0.2                  | 20                    | 210                     | 900 (*)                   |
| 4      | 0.2                  | 40                    | 210                     | -                         |
| 5      | 0.2                  | 40                    | 210                     | -                         |
| 6      | 0.2                  | 60                    | 210                     | -                         |
| 7      | 0.2                  | 60                    | 210                     | -                         |
| 8      | 0.2                  | 60                    | 210                     | -                         |
| 9      | 0.4                  | 20                    | 210                     | -                         |
| 10     | 0.4                  | 20                    | 210                     | -                         |
| 11     | 0.4                  | 20                    | 210                     | -                         |
| 12     | 0.6                  | 20                    | 210                     | -                         |
| 13     | 0.6                  | 20                    | 210                     | -                         |
| 14     | 0.6                  | 20                    | 210                     | -                         |

*Three replications—.

Printed parts were debound in hexane solution at 60 °C for 16 h. After the solvent debinding process, printed samples were placed in an Al₂O₃ crucible and covered with graphite powder to prevent oxidation during sintering. Debound parts were sintered using an electrical furnace (Protherm PLF 140/5) (Protherm Furnaces, Ankara, Turkey). First, the furnace was set to 550 °C with a heating rate of 1 °C/min for 2 h to remove residual binders in the structure after the solvent debinding process. Then, the temperature was raised to the sintering temperature with a heating rate of 3 °C/min and held at sintering temperature for 5 h. After the sintering process, samples were allowed to cool in the furnace. Sintering was carried out at different temperatures (850 °C, 875 °C, and 900 °C) to examine the effect of temperature on porosity, density, shape stability, and mechanical properties of metal samples.
Cylinder samples of 8 mm × 8 mm (diameter × height) were printed and sintered for determination of shape distortion during sintering. The dimensionless distortion parameter (δ) was used to quantify the distortion. It was calculated by the following Equation (1).

\[
\delta = 100 \times \left[ \frac{h_0 \times r}{R \times r_0} \times 10^\left( \frac{\sigma_r}{r} + \frac{\sigma_h}{h} \right) - 1 \right]
\]  

(1)

where, \(r_0, h_0\) refers to the initial radius and initial height of the green sample; \(r\) and \(h\) refer to the mean radius and height of distorted sample, respectively; and \(\sigma_r, \sigma_h\) stand for the standard deviation of measured radius and height of the sintered samples, respectively. Sengupta et al. indicated that a value of the distortion parameter equal to zero denotes isotropic dimensional changes within the compact, which signifies no distortion in the sintered sample [14,15].

2.3. Characterization

Particle size distribution analysis of bronze powder was carried out by using a laser scattering particle size distribution analyzer (HORIBA, Ltd., Kyoto, Japan).

The surface and cross-sectional morphologies of printed samples and morphology of bronze powder (as received) were examined by scanning electron microscope (SEM, JEOL Ltd., Tokyo, Japan). SEM images were obtained with an accelerating voltage of 5 kV for printed samples. Samples were coated with Au-Pd by sputter coating prior to scanning to avoid electron beam charging effects during examination.

The thermal behavior of the feedstock was determined by thermogravimetric and differential thermal analysis (TG/DTA). TG/DTA analysis was performed by Shimadzu DTG-60H (Shimadzu Co., Kyoto, Japan) by heating from room temperature to 700 °C for feedstock and 1100 °C for bronze powder respectively by a heating rate of 10 °C/min under N₂ atmosphere to avoid oxidation effects.

Phase structures of printed, debound, sintered samples and bronze powder (as received) were analyzed using X-ray diffraction (XRD) (Rigaku, D/Max-2200/PC, Rigaku Co., Tokyo, Japan). X-ray radiation of Cu Kα was set at 40 kV and 36 mA with a scanning speed of 4°/min, from 3° to 90° of diffraction angle.

Tensile properties of sintered parts were determined by using a universal tensile testing machine (Shimadzu Autograph AG-IS Series, Shimadzu Co., Kyoto, Japan) with a capacity of 250 kN equipped with a video extensometer system (SHIMADZU Non-Contact Video Extensometer DVE-101/201, Shimadzu Co., Kyoto, Japan) with a crosshead speed of 1 mm/min. The tensile test specimen dimension is shown in Figure 2.

![Figure 2: Tensile test specimen.](image)

3. Results and Discussion

Figure 3 illustrates that the particle shape of the powder was found irregular. The powder size was 18 µm, 46 µm, and 107 µm for D10, D50, and D90, respectively.
Figure 3. SEM image of bronze powder.

