SLS Processing of Functionally Gradient Materials

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Abstract

A developing SLS process, known as Multiple Material Selective Laser Sintering, will allow the material composition of a component to be varied in a controlled manner. This process could allow the fabrication of functionally gradient materials (FGMs) in which a blended interface exists. Two potential applications of FGMs are the reduction of thermal stresses in metal/ceramic joints and the matching of material properties to functional requirements. A tungsten carbide/cobalt system has been examined in which the ceramic/metal ratio has been varied in an attempt to control the hardness/fracture resistance ratio. An FGM powder bed was manually fabricated using a discrete banding technique. Results of traditional SLS processing of this powder bed are presented.

1 Introduction

Due to the nature of the powder delivery subsystem in the current SLS process, fabricated components consist of only one material system. By altering the powder delivery subsystem, laser sintering of multiple materials may be achieved. This new SLS process, known as Multi-Material Selective Laser Sintering (M^2SLS) is currently under development. This process has the potential to create a geometrically complex component while varying the material composition in a controlled manner. The potential ability to control the material composition at an arbitrary interior point could allow the development of unique components whose material properties are tailored to functional requirements. The variation of the material composition could be discrete or gradual in nature. The primary focus of this work is the examination of components containing a gradual change in material composition, or Functionally Gradient Materials (FGMs).

2 Functionally Gradient Materials

2.1 FGMs: A Description

A traditional component containing multiple materials will have regions of distinctly different material compositions which have been joined together as the result of chemical and mechanical interactions. These interactions, or joining processes, create a material interface of between 50 and 200 micrometers, which is basically discontinuous on a macroscopic scale. However, M^2SLS and other developing processes [1,2,3,4,5] allow the creation of a material interface where the material composition gradually changes from one material to another. For example, the bottom of a component could be a 100% metal alloy which gradually changes as one moves up through the component to a 100% ceramic composition at the top of the component. The ability to control the ratio of the two materials making up the FGM allows one to control the material composition and, therefore, the material properties at a given arbitrary interior point in the component. The gradual nature of an FGM also ensures that material properties will possess a continuous distribution throughout the interior of the component, and will not possess the discontinuous changes in material properties traditionally present in multiple material components.

2.2 Potential Applications of FGMs

The ability to control the material composition in an FGM allows one to match the material properties to the functional requirements demanded of the component at a given point. The control of material properties and the gradual nature of FGM interfaces also provide opportunities for increased bond strength, especially at metal/ceramic interfaces.

An example component in which material properties could be controlled is a tungsten carbide cutting tool in which the relative amounts of carbide and cobalt are varied. For this system, the material properties to be controlled are the fracture resistance and the ductility. In general, the cobalt provides ductility and resistance to brittle fracture, while the carbide constituent provides hardness and wear resistance. The functional requirements of the part demand high ductility in regions of stress concentration to prevent cracking and brittle failure while also demanding high hardness and wear resistance in regions such as the cutting edge. The example FGM component should possess relatively more cobalt in areas of stress concentration and more carbide in areas such as the cutting edge to provide a matching of material properties to functional requirements. The use of FGMs should result in an increase in cutting performance while maintaining or improving the fracture resistance of the component.

The nature of FGMs allow a blending of a material property across the interface between two materials. While a number of material properties could be controlled in this fashion, one of the most important properties with respect to bond formation is the coefficient of thermal expansion (CTE). The mismatch in CTEs normally present in a discontinuous metal/ceramic bond lead to destructive thermal stresses during thermal cycling or thermal shock loading[6]. Metal/ceramic bonds are frequently created at an elevated temperature and then cooled. The large CTE mismatch coupled with this large change in temperature will create a residual thermal stress in the component which may weaken the bond. Additionally, metal/ceramic bonds operate at an elevated temperature, which results in thermal cycling during normal repeated use. These repeated large changes in temperature and the metal/ceramic CTE mismatch generate transient thermal stresses which, when coupled with the residual thermal stress generated during bond formation, may destroy the bond[6]. An FGM component allows the CTE value to change gradually from a low value to a high value. This distribution of CTE values will tend to spread thermal stresses out over a volume of material instead of concentrating them at the interface[1]. The maximum thermal stress developed in the bond will therefore become much smaller and the bond will possess lower residual stresses and a greater resistance to thermal cycling, which will act to increase the strength of the metal/ceramic bond.

