DENSIFICATION OF SELECTIVE LASER SINTERED METAL PARTS BY HOT ISOSTATIC PRESSING

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ABSTRACT

Metal matrix alloy composite parts were made from powders by Selective Laser Sintering (SLS). In this study, partially dense (60%-80%) metal parts made by SLS were densified to full density (>98%) by hot isostatic pressing (HIPping) without any loss of shape. HIPping was done by vacuum sealing SLS samples in glass capsules. HIPping parameters, such as, temperature, pressure, and time, were studied with respect to density, linear shrinkage, and microstructures. Anisotropy in linear shrinkage was correlated to the SLS processing parameters. Densification resulting from HIPping was correlated to microstructures and theoretical HIP densification maps. A detailed analysis of such maps is presented.

INTRODUCTION

The ultimate aim of rapid manufacturing processes is to fabricate three-dimensional fully functional parts directly from metals and ceramic materials, without the use of any intermediate binders or any other materials which may require additional processing steps before or after the rapid prototyping operation. Selective Laser Sintering is one of the few rapid manufacturing processes which possesses the capability of producing such structurally sound parts directly from metals and ceramics [1-4]. Feasibility of producing metal parts directly by SLS has been demonstrated using various metal systems [5-12]. However, SLS parts of metals and ceramics are not fully dense and hence require post processing to obtain near full density. Typically, infiltration and conventional sintering, such as solid state or liquid phase sintering have been employed so far as post-processing steps for SLS parts [1-4]. These post-processing techniques produce near-full density parts, but still leave behind closed pores and result in anisotropic and unpredictable shrinkage during densification. In this study, SLS metal parts have been post processed to full density by **Hot Isostatic Pressing (HIPping)**.

Hot isostatic pressing is a materials processing technique in which high isostatic pressure is applied to a powder part or compact preform at elevated temperatures to produce particle bonding [13-15]. This process usually results in a fully dense body, although partially dense bodies can also be intentionally produced. In hot isostatic pressing, the contact between the particles is increased due to deformation by the external forces and the thermal mobility of the atoms. Therefore, with HIPping it is possible to produce a part with a density approaching the theoretical density and with properties of bulk metal.

Based on certain constitutive equations [16-18], the exact mechanism driving the densification process can easily be determined during HIPping at a given set of process parameters. Using these equations, densification maps are constructed for various material systems. From such maps, shown later for single phase pre-alloyed bronze powders, it can be easily seen that at low temperatures and short time, yielding is the dominant mechanism by which densification takes place. At high temperatures and long times, power-law creep is the dominant mechanism by which densification takes place. Although such maps exist for various single phase metal and ceramic systems, densification maps for multi-phase systems have rarely been studied.

Also, such maps do not take into account the effect of phase transformations on the densification process.

As the pressing temperatures increases, the amount of pressure necessary for compaction decreases. Typically, HIPping temperatures range from 450°C for aluminum alloys to 1700°C for tungsten. Pressures are usually applied using argon gas as the pressure transmitting medium and HIPping pressures range from 20MPa to 300MPa. The duration of the pressing cycle is also important, since at high temperatures lengthy pressing leads to creep. The longer the pressing is done, the greater the density and the better the properties of the pressing at a given temperature and pressure. Lengthy pressing also brings about more complete reduction of oxides, softening and recrystallization, which lead to an improvement in the plastic properties of the part. In addition to temperature, pressure, and time, particle characteristics such as particle size also play an important role in densification during HIPping.

In this study, HIPping was explored as a post processing route for SLS metal parts. SLS bronze-nickel parts, previously used to study the structural integrity and effect of post processing by conventional liquid phase sintering [10-12], were used for HIPping. SLS parts typically have surface connected and interconnected pores. Therefore, such parts require the surface to be sealed or the part to be encapsulated such that the pressure transmitting medium, argon gas, does not flow into the pores in the part. Presence of high pressure gas in the interconnected pores will prevent them from "closing" during the HIP processing. To seal such parts, the parts are encapsulated in an envelope such that the "empty space" in the pores is "squeezed" out during HIPping and the pores close to form a fully dense part.

The basic requirements for the encapsulation material are that it should be relatively strong, gas tight, inert and plastic under the applied temperature and pressure conditions, compatible with the material to be densified so as to minimize diffusion reactions and easily removable. Several materials and techniques are used for encapsulation of powders and compact preforms for HIP processing [13-15].