Figure 4a,b show the TGA and DTA curves of the feedstock and binder components, respectively. It is seen that the weight loss for PE-W occurs in a wide range between 220–500 °C. For PP, the weight loss occurred in a narrower range between 450 and 500 °C. For feedstock, the weight change occurred as a combination of the behavior of these two binders. When the DTA curves are examined, it is seen that there is a peak around 118 °C in the PE-W curve and a wide endothermic peak at 475 °C. It is seen that the PP curve exhibits endothermic peaks at 172 and 461 °C. Similarly, feedstock has endothermic peaks at 120, 156, and 465 °C as the combination of these two curves. When the TGA and DTA curves are examined together, the endothermic peaks at low temperatures can be attributed to melting. Although an endothermic reaction occurs at these temperatures, there is no significant change in weight. Additionally, endothermic peaks at high temperatures indicate the decomposition temperatures of the binders. At these temperatures, the weights decreased quite rapidly and almost all the materials decomposed at these temperatures for binders. Considering these results, limit values for nozzle temperature can be determined. For printing to take place, all binders must be melted at the printing temperature. On the other hand, binders should not decompose, or feedstock weight should not change at the printing temperature for stable printing. Therefore, in this study, nozzle temperature was set to a temperature of 210 °C.

SEM images of the printed sample surface and cross-sections with different printing speeds (20–60 mm/s) are shown in Figure 5. As shown in the figure, there is no distinct change between the samples in terms of surface morphology. On the other hand, with the increasing printing speed, the deposited lines became more noticeable. Although the deposited lines had a rectangular shape, small gaps began to appear at the contact regions. According to the SEM images, it is clear that the increased printing speed caused the formation of voids due to printing.

Similarly, the apparent density values of printed samples decreased with increased printing speed. This can be attributed to the formation of printing-induced porosity. The porosity increased with increasing printing speed. Similarly, the apparent density values of the parts decreased slightly due to increased printing speed. Apparent density values were measured as 5.59 ± 0.09 g/cm³, 5.34 ± 0.22 g/cm³, and 4.90 ± 0.10 g/cm³ for print speeds of 20 mm/s, 40 mm/s, and 60 mm/s, respectively. These results are consistent with the SEM results. Due to the increased printing speed, the small gaps formed in the contact areas of the deposited lines caused a decrease in the apparent density. Yang et al. and Carneiro et al. also observed that the density decreased depending on the increasing printing speed in their studies and explained this situation as the decrease in the deposited linewidth due to the increased printing speed [16–18].
By applying different layer thicknesses, it was observed that a significant change between the samples regarding the cross-sectional shape of individual lines and gaps between lines (see Figure 6). A cross-sectional image of the printed sample with a layer thickness of 0.2 mm showed that there is no appearance of the individual deposited lines and induced gaps between the lines in the samples, which means that using a low layer thickness of 0.2 mm provided well contact of lines. Kuznetsov et al. indicated that the cross-sectional shape of deposited lines is an elongated rectangle when the ratio of layer thickness to nozzle diameter is low. As the layer thickness increases, the line thickness increases. The cross-sectional shape of the line starts to resemble a circle, and the line contact becomes more tangential, and as a result, the gap between lines increases [19].

The sintering process and its parameters are important to achieve low porosity and near-full theoretical density and good mechanical and physical properties [20]. To examine the effect of sintering temperature on the physical and mechanical properties, samples were printed with the same parameters and sintered at different temperatures (from 850 to 900 °C for five hours). Optical images of sintered samples at different temperatures are shown in Figure 7. The densities and porosities of samples are given in Table 2. The highest porosity was obtained from the sintered sample at 850 °C with 19.33 ± 3.91%. The images indicate that pores have irregular shapes and indicate that 850 °C is insufficient for neck growth in the diffusion process of sintering. It is seen that the porosity significantly decreased depending on the increased sintering temperature of 875 °C. The porosity decreased to 7.95 ± 1.62% and irregularly shaped pores were located at grain boundaries. The highest density of 8.27 ± 0.19 g/cm³ and the lowest porosity of 4.45 ± 2.89% were obtained at
900 °C, which is the highest sintering temperature used in the present study. The shape of the pores became more rounded. Similar behavior of density with respect to sintering temperature was observed by Singh and Pandey [21].

![Surface and Cross-Sectional SEM Images](image)

**Figure 5.** Surface and cross-sectional SEM images of printed samples at different speeds (a,b) 20 mm/s, (c,d) 40 mm/s and (e,f) 60 mm/s.

**Table 2.** Porosity, density values and microstructures of samples after sintering.

| Sintering Temperature | Porosity (%) | Density (g/cm³) |
|-----------------------|--------------|-----------------|
| 850 °C                | 19.33 ± 3.91 | 7.11 ± 0.39     |
| 875 °C                | 7.95 ± 1.62  | 7.92 ± 0.13     |
| 900 °C                | 4.45 ± 2.89  | 8.27 ± 0.19     |

XRD graphs of bronze powder (as received) and sintered samples are shown in Figure 8. Regardless of the sintering temperatures, all samples consist of a single solid-solution α-phase (JCPDS no. 044-1477). According to results, sintered samples were found to be free from any contamination or oxide formation.