3 Experimental

3.1 Development of Experiments

3.1.1 FGM Fabrication Via Traditional SLS

A experimental study of the use of laser sintering to produce FGMs is being undertaken during the conceptual design phase of the M^2SLS process. This study will examine the effect of various laser sintering parameters on the quality of the resulting FGM samples. The purposes of this study are to ensure that the formation of an FGM is physically possible using a laser sintering technique and to provide information regarding process behaviors which could impact design choices made during the development of the M^2SLS process. Additionally, this study provides insight into the fabrication of FGMs using laser sintering without the costs associated with the development of the M^2SLS process.

Therefore, the performance of this study consists of the fabrication of FGMs using traditional SLS processing. Since the distinguishing difference between traditional SLS and M^2SLS processing is the powder delivery subsystem, performance of this study will require an alteration in the method of powder delivery. In general, these alterations should be as simple as possible and must result in the fabrication of an FGM on the powder bed. The simplest method to create an FGM consists of a manual discrete banding technique.

This technique involves mixing powder blends with differing ratios of the two FGM materials and manually laying these blends out in bands to create a gradual change in material composition. This method produces an FGM with discrete steps which approximates a fully continuous FGM. The FGM powder bed examined in this study was fabricated using this technique and then laser sintered with the traditional SLS process. Another FGM fabrication technique would utilize vibration-induced segregation to create a fully continuous FGM. In this technique, a thin, sealable powder dish is filled with a one-to-one ratio mixture of the two FGM materials. This dish is placed on its side and vibrated to induce segregration of the two FGM materials. After segregation is achieved, the dish would be placed into an SLS workstation and laser sintering would proceed. The third FGM fabrication technique involves the creation of a vertical FGM in the powder side cylinder, either manually with discrete layers or using the vibration technique described above. As the SLS process proceeds, the roller would deposit material of a gradually changing composition on the part side piston. This technique would create a multilayer FGM sample in which the FGM would be oriented vertically. However, potential problems of segregation and mixing during roller transport of the material may be encountered with this technique.

3.1.2 Material Selection

The selection of the material system was based on the following criteria. First, the material system should be extensively documented in the literature so that the experimental FGM results can be compared to standard fabrication techniques. This comparison is required in order to gauge any improvements in the overall performance of a FGM component versus a standard component. Second, the material system should be commonly used to provide relevance to industrial applications. Third, the two material components in the system should be clearly identifiable with two distinct material properties which act independently to fulfill the functional requirements of the component.

The material system selected is the tungsten carbide system discussed earlier (Ref. Section 2.2). This system is very well documented in the literature with an extensive collection of data regarding its material properties and behavioral characteristics. Tungsten carbide is the most commonly used material system for the fabrication of the carbide cutting tool inserts found in practically all industrial machine shops. Finally, the cobalt matrix correlates with ductility to fulfill the functional requirement of preventing brittle fractures in regions of stress concentration. The tungsten carbide constituent correlates with hardness and wear resistance to fulfill the functional requirement of maintaining a sharp and properly shaped cutting edge.