In this study, the SLS bronze-nickel parts for HIPping were contained in a vacuum-sealed glass capsule. Glass encapsulation has so far been used only on experimental basis. Commercially, sheet metal containers or ceramic containers are commonly used. Glass encapsulation was used instead of sheet metal containers or ceramic containers because of the ease with which glass can be shaped to take the form of SLS parts irrespective of the complex nature of the part. Unlike sheet metal and ceramics, glass becomes vitreous at high temperatures and hence can flow easily and conform to the shape of the preform or in this study, the SLS part.

EXPERIMENTAL PROCEDURE

GLASS ENCAPSULATION OF SLS BRONZE-NICKEL PARTS

SLS bronze-nickel samples of ~70% density were cut from the shoulder section of broken tension test coupons, previously tension tested [10-12], into small sections of 1.5cm x 1.5cm x 0.5cm dimensions. The samples were degassed by annealing them at 450°C in H_2 for 2 hours. Commercial borosilicate glass tubes of O.D. 19 mm and I.D. 15.5 mm were used as the container material. The tube and the sample were thoroughly dried in an oven at 150°C. One end of the glass tube was sealed by a conventional glass blowing method using an oxy-acetylene torch. The SLS sample was then placed in the tube and a small constriction was made in the tube, ~2-4 cm above the sample, by the usual glass blowing technique using a torch. Figures 1A and 1B show schematically the stages involved in vacuum sealed glass encapsulation of the SLS parts. The constriction was as small as possible.

The glass tube with the sample and the constriction was then evacuated using a diffusion pump which was backed up by a mechanical pump, Figure 1B. A liquid nitrogen cold-trap was used in the evacuation to ensure efficient evacuation and prevent any contamination of the diffusion pump. The glass tube with the sample was evacuated to a vacuum of ~10^{-5} torr. Once the desired vacuum level was reached, the constricted part of the tube was sealed completely, using the torch, and removed from the remaining part of the tube.

HOT ISOSTATIC PRESSING OF SLS BRONZE-NICKEL PARTS

HIPping of the vacuum-sealed glass encapsulated SLS bronze-nickel samples was done at three different temperatures of 750°C, 825°C, and 900°C with a pressure of 124MPa (18ksi) for 1, 2 or 3 hours. Similarly, HIPping of SLS bronze-nickel samples of starting density 70% was done with three different pressures of 69MPa (10ksi), 124MPa (18ksi), and 180MPa (26ksi) at 825°C for 1, 2 or 3 hours. Sample dimensions and weight were measured before and after HIPping to determine densification.

RESULTS AND DISCUSSION

SLS bronze-nickel samples exhibited densification at the various HIPping temperatures, pressures and times used in this study. The extent of densification observed varied with these variables. As shown in Figures 2, density increased with increasing temperatures, pressures and times and approached theoretical density at high values of these variables.

Densification of SLS bronze-nickel parts during HIPping was accompanied with phase transformation between bronze and nickel phases, Figures 3 and 4. Bronze, predominantly copper (90 Wt.%), and nickel homogenize at high temperatures by interdiffusion between bronze and nickel to form a homogeneous solid solution. The degree of homogenization depends on temperature and time of sintering or HIPping. As evidenced by (311) peak broadening of copper and nickel, shown in Figures 3, at low temperatures and short times only partial homogenization occurs and complete homogenization occurs at high temperatures and long times. As a result of chemical homogenization, the bronze phase disappears completely leaving behind pores in its place and an expanded solid solution of bronze in nickel. This happens due to a faster diffusion rate of copper into nickel than that of nickel into copper, which results in the Kirkendall effect. Therefore, the pores created at this stage are referred to as Kirkendall porosity. Due to the generation of Kirkendall pores and expansion of nickel phase, the samples may undergo growth or swelling instead of shrinkage, as has been observed in SLS bronze-nickel parts during conventional sintering below 1000°C [10-12]. Although, as shown in Figures 3 and 4, chemical homogenozation between bronze and nickel was observed in the SLS bronze-nickel parts during HIPping also, no overall expansion or growth of the SLS parts was observed during HIPping. Parts processed at lower temperatures and pressures, which exhibit low density, show partial homogenization with Kirkendall pores present at the boundaries of homogenized and non-homogenized phases, Figure 4b. However, parts processed at high temperatures and pressures, which exhibit nearly full density, also show partial homogenization but absence of any Kirkendall pores, Figure 4c.

