

SUPERSOLIDUS LIQUID PHASE SELECTIVE LASER SINTERING OF PREALLOYED BRONZE POWDER

Gopalakrishna B. Prabhu and David L. Bourell,
Center for Materials Science and Engineering,
The University of Texas at Austin, Austin TX 78712.

Abstract

The use of Selective Laser Sintering (SLS) as a method of Solid Freeform Fabrication (SFF) in the direct sintering of metal powders to form the final part has been investigated earlier [1,2,3]. The phenomenon of supersolidus liquid phase sintering (SLPS) is studied using prealloyed bronze powder. The influence of laser parameters, bed temperature and secondary heat treatment on the density and the dimensional stability of the final product are discussed.

Introduction

Supersolidus Liquid Phase Sintering (SLPS) involves heating prealloyed powder to a temperature between the solidus and the liquidus to attain partial melting [4]. Densification occurs by capillary-induced rearrangement and solution reprecipitation in the partially liquid particles and is shown in Figure 1. Liquid forms along grain boundaries and regions of contact. The sintering temperature and alloy composition are thus the most important process variables needing optimization since they dictate the liquid fraction. A typical amount of liquid is 30% which is slightly higher than that observed in liquid phase sintering of mixed powders [5,6,7]. The requisites for a material system include a higher concentration of the alloying element and steep solidus and liquidus lines. A higher concentration in general gives a larger separation between the solidus and the liquidus and so a larger temperature range to investigate. Steep solidus and liquidus lines are desirable because temperature control becomes less critical as the volume fraction of the liquid does not change rapidly. Taking into account these requisites and making use of phase diagrams, the Cu-Sn system with a nominal composition of 89Cu-11Sn was chosen for study. The phase diagram of the Cu-Sn system is shown in Figure 2.

Experimental Procedure

The SLS technique and the workstations used have been described elsewhere [8,9]. In the present study, the prealloyed bronze powder was sintered using either a Nd-YAG or a CO₂ laser. The main laser parameters are listed in Table 1.

Initial experiments were conducted in a room temperature environment to establish optimum laser parameters. Multilayer parts were made with a laser power of 35 watts and a scan speed of 2.5 cm/sec. Attempts to make parts with higher powers were unsuccessful because of excessive curling of the part due to the presence of residual stresses in the part when it cooled down to room temperature. The next set of experiments were run using a high temperature bed in order to alleviate the residual stresses and to

unsuccessful because of excessive curling of the part due to the presence of residual stresses in the part when it cooled down to room temperature. The next set of experiments were run using a high temperature bed in order to alleviate the residual stresses and to reduce the curling seen earlier. Bed temperatures of 300-500 °C and nitrogen at a flow rate of 60 lit/min were used. Parts were made using both the Nd-YAG and the CO₂ lasers. Post processing heat treatments were performed on some of the parts and the effect on part density was studied. Post-processing involved long-term (12-15 hr) heat treatments at different temperatures (700-800°C) and short-term heat treatments(15 min-1hr) at 830-1000°C in either flowing hydrogen or forming gas (96 N₂ - 4 H₂).

Results and Discussion

Selective Laser Sintering:

Prealloyed bronze powder of nominal composition 89Cu-11Sn shown in Figure 3 with a mean particle size of -150# was used to study supersolidus sintering. The best parameters for laser sintering using a room-temperature bed were determined to be a power of 35 watts, a scan speed of 2.5 cm/sec and a layer thickness of 300 μm. The top surface of the part made is shown in Figure 4. Wetting is not very good. Poor interlayer bonding is seen (Figure 5). This is attributed to the use of low power in sintering the bronze powder. Higher powers could not be used because of the excessive curling of the individual layers on sintering. Sintering carried out in a high-temperature environment yielded much better results. Figure 6 shows the top surface of the part made at 400°C using a laser power of 100 watts and a scan speed of 2.5 cm/sec. Good wetting is seen. The end view of the part made with a bed temperature of 350°C is shown in Figure 7. The layers are barely distinguishable indicating that a high-temperature bed and a higher laser power help in improving interlayer bonding. The densities of the parts were determined. The parts made in a room-temperature environment have densities ranging from 4.21 to 4.27 g/cm³, while those made in a high-temperature environment ranged from 4.75 to 5.65 g/cm³. The influence of bed temperature on part density was studied. The results are shown in figure 8. As the bed temperature increased, the density of the part also increased. This can be attributed to less curling observed because of a decrease in residual stress. However 500°C seemed to be an upper limit of temperature for bed heating, as levelling problems were encountered due to caking of the whole powder bed.