Tungsten carbide powder is available in two primary forms. The first form is a pre-alloyed powder in which each individual particle is made up of a cobalt matrix containing a tungsten carbide secondary phase. The other form is a powder mixture made up of individual particles of pure tungsten carbide and pure cobalt. The pre-alloyed powder is expected to provide a better microstructure since less migration of the molten cobalt is required to form a uniform cobalt matrix. However, the powder mixture form offers much more flexibility in the creation of custom blends of tungsten carbide with varying amounts of cobalt. A compromise choice of these two forms was made by choosing a 12% cobalt pre-alloyed powder as a base mixture. This base mixture was combined with a pure cobalt powder to create a series of powder blends with gradually increasing amounts of cobalt. This choice ensured a fairly uniform mixture of cobalt in each blend with little likelihood of cobalt depleted regions present in the mixture.

3.1.3 Experimental Parameters

The basic goal of this study is to examine the effect of different processing parameters on the quality of the resulting FGM coupons. Therefore, it was necessary to consider what the different processing parameters are and which parameters should be examined in this study. The potential parameters which could be examined are tabulated in Table 1 below.

An initial examination of the list of experimental parameters reveals that some of the parameters may be eliminated from the study based on the results of previous research on the direct SLS processing of cermet composite systems[7]. Results of previous work indicate the need for a high vacuum atmosphere and pre-heating of the powder bed for satisfactory sintering of metal systems to occur. Therefore, experiments to examine these parameters do not need to be conducted and they may be eliminated from the list of potential parameters. Additionally, an experimental examination of some of the parameters will require substantial process development. For example, the laser pre-heat and laser modulation parameters would require the development of a laser pre-heating system and a real-time laser power control system before an experimental examination of these parameters could occur. These more problematic parameters will be examined after experiments have been performed with respect to the other parameters.

Experimental Parameter	Description
Processing Atmosphere	Process in air versus high vacuum
Discreteness of FGM	Process discrete FGM created using the manual discrete banding technique versus continuous FGM created using the vibration-induced segregation technique
Pre-Heat	Process with versus without pre-heat
Laser Pre-Heat	Process with normal pre-heat versus pre-heat from laser
Scanning Speed	Process at different scanning speeds along fast axis
Laser Power	Process at different laser powers
Vector Density	Process at different scanning speeds along slow axis
Scanning Orientation	Process with vectors parallel to FGM versus perpendicular to FGM.
Laser Power Modulation	Process with real-time laser power modulation to match material composition.

Table 1: Experimental Parameters

The results of previous work and a desire to avoid excessive process development reduces the list of potential parameters to (1) Discreteness of FGM, (2) Scanning Speed, (3) Laser Power, (4) Vector Density, and (5) Scanning Orientation. The two parameters, Scanning Speed and Scanning Orientation, were chosen as the parameters to be examined in the initial set of experiments.

3.2 Experimental Preparation

The percentages of cobalt used in industrial environments generally range from approximately 3% to 12%. While the most industrially relevant choice would have been a series of roughly 10 blends ranging from 3% to 12% cobalt, the resulting material composition gradient would have been somewhat mild. A steeper gradient in which the percentage of cobalt ranges from roughly 10% to 100% will tend to reveal the effects of changes in the laser sintering parameters more strongly then a mild material composition gradient. Therefore, the composition of the different blends started from the 12% cobalt pre-alloy (Ref. Section 3.1.2) and increased in increments of roughly 10% up to a fully

100% cobalt metal blend (Ref. Table 2). The mixing of the pure cobalt powder with the 12% cobalt pre-alloy was performed using an industrial roller mixer for approximately 8 hours per blend.

Blend Number	% Cobalt	% Tungsten Carbide
1	12 %	88 %
2	24 %	76 %
3	36 %	64 %
4	48 %	52 %
5	60 %	40 %
6	72 %	28 %
7	84 %	16 %
8	100 %	------

Table 2: FGM Blends

The FGM sample size was chosen to be 0.5 inches square with a resulting band width of each blend of roughly 0.0625 inches as shown in Figure 1. This small size allowed several samples to be fabricated in one experimental run and yielded a more continuous FGM by forcing the bands widths to be narrow. In order to optimize the number of samples which could be fabricated, the FGM bands were oriented as shown in Figure 2. Note that the band labeled 'fgm' only contained the 24% Co to 84% Co blends since the 12% Co and 100% Co blends also acted as boundaries between the fgm bands. This arrangement allowed the processing of 12 samples per run and the raster scanning of samples both parallel and perpendicular to the FGM gradient.

