

Fabrication of x-graded H13 and Cu powder mix using high power pulsed Nd:YAG laser

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Abstract

The manufacturing of Functionally Graded Material (FGM) parts using Solid Free Form manufacturing technologies has been carried out since early 1980. At present, most of the powder manufacturing techniques are being focused on layering powder with different powder blend compositions with Z gradients (graded in direction of layer build). Although, there are a few researchers working on multi powder feeder and deposition system, the study of laser fusion of the deposited powder (by a powder deposition system) is minimum or not known to date. Consequently, the manufacturing of functionally graded structures is still geometry limited. This work was focused on the manufacturing of X-graded (graded along the powder bed plane) specimens with H13 tool steel and Cu mix. Five bimodal powder blends were used with a multi-container feed hopper to spread powder layers for the selective laser fusion of the powder. The powder was fused using a high power Nd:YAG pulsed laser using a specific scanning strategy to reduce porosity. Specimens were produced with graded Cu within the H13 matrix. The specimens were analysed for dimensional accuracy, microstructure, porosity, cracks and micro hardness of the FGM.

Keywords: functionally graded materials, laser fusion

Introduction

Functionally Graded Materials (FGM) has been in the focus of researchers since the 80's (Suresh & Mortensen; 1998). The main advantage of exploring the functionally graded manufacturing techniques and materials combinations is the possibility of producing highly optimised parts for high performance applications. Unfortunately, most of the graded materials and processes are geometry dependant and engineers are not able to use these materials easily.

Lately a number of researchers are working on the application of layered manufacturing (LM) technologies to produce FGM parts. As these technologies can virtually manufacture free forms without any geometry limitations, it would be the best possible approach to make the FGM parts fabrication successful. Furthermore, as the LM technology is an additive process, this would make the deposition of the powder in the required composition easy and flexible.

The use of layered technologies to produce FGM parts is limited to the development of Computer-aided Design (CAD) and Finite Element Analysis (FEA) data, Computer-aided Manufacturing (CAM) processing and Local Composition Control (LCC) of materials. The CAD and FEA are important tools as these aid the engineer in deciding the composition of material in various regions of the part and the transition between the materials. Also, CAD

data for FGM parts should be interchangeable with FEA for optimum engineering design. The CAM must be able to read the CAD data with its different materials and process it for each layer/step during the fabrication process. Finally, LCC is a new and innovative devices that can deliver different compositions of materials to a layer according to the CAM instructions. However, at present the LCC devices are in early stages of research and development.

This work uses a simple X-graded powder deposition system to deposit graded layers to produce FGM parts. However, these parts are limited to X-grading along the powder bed surface for selective laser fusion with high power Nd:YAG pulsed laser. At this stage, the system is used for parts fabrication in order to optimise laser parameters which improve the mechanical properties and minimizes porosity and cracks. The paper presents the results obtained from the initial work. The microstructure of the material, dimensional accuracy, microhardness, porosity and cracks were analysed.

Background

Functionally graded materials are given importance because of the possibilities of combining distinctive materials to add new functions to the same component. Also, designers could optimise form and function in their new projects improving the performance of the products. According to Suresh & Mortensen (1998), FGM could be used to manufacture components with engineered gradual transitions in microstructure and/or composition. This gradual transition results in different functional performance requirements that vary with location within the component.

Nowadays, the manufacturing of the FGM parts is based on constructive and transport-based processes (Suresh & Mortensen; 1998). Constructive processes are based mainly on powder densification/sintering techniques and coating processes. Transport-based processes are those associated with mass transport, thermal process, settling and centrifugal processes and infiltration and macro segregation processes. Many pre-forms and simple geometries are being produced using these processes. However, there is still lack of “smart” processes (such as free-form fabrication) that could deliver complex parts to unleash all the possible applications for FGM.

