SELECTIVE LASER SINTERING TO PRODUCE NI-SN INTERMETALLICS

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ABSTRACT

A model system has been employed to investigate the use of selective laser sintering (SLS) with a post-processing step to produce a compound having different properties than the precursor powders. The powder mixture examined consisted of 95% (59% Ni and 41% Sn) plus 5% $ZnCl_2$. This weight fraction of nickel and tin produces the intermetallic compound Ni_3Sn upon equilibrium annealing. $ZnCl_2$ was used as a wetting agent. Parts were fabricated using SLS and were then post-process annealed to create the intermetallic. Metallographic techniques and x-ray powder diffraction were used to characterize the parts before and after annealing.

INTRODUCTION

Selective Laser Sintering is a process in which a three- dimensional object is constructed directly from a computer aided design (CAD) database without part specific tooling or human intervention.[1] The process involves using a laser to selectively sinter thin layers of powdered material. The process is described in detail in references 2-3.

Selective laser sintering, in conjunction with a post-process heat treatment, is being utilized to explore the possibility of directly producing parts having different properties than the precursor powders. Two or more elemental powders are selectively laser sintered and then given a post-process anneal. During the heat treatment a transformation occurs and a new phase is formed, having new properties. One possible application for this use of selective laser sintering is the production of parts consisting of intermetallic compounds. The precursor powders can be blended so that, upon annealing, the entire part develops into the intermetallic, or the part consists of two or more phases, one of which is the intermetallic. Because many intermetallics are difficult to machine and form[4] selective laser sintering could prove to be a valuable method of directly producing parts from these materials.

EXPERIMENTAL

A nickel-tin system was used to explore selective laser sintering to produce intermetallics. This system has three prospective intermetallic compositions (Fig. 1)[5]. The composition of 41% Sn and 59% Ni (by weight) was chosen because it had an appropriate amount of low melting temperature material. Ni_3Sn is the compound formed from the above composition. Elemental 99.9 Ni(-100 mesh) and 99.5% Sn (-325 mesh) powders were mixed and $ZnCl_2$ was added as a wetting agent. Crystals of $ZnCl_2$ were dissolved in ethyl alcohol, and the solution was added to the powder blend. The mixture was stirred and then dried in a vacuum oven at approximately

90°C. The dried mixture was sieved to yield $ZnCl_2$ coated nickel and tin powders. The final powder mixture used was 95% (41% Sn + 59% Ni) + 5% $ZnCl_2$.