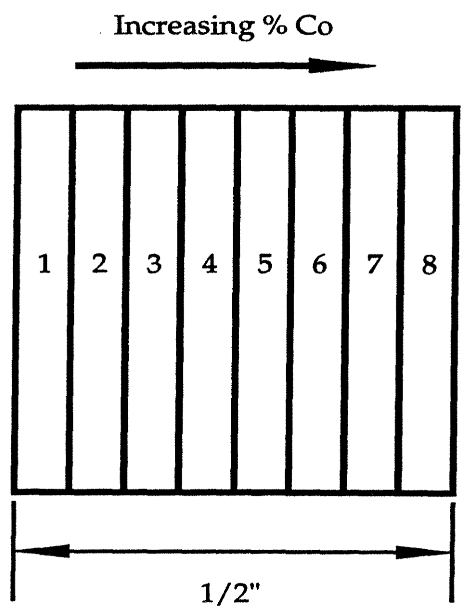

Figure 1: FGM Sample Coupon

Before scanning was initiated, the SLS chamber was brought to a high vacuum state and the powder bed was pre-heated to approximately 700°C. Proper alignment of the scanning location and the FGM bands was of critical importance to the fabrication of relevant FGM samples. An iterative process was used with a solid state positioning laser to confirm the scanning location prior to actual laser sintering. All of the samples were scanned using constant laser power with control of laser energy input achieved via changes in scanning speed.