As a result of chemical homogenization between bronze and nickel, and generation of Kirkendall pores, the densification of the bronze-nickel samples is considerably slower than predicted by theoretical densification maps for single phase pre-alloyed bronze. Figures 5 show the densification of bronze-nickel samples observed in this study relative to theoretical densification curve for pure bronze of 50μm particle size. The theoretical HIP densification map for bronze was

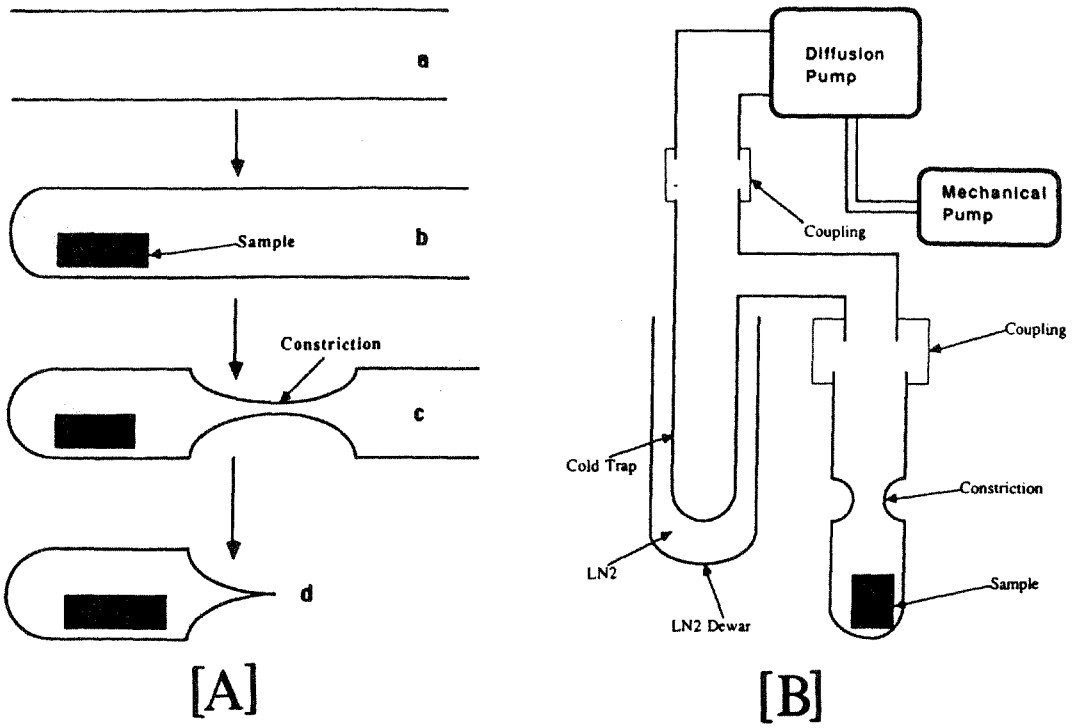


Figure 1: [A] A schematic representation of the glass encapsulation used in this study (a) glass tube with both ends open, (b) glass tube with one end sealed and the sample in the tube, (c) glass tube with the sample in it and a constriction, and (d) vacuum sealed glass capsule with the sample in it, and [B] A schematic representation of evacuation procedure for glass encapsulation of SLS part.