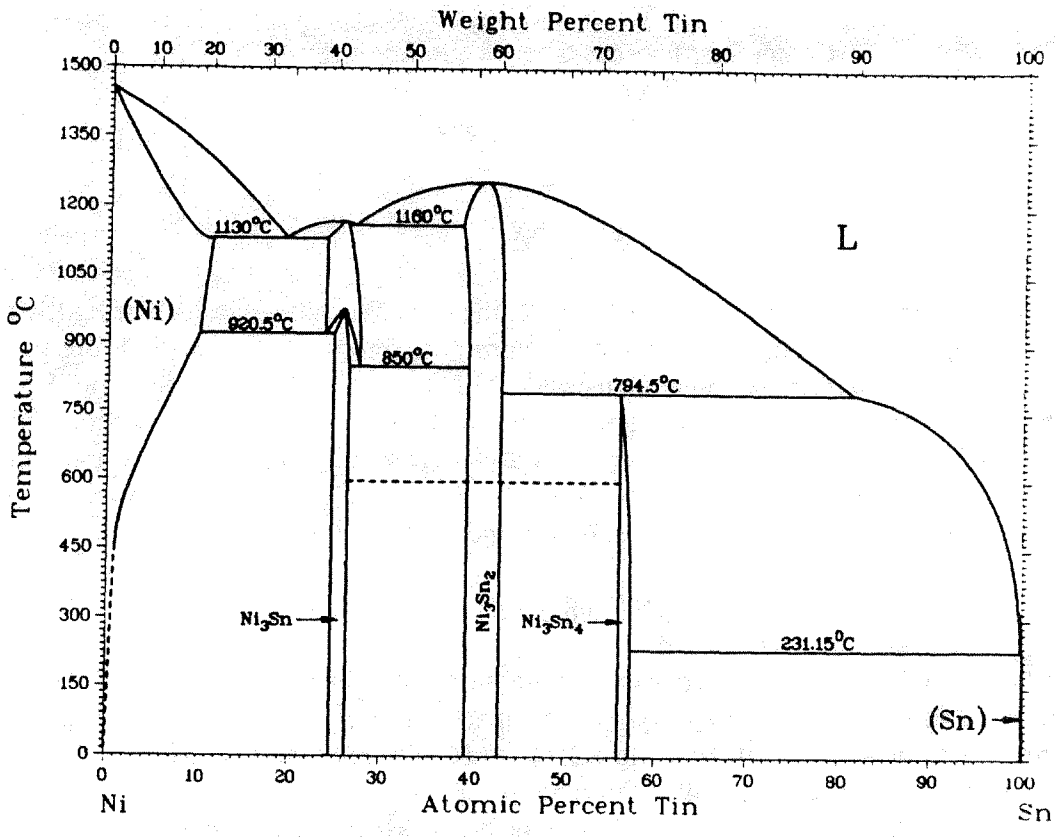

Figure 1. The Ni - Sn Phase Diagram. (From Binary Alloy Phase Diagrams).

The powder mixture was selectively laser sintered using a computer controlled, Q-switched, TEMoo Nd:YAG laser. The laser process conditions were as follows:

$\lambda = 1.06\ \mu m$
Beam diameter = 0.5 mm
Maximum power = 100 W
Incident power = 17 - 25 W
Scan speeds = 2 cm/s - 6 cm/s

Before the SLS processing began, the powder bed was heated to 150°C using a resistive ring heater mounted in the work station. The laser sintering was done in a nitrogen atmosphere to prevent oxidation and for safety reasons. Some parts were then post- process annealed in a hydrogen atmosphere at 800°C for approximately 24 hours. Scanning electron microscopy (SEM) was used to determine the microstructure of the sintered part and to ascertain the degree to which the tin wet the nickel. X-ray powder diffraction was used to determine the phases present,

including the intermetallic compounds, and energy dispersive spectroscopy was used to examine the effects of laser sintering and post processing on the $ZnCl_2$. All of these techniques were utilized before and after the post-process heat treatment.

RESULTS and DISCUSSION

Single Layers

Single layers were produced initially to determine system viability. In other words, single layers were used to examine how and to what degree the tin wet the nickel and how the powder system behaved under laser processing. This step was necessary since wetting problems were found in earlier work with metal powders[6]. SEM was used to examine the samples. The use of $ZnCl_2$ flux resulted in good wetting, Figure 2, evidenced by the flow of molten tin around the nickel particles. The nickel particles are engulfed in the tin, rather than the tin agglomerating and not surrounding the nickel.

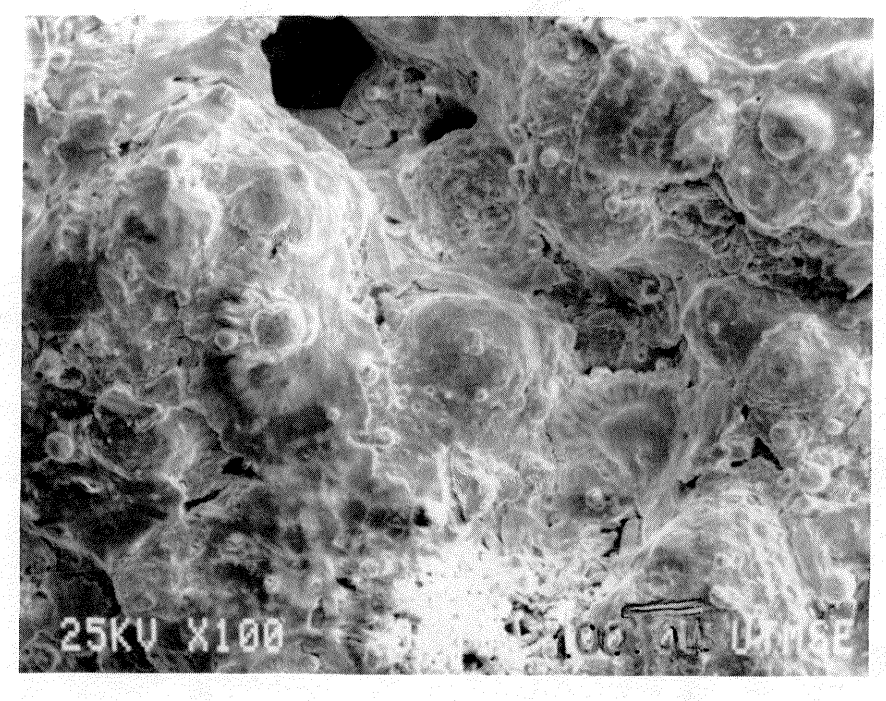

Figure 2. Micrograph showing the surface of an unannealed single layer sample. The laser power was 17 W and the scan speed was 3.41 cm/s. The magnification is 100X.