Post-Processing Heat Treatments:

From the phase diagram, prealloyed bronze powder of composition 89Cu-11Sn starts melting at 830°C and melts completely at 1025°C. However, because of the non-equilibrium cooling experienced when powders are produced by atomization, these temperatures for the prealloyed bronze powders may not be very accurate. The list of heat treatments with the post-processing density and physical characteristics are shown in Table 2.

Among the long-term heat treatments involving solid-state diffusion, 800°C for 15 hours was the best heat treatment giving the highest density. For the short-

term heat treatments involving liquid phase formation, 850 °C for 15 minutes was the best heat treatment. Beyond 850 °C swelling and loss of shape were observed. Swelling occurred due to the presence of an excess amount of liquid which causes compact slumping, non-uniform densification and pore coalescence [5,10].

Conclusions

The feasibility of Supersolidus Liquid Phase Sintering has been demonstrated using prealloyed bronze powder. The parts that were produced in a high-temperature environment had improved density and surface finish as compared to parts that were made in a room-temperature environment. Post-processing heat treatments to optimize the part density have been determined. The use of a wetting agent to increase the as-laser-sintered part density and the influence of a higher starting density on post-processed part density needs further study.

Acknowledgements

This research was funded by the DARPA-ONR grant # 26-0679-7750 and DTM Corporation.

References

1. J.A.Manriquez-Frayre and D.L.Bourell, "Selective Laser Sintering of Cu-Pb/Sn solder powders", Solid Freeform Fabrication Symposium Proceedings, 1991.
2. Wendy Weiss and D.L.Bourell, "Selective Laser Sintering to produce Ni-Sn intermetallics", Solid Freeform Fabrication Symposium Proceedings, 1991.
3. Paul W. Haase, Masters Thesis, "Selective Laser Sintering of Metal Powders", The University of Texas at Austin, December 1989.
4. R.M.German, "Supersolidus Liquid Phase Sintering Part-1: Process Review", International Journal of Powder Metallurgy, 26[11], p 23-35.
5. R.M.German, "Powder Metallurgy Science", Metal Powder Industries Federation, 1984, p 146.
6. R.M.German, "Phase Diagrams in Liquid Phase Sintering Treatments", JOM, August 1986, pp 26-29.
7. R.M.German, "The Use of Phase Diagrams in Predicting Sintering Behavior", Conference Proceedings, Horizons of Powder Metallurgy Part-2, 1986 International Powder Metallurgy Conference and Exhibition.
8. Harris L. Marcus, Joseph J. Beaman, Joel W. Barlow and David L. Bourell, "Solid Freeform Fabrication: Powder Processing", American Ceramic Society Bulletin, 69[6], 1990 pp 1030-1031.

9. Harris L. Marcus, Joseph J. Beaman, Joel W. Barlow and David L. Bourell, "From Computer to Component in 15 Minutes: The Integrated Manufacture of Three Dimensional Objects", JOM, 42[4], 1990, p 8.
10. R.M.German, "Liquid Phase Sintering", Plenum Press, New York & London, 1985.
11. Binary Alloy Phase Diagrams, Second Edition, ASM International, 1990.

Table 1: Laser Parameters

Laser	Nd-YAG	CO ₂
Wavelength	1.06 micron	10.6 micron
beam diameter	0.5 mm	4.5 mm
Laser power used	20-60 watts	90-110 watts

Table 2: Post-Processing Heat Treatments

Heat treatment	Starting density (ρ/ρ_{th})	Final density (ρ/ρ_{th})	Physical characteristics
700 °C - 15hrs	45.5 %	54.2%	shape retained
750 °C - 15hrs	45.5%	59.1%	shape retained
800 °C - 15hrs	50%	74.2%	shape retained
830 °C - 15min	48.5%	70.5%	shape retained
850 °C - 15min	47%	73.5%	shape retained
875 °C - 15min	46.5%	-	loss of shape
850 °C - 30min	47%	-	loss of shape
850 °C - 15min	59%	78.1%	shape retained
875 °C - 15min	59%	-	loss of shape