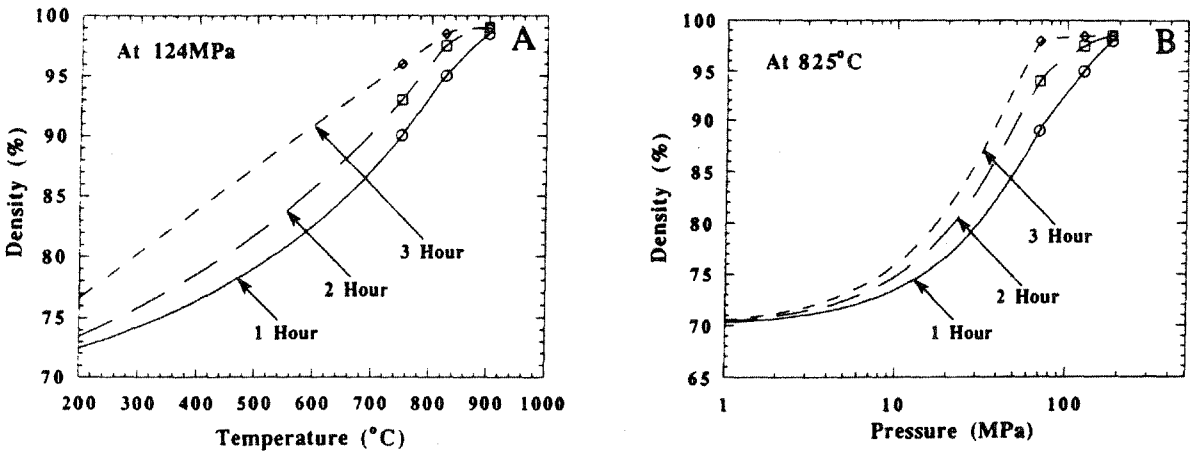
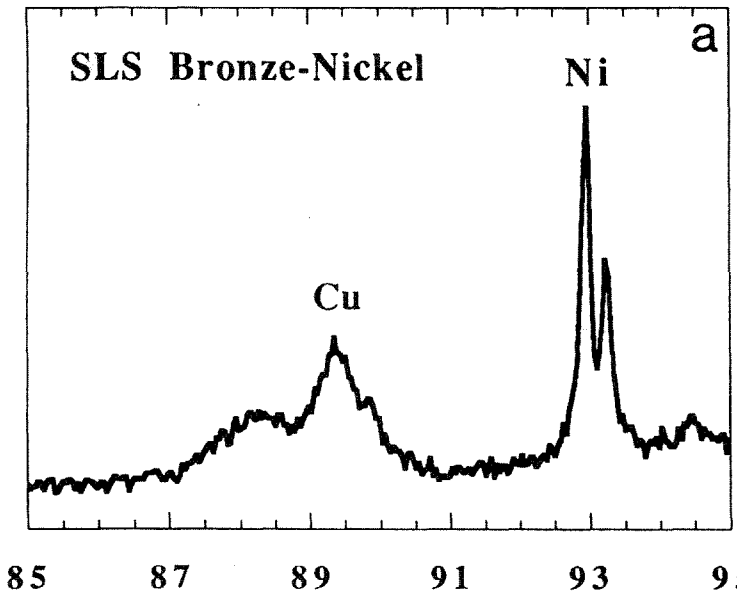
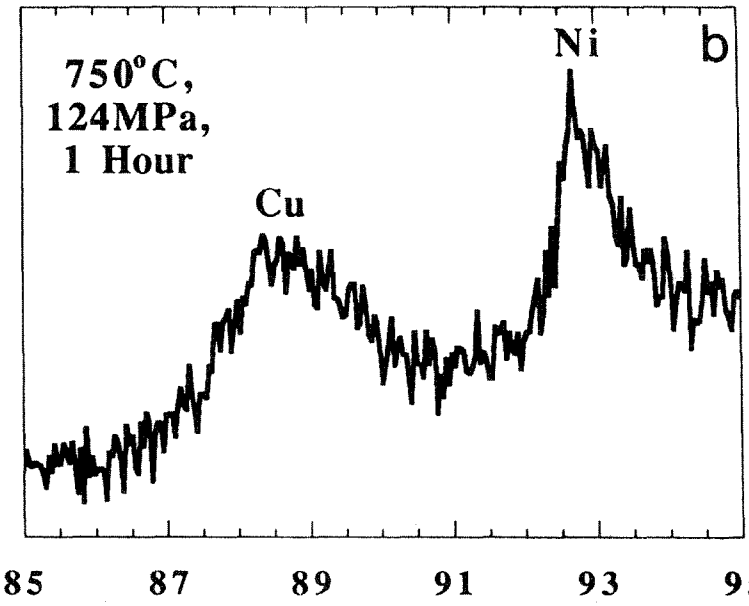


Figure 2: Density of SLS bronze-nickel parts as a function of (A) HIPping temperature and time at a constant pressure of 124MPa, (B) HIPping pressure and time at a constant temperature of 825°C.