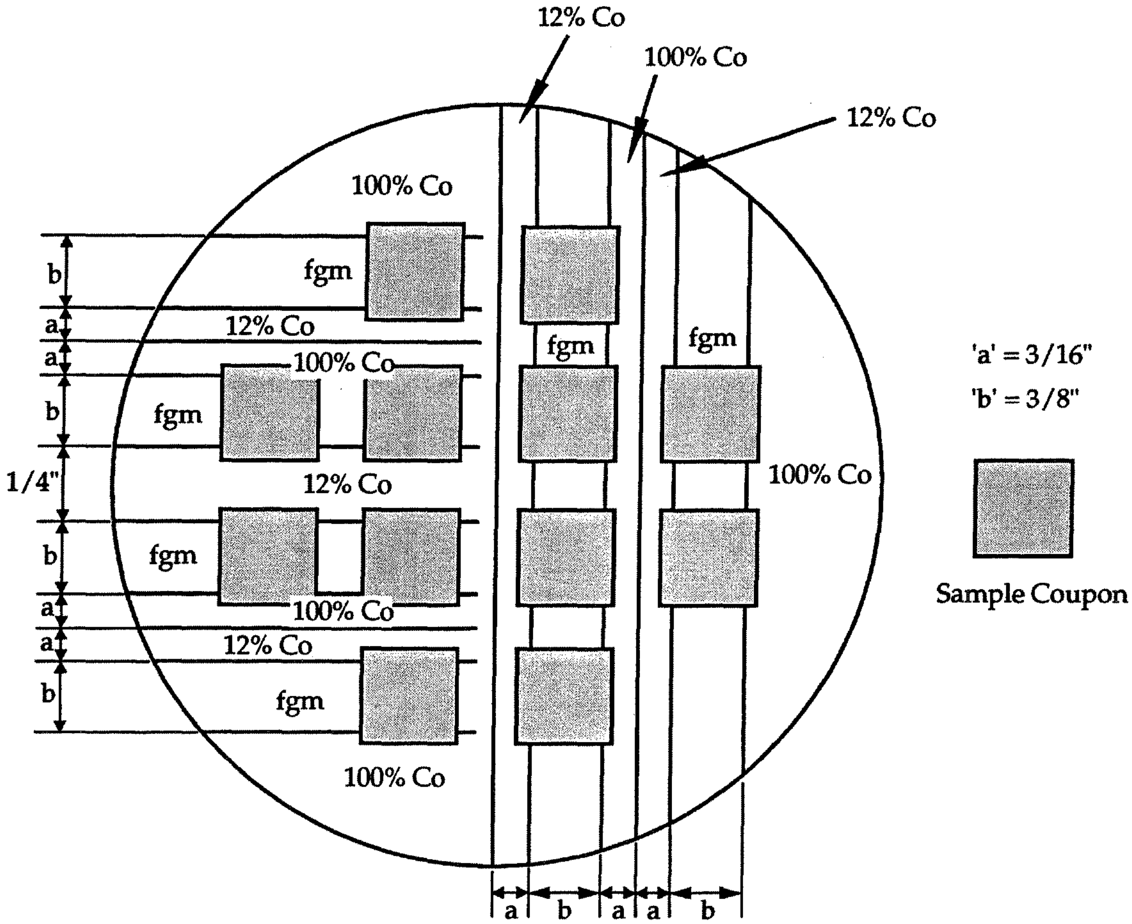

Figure 2: FGM Sample Placements

Laser power and scanning speed can be used to calculate the energy density, which is basically a description of the laser energy input per unit area. The energy density is calculated using the Andrew number equation[8] as shown below.

$$A_N = \frac{P}{\nu\,\delta}\left[\frac{\mathrm{J}}{\mathrm{in}^2}\right] \qquad (1)$$

where

P is the incident laser power (Watts)

ν is the scan speed (in/sec)

δ is the scan spacing (in)

Using the Andrew number equation generates the following three unique values of energy density: (1) 31.4 kJ/in^2, (2) 15.7 kJ/in^2, and (3) 10.5 kJ/in^2. Four samples were fabricated for each of the three unique energy densities. Two samples for each density were scanned parallel to the FGM and the remaining two samples were scanned perpendicular to the FGM. This arrangement allowed three unique energy densities and two scanning orientations to be examined. Two samples were created for each of the six possible combinations of experimental parameters for a total of 12 samples. Scanning was initiated in the 12% cobalt region for all samples scanned parallel to the FGM.

3.3 Experimental Results

The results of the experiment must be examined with respect to the two experimental parameters studied. First, a comparison is made of those samples fabricated with the same energy density, but with different scanning orientations. This comparison indicates that a raster scan parallel to the FGM produces less cracking and balling then a raster scan perpendicular to the FGM (Ref. Figure 3) In Figure 3, the cobalt-rich region is at the top of the samples and scanning occurred in a vertical direction for those samples to the left while scanning occurred in a horizontal direction for those samples to the right. The cracking and balling of the samples on the right is most prevalent in the cobalt-rich region. The large percentage of cobalt in this region promotes the formation of melt pools that ball up due to