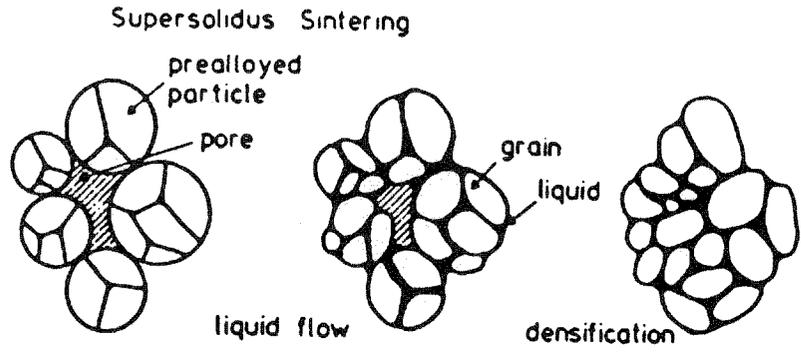


Figure 1: Supersolidus sintering involves the formation of a liquid along the grain boundaries in a prealloyed powder which leads to densification [4].

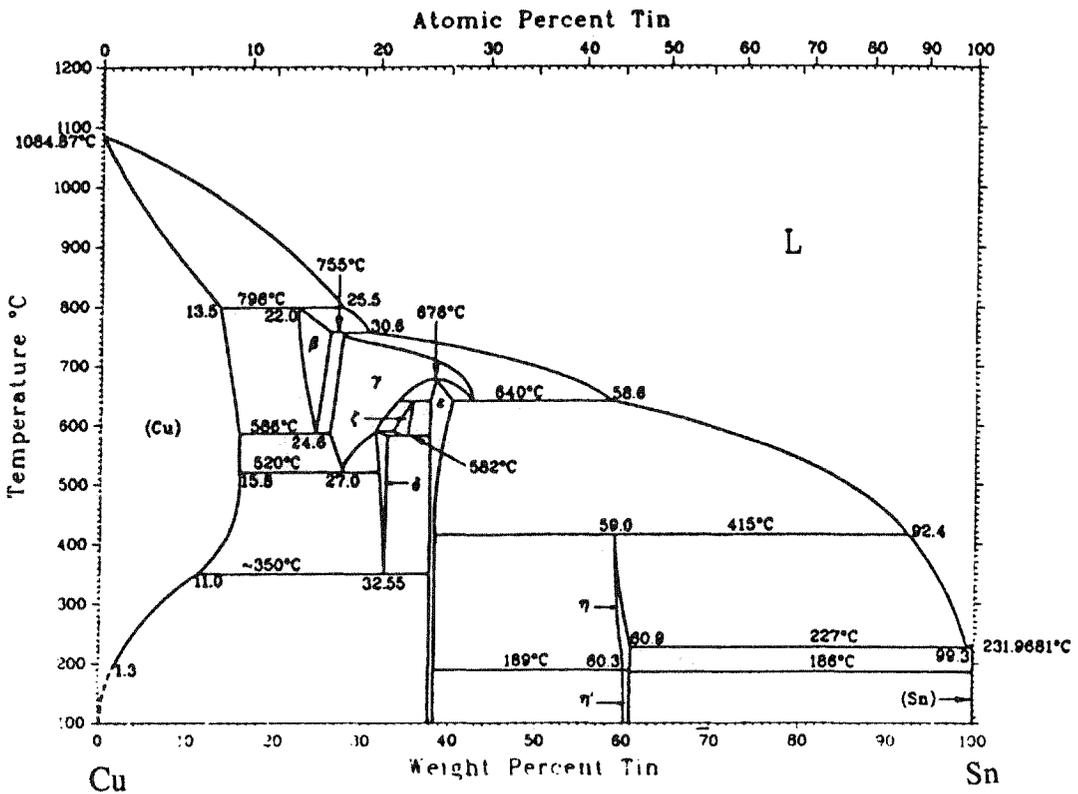


Figure 2: The Cu-Sn phase diagram [11].