Maintaining the uniform shape of a product following the sintering temperature is an important feature for the additive manufacturing process [22]. Figure 9 shows the images of the samples after 5 h of sintering at 850 °C, 875 °C, and 900 °C, respectively, and TG/DTA graph of bronze powder. As a result of sintering at 850 °C, there was no obvious shape distortion in the sample. The calculated shape distortion parameter value is 4.37 ± 0.48. When the sintering temperature was increased to 875 °C, liquid phase formation occurred.
during sintering, and the value of the shape distortion parameter increased slightly to 5.03 ± 0.34. The broad endothermic peak located between 864 °C and 1046 °C in the DTA graph, corresponds to solidus-liquidus transition.

Figure 6. Surface and cross-sectional SEM images of printed samples at layer thickness of (a,b) 0.2 mm, (c,d) 0.4 mm and (e,f) 0.6 mm.

When the sintering temperature was 900 °C, the amount of liquid phase in the structure increased during sintering and the highest shape distortion parameter value was obtained 8.57 ± 0.83. This issue is reported by Mousapour et al. They indicated that when bronze samples are sintered at relatively higher temperatures than the solidus temperature, liquid fractions increase, and the sample slump when the solid-liquid structure is too weak to sustain gravity, which results in compression in the axial direction and expansion in the radial direction [23].

Figure 10a,b show test samples after printing and sintering, respectively. The stress-strain curves of sintered (at 850 °C, 875 °C, and 900 °C) bronze samples are shown in Figure 11, and the mechanical properties are summarized in Table 3. Strength values of sintered samples increased with increasing sintering temperature. Moreover, an increase in elongation values was observed with increasing sintering temperature. Higher sintering temperature causes size reduction in porosity. This improves the mechanical properties of sintered samples, by a change in the microstructure, which could be explained with the bond formation between particles. Therefore, the improved bond formation promotes an increase in strength of the parts as reported by Wahi et al. [24].
Figure 7. Microstructural images of sintered samples at (a) 850 °C, (b) 875 °C and (c) 900 °C.

Figure 8. XRD results of sintered bronze samples and powder (as received).
| Sintering Temperature (°C) | Yield Strength (MPa) | Ultimate Tensile Strength (MPa) | Elongation at Break (%) |
|----------------------------|-----------------------|---------------------------------|-------------------------|
| 850                        | 43.4 ± 11.48          | 77.3 ± 3.85                     | 11.38 ± 1.53            |
| 875                        | 103.6 ± 3.09          | 191 ± 5.35                      | 33.78 ± 1.61            |
| 900                        | 110.66 ± 2.62         | 212 ± 17.72                     | 35.65 ± 4.3             |

Figure 9. Shape distortion test samples sintered at 850 to 900 °C from left to right and TG/DTA graph of bronze powder.

Figure 10. (a) Printed and (b) sintered test samples.
Scudino et al. investigated the properties of CuSn10 parts produced by the selective laser melting (SLM) method. They revealed that the parts have 420 MPa tensile strength [25]. The tensile strength of the bronze part produced by the FDM method (900 °C) corresponds to ~50% of the SLM method. The SLM method allows the production of high-density parts, which contain fine-grained structure and low porosity. Rapid cooling during production induces the formation of fine grains [26]. When the mechanical properties of FDM parts produced at 900 °C were compared with the casting method, it is seen that the results are comparable (84% of tensile strength obtained by the casting method) [27]. The reason for the difference can be explained by the existence of printing traces on the surface and the porosity in samples, which were produced by the FDM method.

4. Conclusions

In this study, the feasibility of metallic CuSn10 part production by an FDM machine with optimized printing and sintering conditions was demonstrated. The results show the effectiveness of the FDM method for the fabrication of bronze parts with comparable mechanical properties in a complex shape.

In this paper, the effect of printing parameters on the properties of 3D printed bronze parts by screw-based extrusion technique was investigated as printing parameters, printing speed and layer thickness between 20–60 mm/s, and 0.2–0.6 mm were employed, respectively. SEM images showed that printing speed has a slight effect on the density of printed samples. On the other hand, layer thickness as a second parameter considered in this study influenced the surface and the cross-sectional morphology. Consequently, both printing speed and layer thickness has an impact on the resultant printed sample. The higher the printing speed and layer thickness were used, the more porous the printed parts were obtained. It was found that the layer thickness of 0.2 mm did not show a noticeable printing-induced gap in the printed samples.

When the sintering temperature was taken into consideration, higher temperatures allow higher densities. The sintering temperature of 900 °C gave the highest density and tensile strength values, and a partial slumping in terms of shape stability.
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