Single layer tests were also done to readily assess various laser parameters (Fig. 3). There was not much contrast in the resulting single layer samples based on the aforementioned range of laser parameters. However, based on SEM observations, a lower laser power (17W) and a slower scan speed (3.41 cm/s) seem to produce the best wetting and the least porous parts. None of the single layers were post-process annealed.

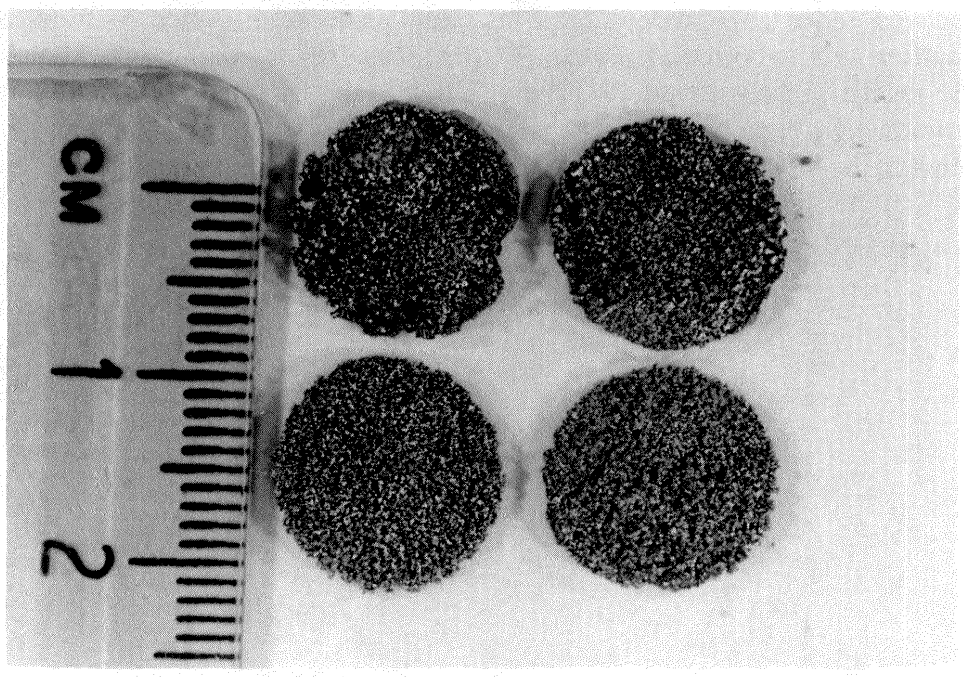

Figure 3. Single layer samples showing the influence of laser parameters. The top samples had a laser power of 25 W; the bottom samples had a laser power of 17 W. The left side had a scan speed of 4.55 cm/s; the right side had a scan speed of 3.41 cm/s.

Powder Compacts

Small cylindrical powder compacts were made using standard powder processing approaches to evaluate the effect of post processing. Based on effective diffusion distance calculations, the highest temperature feasible would be the best choice. Compact samples were fired at 900°C, 800°C, and 700°C for 24 hours. Both of the lower temperatures appeared to be acceptable. At a temperature of 900°C the sample did not retain its dimensionality. X-ray powder diffraction on all of the samples showed that some intermetallic formed.

Multiple Layers

Multiple layer samples of 20 to 40 layers were made using a laser power of 17 W and a scan speed of 3.41 cm/s. The samples were analyzed both before and after post processing. Post processing consisted of a 24 hour anneal at 800°C in a hydrogen atmosphere. Figure 4 shows a SEM micrograph of the surface of an unannealed sample. The tin wet the nickel particles very well, and the sample is sturdy although porous. The density was measured to be approximately 74% of theoretical density. EDS analysis indicated that some of the $ZnCl_2$ flux was still present after laser sintering, and x-ray powder diffraction showed that no intermetallic formed during the laser processing. The phases present were Ni, Sn, and a small amount of Ni_2O_3.