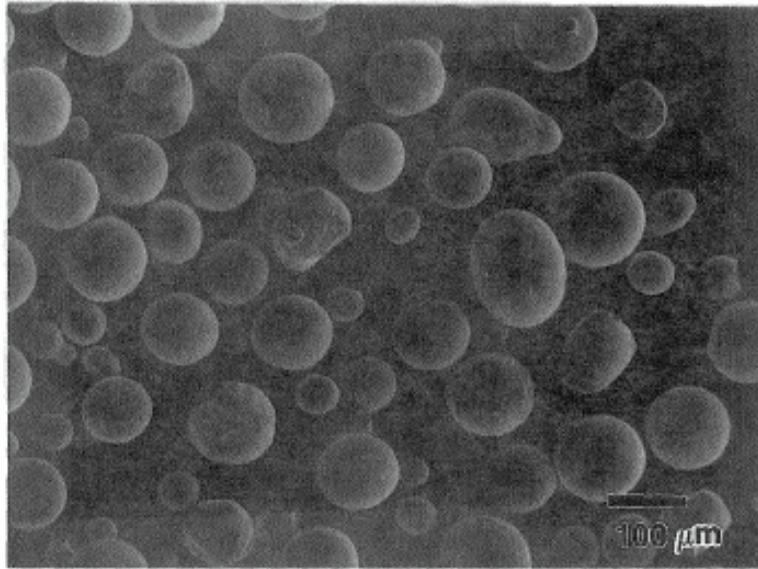


Figure 3: Scanning Electron Micrograph of as received Bronze (89Cu-11Sn) powder.

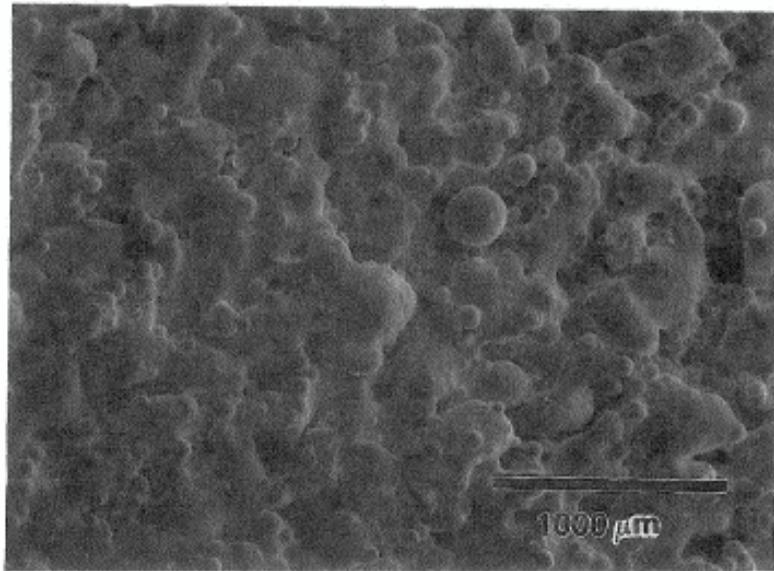


Figure 4: Top surface of the part made in a room-temperature environment shows poor wetting. Laser Power = 35 Watts, Scan Speed = 2.5 cm/sec.

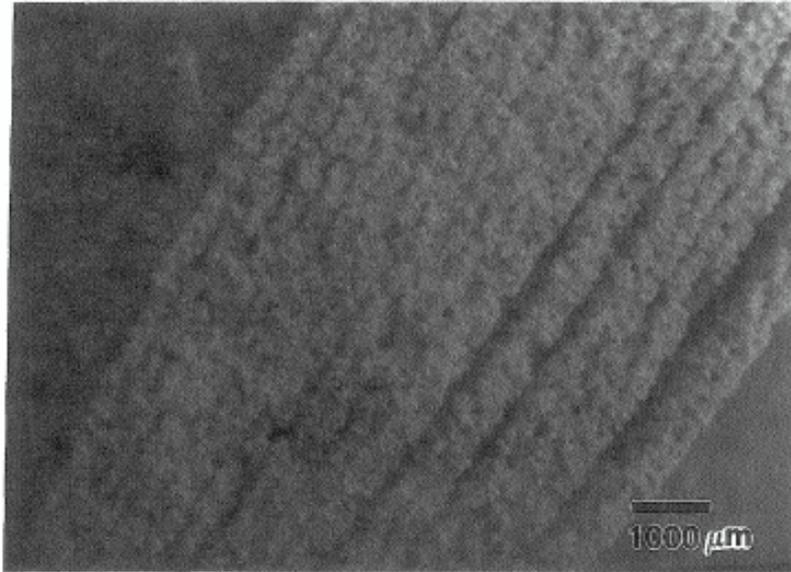


Figure 5: End-view of the part made in the room-temperature environment shows poor interlayer bonding. Laser Power = 35 Watts, Scan Speed = 2.5 cm/sec.