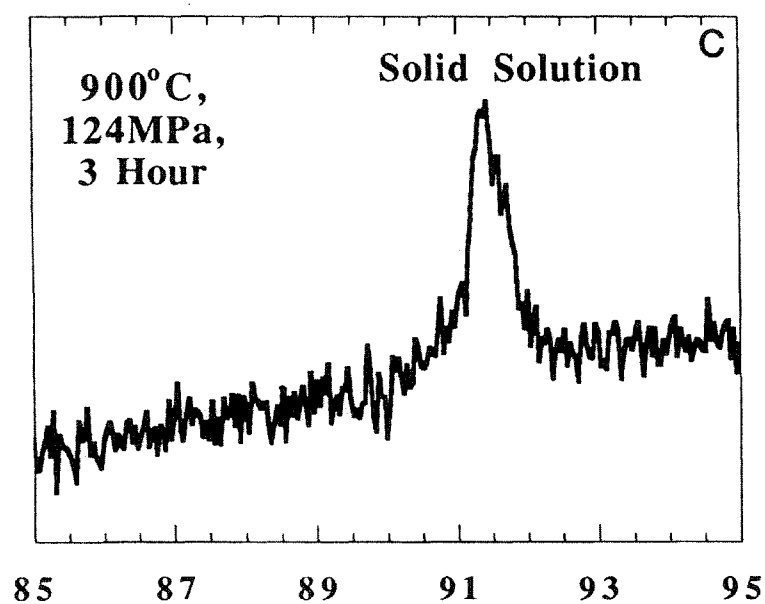


Figure 3: XRD patterns of (311) peaks of Bronze and Nickel showing peak broadening due to homogenization (a) SLS Bronze-Nickel part showing Cu and Ni as separate phases, (b) SLS Bronze-Nickel sample HIPped at 750°C for 1hour at 124MPa, showing partial homogenization, and (c) SLS Bronze-Nickel sample HIPped at 900°C, for 1hour at 124MPa, showing complete homogenization.