Figure 5 shows a SEM micrograph of the surface of a post- processed sample (800°C). This sample appears much more porous than the unannealed sample. It is also very sturdy but brittle. There is no longer an obvious distinction between the melted tin and the nickel particles. X-ray powder diffraction established that intermetallic compounds did form. However, the sample did not convert entirely to Ni_3Sn. While Ni_3Sn was the most predominate compound, Ni_3Sn_2 and beta Sn were also present. EDS showed that no $ZnCl_2$ was left after the post processing.

Figure 4. A micrograph showing the surface of an unannealed, multiple layer sample. The laser power was 17 W and the scan speed was 3.41 cm/s. The magnification is 100X.

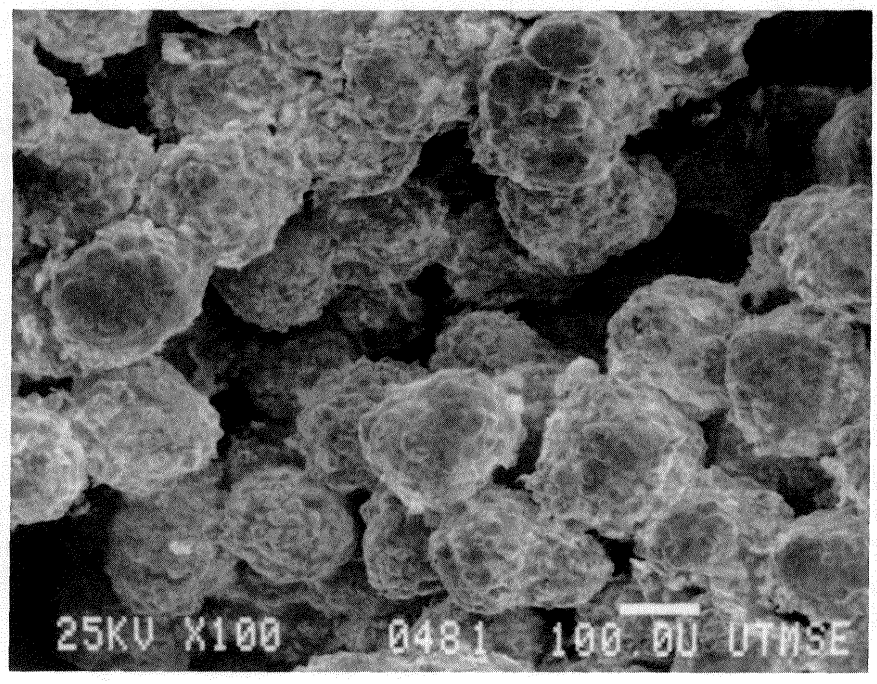

Figure 5. A micrograph of an annealed, multiple layer sample. The sample was annealed in H_2 at 800°C for 24 hrs. The laser power was 17W and the scan speed was 3.41 cm/s. The magnification is 100X.

Figures 6 and 7 show cross sections of the unannealed and post-processed parts, respectively. The layers are indistinguishable from one another which implies good interlayer bonding. It is evident that the post processing improved the interlayer bonds. Upon visual examination, no change in dimension or shape were observed after annealing.

Selective laser sintering appears feasible for producing intermetallic parts. The nickel-tin-$ZnCl_2$ system performed well under the laser and the green parts held together nicely. Several problems did surface, however. The biggest problem was the low density, which resulted in parts that were not of structural quality. The need to control which intermetallic compound forms is also a potential obstacle for this process. Longer annealing times and\or step-wise annealing will influence the resulting phases. Another consideration, when working with systems that form intermetallics, is the possibility of reactive sintering. This could not only make it very difficult to control the shape of a part being laser processed, it also could produce a highly undesirable uncontrolled transformation of the entire powder bed. However, if controlled properly, the reactive nature of intermetallic systems could also be beneficial, leading to intermetallic production during the laser sintering.