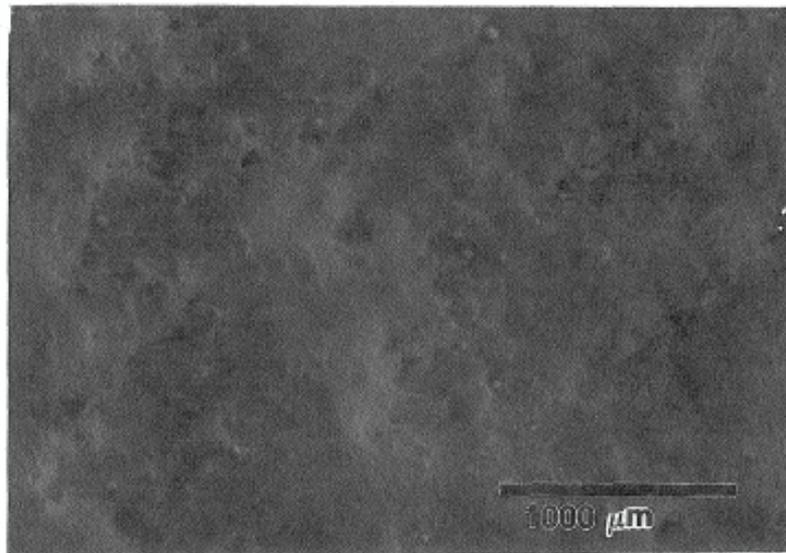


Figure 6: Top surface of the part made with a bed temperature of 350°C shows better wetting than before. Laser Power = 100 Watts, Scan Speed = 2.5 cm/sec.

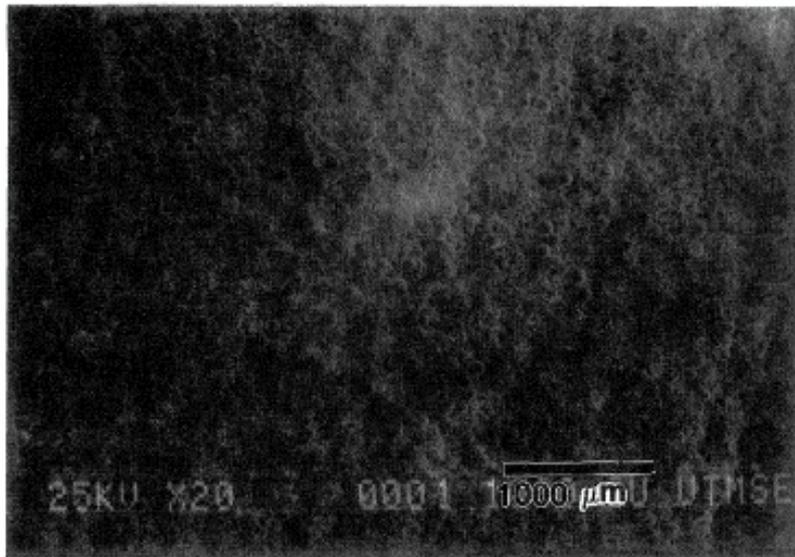


Figure 7: End-view of the part shown in Figure 6. The layers are barely distinguishable. Laser Power = 100 Watts, Scan Speed = 2.5 cm/sec.

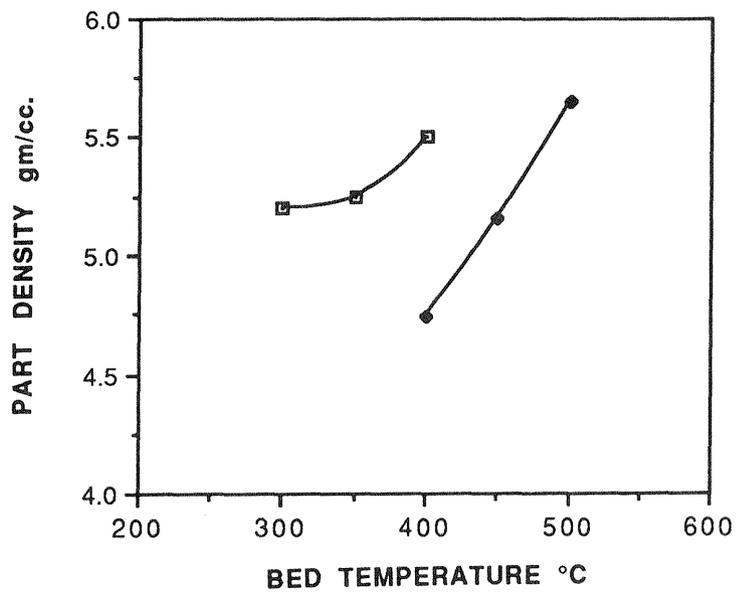


Figure 8: Part density versus bed temperature.