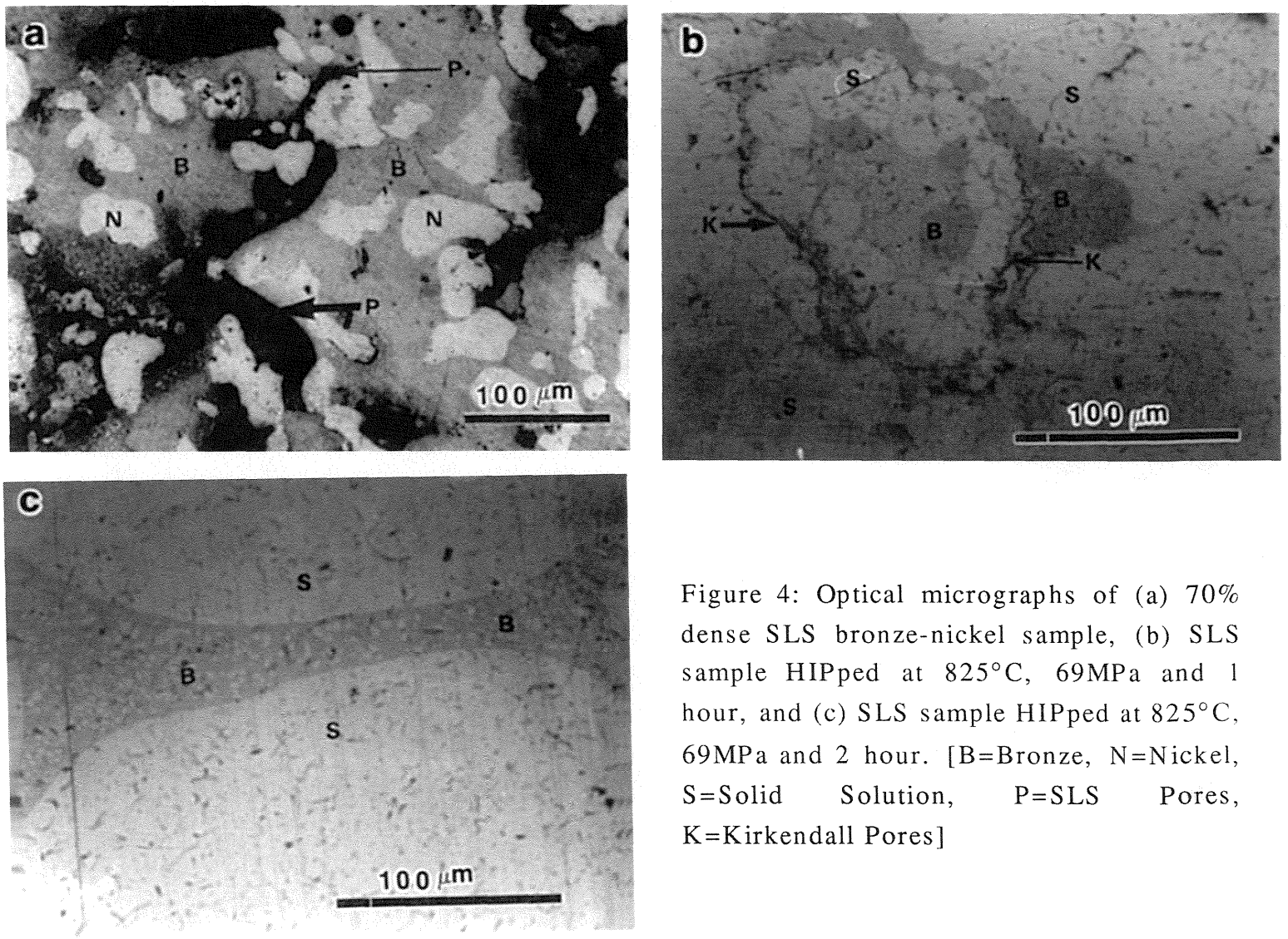


Figure 4: Optical micrographs of (a) 70% dense SLS bronze-nickel sample, (b) SLS sample HIPped at 825°C, 69MPa and 1 hour, and (c) SLS sample HIPped at 825°C, 69MPa and 2 hour. [B=Bronze, N=Nickel, S=Solid Solution, P=SLS Pores, K=Kirkendall Pores]

validated experimentally by HIPping of a single phase pre-alloyed bronze sample at 750°C at 69MPa for 1 hour which yielded nearly full density in accordance with the map. However, the experimental data for SLS bronze-nickel parts are well to the right of the theoretical curves indicating a much slower densification in the bronze-nickel samples. Therefore, densification is slowed down during HIPping of mixed phases, such as bronze-nickel, which form a solid solution at HIPping temperatures resulting in Kirkendall pores which oppose the on-going densification. Theoretical HIP maps do not account for such phase transformations and their effect on densification during HIPping. Low HIPping temperatures and pressures and short time of HIPping cause nearly complete removal of pores present due to SLS but do not result in removal of Kirkendall pores created due to homogenization during HIPping. Therefore, such processing conditions which do not remove the Kirkendall pores completely result in less than full density. However, high temperatures and pressures result in removal of both the pores due to SLS as well as the Kirkendall pores created due to homogenization during HIPping.

Net volume changes (shrinkage) of SLS parts during HIPping was in accordance with the observed density changes. However, the linear dimensional changes, $\Delta L/L_0$, were not the same in all three directions, as observed earlier in conventional liquid phase sintering of such parts. Figure 6A shows the shrinkage in parts HIPped at 825°C and 69MPa for varying times. The linear dimensional shrinkage in these parts was anisotropic with least shrinkage in the scan direction and maximum shrinkage in the thickness or build-up direction. Similar shrinkages have been observed in SLS parts when subjected to conventional liquid phase sintering [10-12]. In either case, maximum shrinkage in thickness direction is observed due to lowest density along that direction due to poor sintering between layers during SLS and minimum shrinkage in the scan direction due higher degree of sintering during SLS along the scan direction. However, the degree of anisotropic shrinkage observed during HIPping is lower than that observed during liquid phase sintering. Comparision of Figures 6A and 6B shows that the difference in shrinkage between thickness and scan direction is lower in HIPped parts. HIPping is done at temperatures well below the liquid phase sintering temperatures, therefore there is little or no liquid phase formed during HIPping. Also, HIPping is done under isostatic pressures, therefore there is minimal effect of gravity aiding the densification or flow of liquid in the direction of gravity which coincides with the thickness or build-up direction during both the HIPping as well as liquid phase sintering processes. Therefore, the anisotropy observed in HIPping of SLS parts represents a more precise picture of density gradient present in parts due to SLS.

CONCLUSIONS

A framework for encapsulation of SLS parts for HIPping has been established using glass containers. Glass encapsulation is an easy, flexible, and economical method for encapsulating complex shapes. Densification studies of SLS bronze-nickel parts by HIPping resulted in higher density parts with increasing temperatures, pressures and times. However, the densification of SLS bronze-nickel parts was slower than that predicted by theoretical HIP densification maps due to generation of Kirkendall pores arising from the chemical homogenization between bronze and nickel phases during HIPping. The anisotropy in linear dimensional changes during HIPping is not influenced by liquid phase or effect of gravity, thus giving a precise picture of anisotropy present in parts due to SLS.