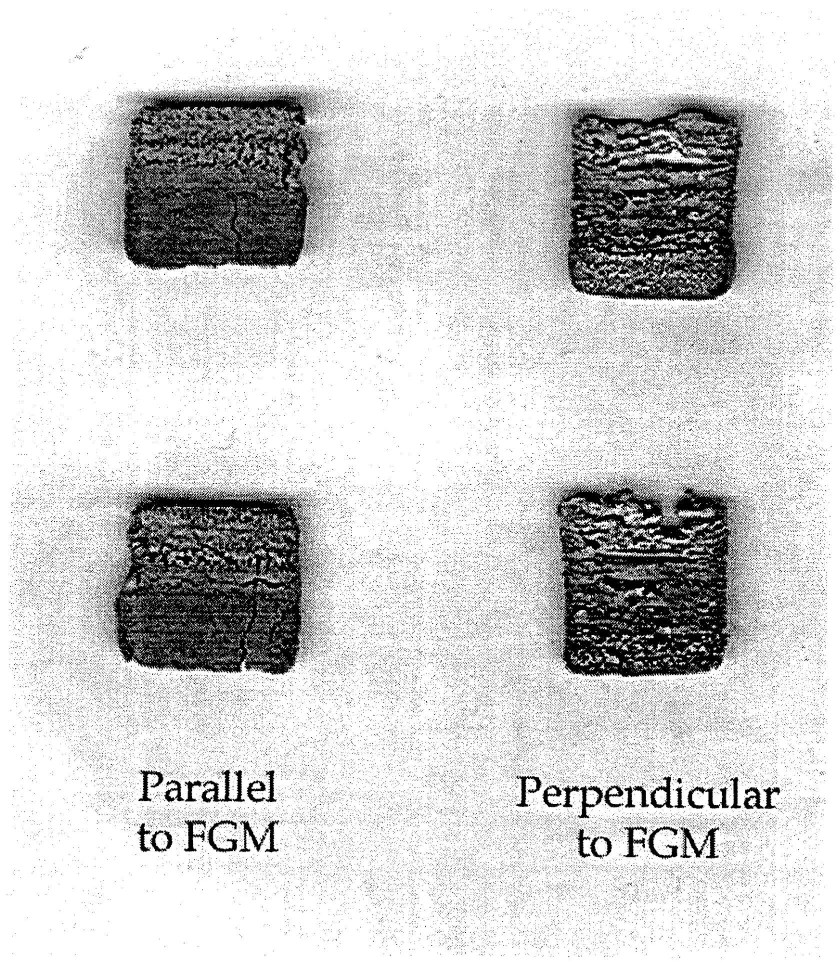

Figure 3: Results of FGM Scanning Orientation

surface tension and leave large cracks and voids in the sample. In the tungsten carbide-rich regions, less cobalt is present so that melt pools and associated balling and cracking does not occur. For scanning orientations parallel to the FGM, the laser will remain in the cobalt-rich region for a short time and then scan down into the tungsten carbide-rich region. By the time the laser has returned to the cobalt-rich region, the previously melted cobalt has partially resolidified so that only a small amount of cobalt remains in the liquid state. Therefore, the large amount of liquid cobalt required to form melt pools does not develop and the associated cracking and balling does not occur in the parallel scanning orientation.

Next, a comparison is made of those samples fabricated with the same scanning orientation, but with different energy densities. It was observed that increases in scanning speed reduced the amount of cracking and balling in those samples with a scanning pattern parallel to the FGM (Ref. Figure 4), however, no correlation between scanning speed and the amount of cracking and balling can be drawn for those samples fabricated with a scanning pattern perpendicular to the FGM. For the scanning pattern parallel to the FGM, faster scan speeds decrease the energy density and melt less of the cobalt during each vector scan. Therefore, at higher scan speeds, insufficient liquid cobalt is formed to develop melt pools and the associated balling and cracking behaviors are reduced. For the scanning pattern perpendicular to the FGM, increased scan speeds should also tend to reduce the amount of observed balling and cracking. However, the melt pools created by the perpendicular