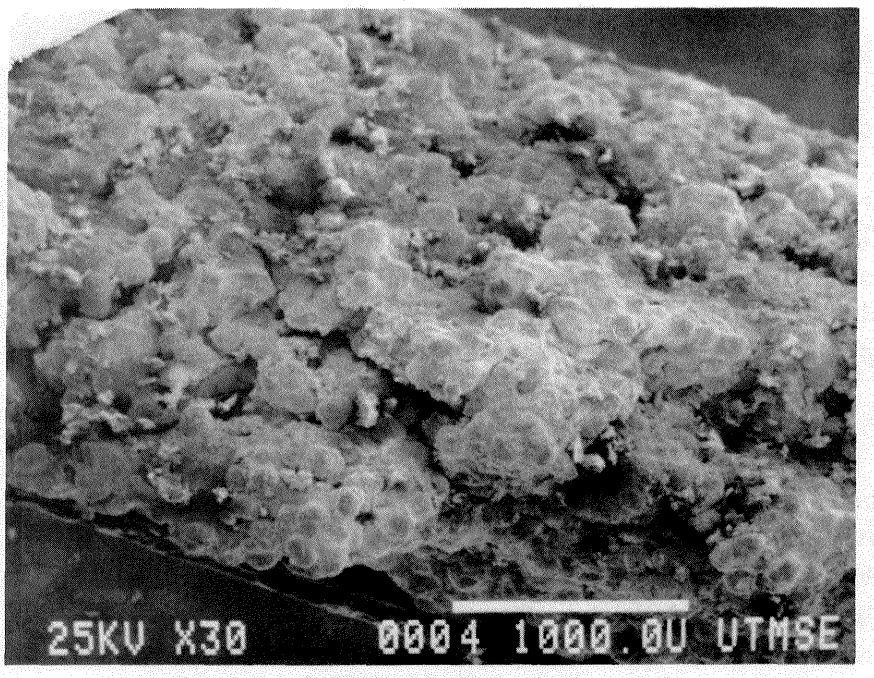

Figure 6. A micrograph showing a cross section of an unannealed, multiple layer sample. The layers are barely distinguishable. The laser power was 17 W and the scan speed was 3.41 cm/s. The magnification is 30X.

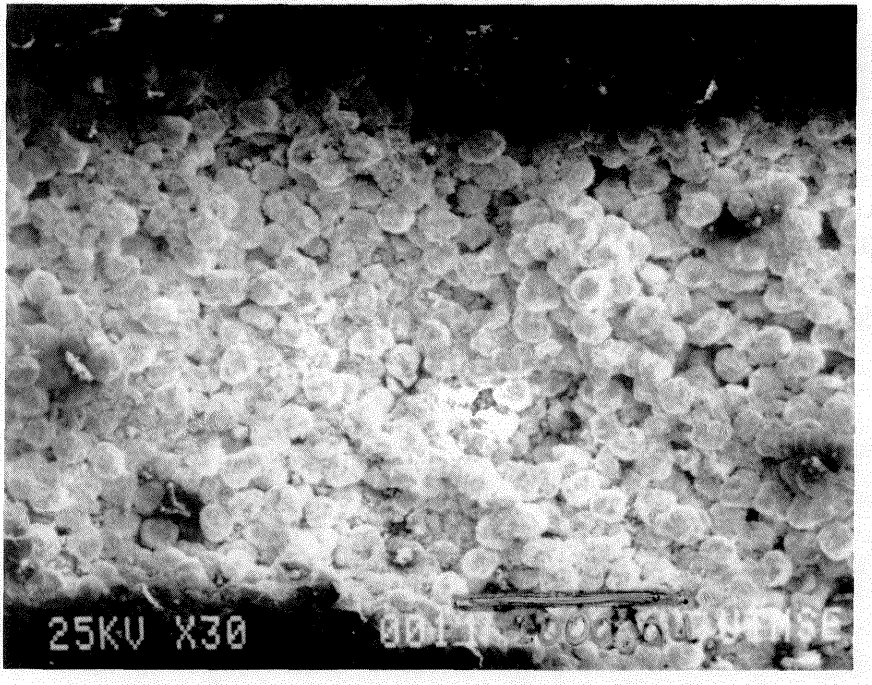

Figure 7. A cross section of an annealed sample (800°C for 24 hrs in H_2). The layer are completely indistinguishable. The laser power was 17 W and the scan speed was 3.41 cm/s. The magnification is 30X.

CONCLUSIONS

The selective laser sintering processing method was used to produce parts from intermetallic compounds. A mixture of $ZnCl_2$ fluxed 59% Ni and 41% Sn was laser sintered to form samples. The samples were post-process annealed to allow a Ni-Sn intermetallic to form. The parts were easily handled, but were not sufficiently dense to be of structural quality. X-ray powder diffraction established that Ni-Sn intermetallics did form. Selective laser sintering may prove to be a very valuable tool in the processing of intermetallics when research addressing the density and behavior of phase formation is complete.

ACKNOWLEDGEMENTS

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