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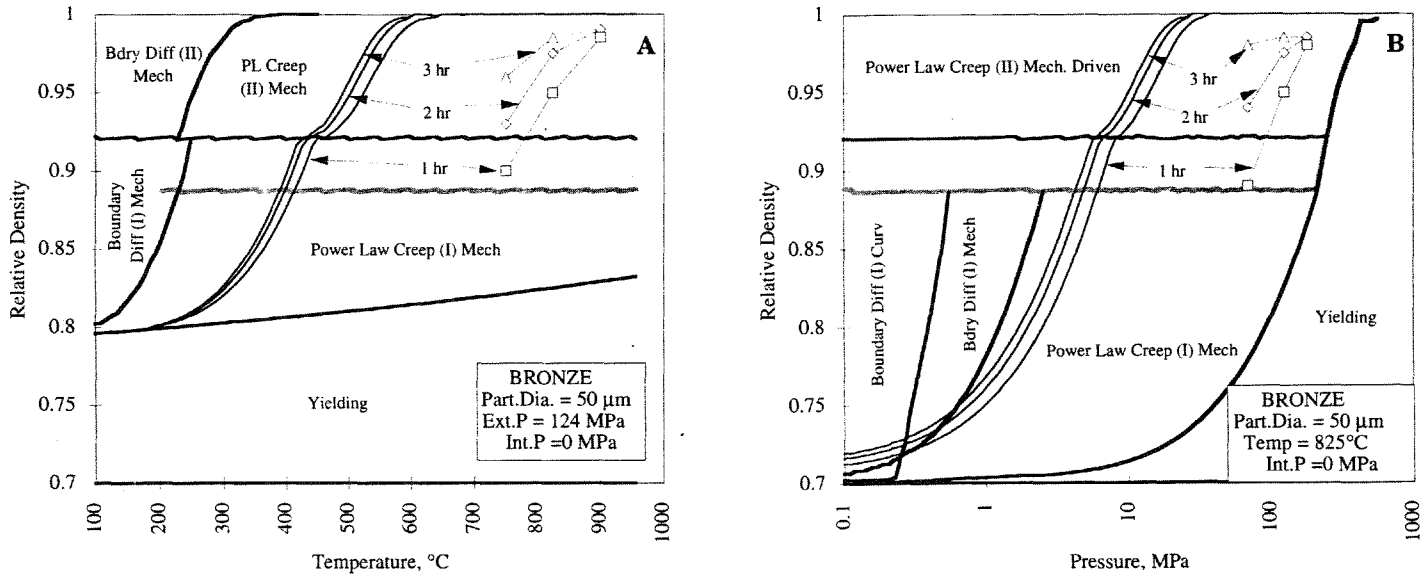

Figure 5: Comparision of densification curves of SLS bronze-nickel samples (symbols) with the theoretical HIP densification curves for single phase pre-alloyed bronze, as a function of (A) HIPping temperature and time, and (B) HIPping pressure and time.

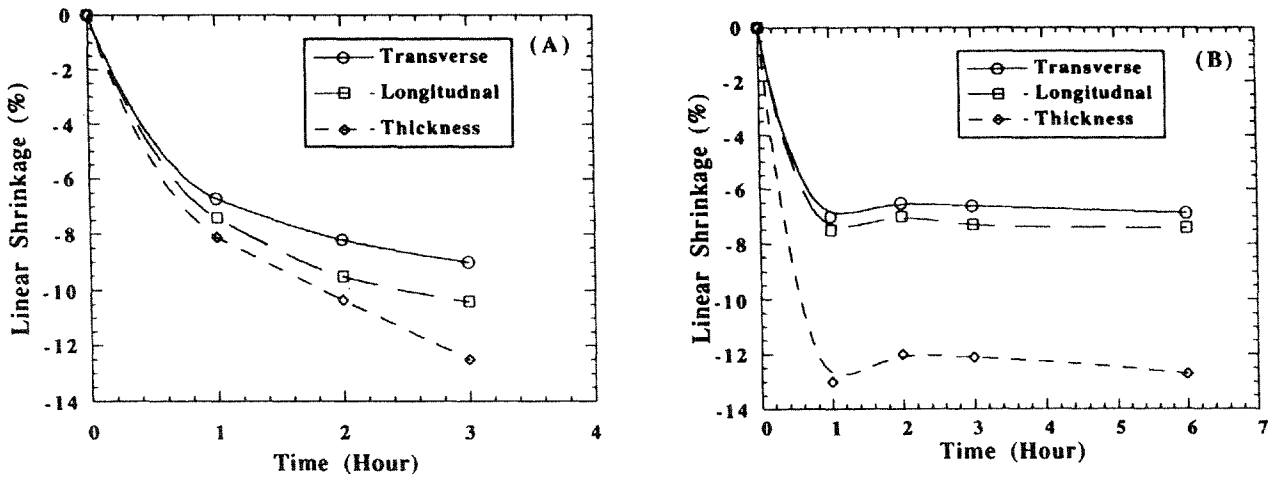

Figure 6: Anisotropic linear dimensional shrinkages observed in SLS bronze-nickel parts subjected to (A) HIPping at 825°C and 69MPa, and (B) Conventional liquid phase sintering at 1030°C.