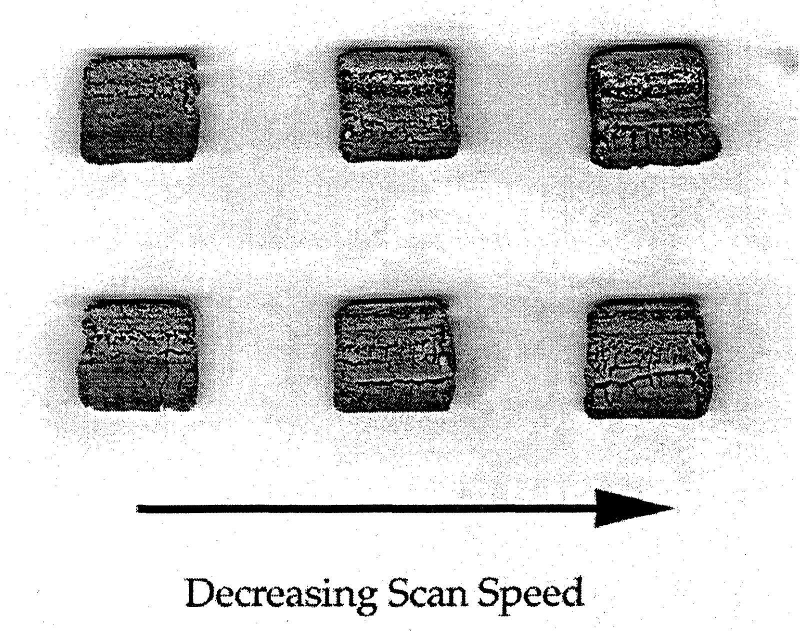

Figure 4: Effect of Scanning Speed (Parallel Scanning Pattern)

scanning orientation obscure this effect, and in general prevent the development of any correlation between scanning speed and the amount of balling and cracking. Therefore, the onset of balling and cracking is in general more sensitive to scanning orientation then to scanning speed. A large amount of sparking and off-gassing was also observed during processing which may be attributed to oxides and moisture contaminates in the powder.

4 Future Work

The immediate work to be completed is further testing and measurement of the samples presented above. These measurements include the development of micrographs to examine sample microstructures, and impact and hardness testing to determine the fracture resistance and hardness of the samples. Additional testing may include measurements of the sample density to determine the extent of porosity and SEM analysis to determine the material composition along the FGM. This analysis will check for disturbance of the FGM due to segregation or diffusion during the sintering process.

The next set of experiments to be conducted will duplicate the experiments described above with the following changes. First, an extensive bake-out of the powder for 12 to 14 hours at 500° C will be conducted to remove oxides and moisture from the powder before sintering. The experiments will be conducted at a higher pre-heat temperature of approximately 900° C. The higher pre-heat temperature will tend to reduce the extent of the balling by decreasing the surface tension and viscosity of the molten cobalt. Additionally, the increased temperature of the powder bed will cause an increase in the surface energy of the unmelted powder. In order to minimize the surface energy of the system, the area of the unmelted powder will tend to decrease so that increased wetting of the powder bed by the liquid cobalt will occur and the amount of balling will be reduced. These next set of experiments will also be conducted with a scanning orientation parallel to the FGM and at faster scan speeds based on the experimental results presented above (Ref. Section 3.3).

For a scanning pattern perpendicular to the FGM, additional experiments will orient the FGM so that scanning is initiated in the cobalt-rich region. It is expected that the amount of balling and cracking will increase due to the lack of powder bed pre-heat from previously sintering material. Future experimental work may examine the effect of real-time control of laser power during scanning of the FGM in an attempt to match laser energy input to material composition. Additionally, attempts will be made to develop a more continuous FGM using the vibration-induced segregation technique described previously (Ref. Section 3.1.1).

5 Conclusions

These experiments represent one of the first attempts to produce FGMs using the traditional SLS process. FGMs were manually fabricated in a discrete configuration using a series of tungsten carbide and cobalt metal blends. Eight blends were used in which the percentage of cobalt varied from approximately 10% to 100%. SLS processing of the FGM was performed with scanning orientations parallel and perpendicular to the FGM and with three unique energy densities. Experimental results indicate that a raster scan parallel to the FGM and high scan speeds produce samples with the least amount of balling and cracking. The results also indicate that the onset of balling and cracking is in general more sensitive to scanning orientation then to scanning speed. The next set of experiments will include a pre-processing bake-out to reduce powder contamination, higher pre-heat to reduce the amount of balling, and faster scan speeds based on the results presented above.

6 Acknowledgments

Funding for this work was provided by the National Science Foundation Program Materials Processing and Manufacturing: NSF GOALIE under award number DMI 9622287.

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