REFERENCES

1. 1990 Solid Freeform Fabrication Symposium Proc., Edited by J.J.Beaman, H.L.Marcus, D.L.Bourell, and J.W.Barlow, Aug. 6-8, 1990, The University of Texas at Austin, Austin, Texas.
2. 1991 Solid Freeform Fabrication Symposium Proc., Edited by H.L.Marcus, J.J.Beaman, J.W.Barlow, D.L.Bourell, and R.H.Crawford, Aug. 12-14, 1991, The University of Texas at Austin, Austin, Texas.
3. 1992 Solid Freeform Fabrication Symposium Proc., Edited by H.L.Marcus, J.J.Beaman, J.W.Barlow, D.L.Bourell, and R.H.Crawford, Aug. 3-5, 1992, The University of Texas at Austin, Austin, Texas.
4. 1993 Solid Freeform Fabrication Symposium Proc., Edited by H.L.Marcus, J.J.Beaman, J.W.Barlow, D.L.Bourell, and R.H.Crawford, Aug. 9-11, 1993, The University of Texas at Austin, Austin, Texas.
5. J.A.Manriquez-Frayre and D.L.Bourell, "Selective Laser Sintering of Binary Metallic Powder," ibid., Reference #1, pp. 99-106.
6. J.A.Manriquez-Frayre and D.L.Bourell, "Selective Laser Sintering of Cu-Pb/Sn Solder Powders," ibid., Reference #2, pp. 236-244.
7. W.Weiss and D.L.Bourell, "Selective Laser Sintering to Produce Ni-Sn Intermetallic Parts," ibid., Reference #2, pp. 251-258.
8. G.Zong, Y.Wu, N.Tran. I.Lee, D.L.Bourell, J.J.Beaman, and H.L.Marcus, "Direct Selective Laser Sintering of High Temperature Materials," ibid., Reference # 3, pp.72-85.
9. G.Prabhu and D.L.Bourell, "Supersolidus Liquid Phase Sintering of Prealloyed Bronze Powder," ibid., Reference #4, pp.317-324.
10. M.K.Agarwala, D.L.Bourell, B.Wu and J.J.Beaman, "An Evaluation of the Mechanical Behavior of Bronze-Nickel Composites Produced by Selective Laser Sintering," ibid., Reference # 4, pp.193-202.
11. M.K.Agarwala, D.L.Bourell, B.Wu and J.J.Beaman, "Structurally Sound Metal Parts by Selective Laser Sintering," The EPD Congress 1994, Proc. of a symposium sponsored by the Extraction and Processing Division, At the 123rd TMS Annual Meeting, Mar.1994, San Francisco, CA, pp. 833-851.
12. M.K.Agarwala, D.L.Bourell, J.J.Beaman, B.Wu, H.L.Marcus, R.H.Crawford, and J.W.Barlow, "Selective Laser Sintering of a Bronze-Nickel Powder Mixture," Proc. of the IMS Int. Conf. on Rapid Product Development, Feb. 1994, Stuttgart, Germany.
13. P.E.Rice and S.P.Kohler, "Hot Isostatic Pressing of Metal Powders," Metals Handbook, pp. 419-443, Vol.7, 9th Ed., Am. Soc. of Metals, Metals Park, Ohio.
14. H.V.Atkinson, B.A.Rickinson, "Hot Isostatic Pressing," The Adam Hilger Series on New Manufacturing Processes and Materials, Adam Hilger, IOP Publishing Ltd.,New York, NY, 1991.
15. "Isostatic Pressing: Technology and Applications," Edited by M.Koizumi and M.Nishihara, Elsevier Applied Science, London, 1991.
16. M.F.Ashby, "A First Report on Sintering Diagrams," Acta Metall., 22, pp. 275-89, 1974.
17. F.B.Swinkels and M.F.Ashby, "A Second Report on Sintering Diagrams," Acta Metall., 29, pp. 259-81, 1981.
18. A.S.Helle, K.E.Easterling, and M.F.Ashby, "Hot-Isostatic Pressing Diagrams: New Developments," Acta Metall., 33, pp. 2163-74, 1985.