In order to improve and produce complex parts using the FGM technique, many researchers from layered manufacturing technologies, often referenced as Rapid Prototyping and Manufacturing (RP&M), are working on this area. Selective laser sintering (SLS) works with fine layers of powder deposited over a platform. Each layer is selectively sintered in some regions using a laser beam. After the layer is completed, the platform is lowered and a new powder layer is spread over the previous one. The laser beam sinters the new layer adding it to the previous one, and the process is repeated until a complete part is built. SLS uses a metallic powder coated with a thermoplastic binder to give shape to the part through the melting of the binder. Subsequent steps are binder removal, pre-sintering, sintering and copper/bronze infiltration. Currently, three kinds of materials that can be used; LaserForm ST 200 (54% 420 stainless steel and 46% bronze), LaserForm ST 100 (60% 420 stainless steel and 40% bronze) and LaserForm A6 (Steel, not much information available) (3D System 2004). Direct metal laser sintering (DMLS) is based on the same process of SLS but it uses the power of the laser to sinter *in loco* the powder, without the necessity for special post-processing in an oven to increase the density of the part. Nevertheless, the parts present high porosity and lower mechanical properties compared to those obtained by selective laser sintering (Storch et al., 2003). There are two materials currently available for this process; DirectMetal 20 & 50 μ m (nickel, bronze and copper phosphate mix) and DirectSteel 20 &

50 μ m (steel alloy, details are not available). Laser cladding works by coaxially blowing powder with shield gas over a high power density laser spot and subsequently fusing the powder. Laser cladding is successful in using more materials than other two technologies (Atwood et al., 1998; Ensz, et al., 2002; Lü et al., 2001). This technique can be used for building fully dense parts. Unfortunately, parts made by this method present some geometrical limitations and low tolerances. Usually it is necessary to perform milling operations after building the part but many advances have been made in recent years (Lü et al, 2001).

These additive free-form manufacturing technologies can produce free-form parts of any complex geometry. One of the main issues to produce FGM in RP&M machines is the Local Composition Control (LCC). A couple of works such as Kumar et al (2004) and Yang et al. (2004) are investigating the use of capillary tubes to control the deposition of powder. Ensz et al (2002) have been studying the optimization of two powder flows into the LENS process (Laser Engineering Net-Shaping) to build gradients from H13 to M300 steel alloys. Jepson et al (2003) have been working on the development of a selective laser machine to build FGM layers using a polymeric binder to give shape to the parts prior to de-bind and to sinter in the furnaces. Cho et al (2002) have investigated the LCC for the 3D Printing process by obtaining data for a model from FEA and Voxel space geometries.

Various researchers have investigated the materials that can be processed by this method. In Liverpool University, UK fully dense stainless steel specimens have been made using nanosecond pulsed Nd:YAG lasers by Direct Metal Laser Re-melting (DMLR), a variation of SLS process (Morgan et al, 2001). Also porous thin wallets structures with copper powder melting have been investigated through the same method (Pogson et al, 2003). It was found that an insufficient melting of the copper occurred in the samples. Also, continuous wave mode achieved better results than the pulsed laser mode in the fusion of the Cu powder. The production of dense H10 tool steel parts and graded H10 with tungsten carbide (WC) was evaluated by Su (2002). FGM structures of H10 with percentages up to 20% of WC had increased the micro hardness of the H10 matrix. Leong et al (2002) have produced copper matrix composites through laser sintering with a CO₂ laser. Porous parts with Ti-B₄C-Cu were made. The addition of nickel (Ti-B₄C-Cu-Ni) improved the wetting and diffusion between the particles and decreased the porosity. Also nickel, aluminium and stainless steel powder fusion was investigated by Abe et al (2001) using two sources of laser energy, CO₂ and Nd:YAG, with strategies of pre-heat and re-heat the material. Using dual scanning with Nd:YAG laser melting and CO₂ laser re-heating improved the materials ductility.

Methodology

At present, a simple and manual X-graded powder deposition system, Figure 1, was designed to evaluate parts produced and study the processing issues. In order to obtain the X-graded regions, a powder feed hopper was built with eight compartments for the mixture of powders. The pre-mixture powders were placed in the compartments of the hopper according to the desirable composition design. The hopper slid over a platform spreading the powder mixtures in trails over a substrate placed on the platform. A high power fibre-optic delivered Nd: YAG laser was used to fuse the layer. Argon gas assist was used in order to avoid the oxidation of the material. After completion of the layer, the platform was lowered and a new powder layer was spread over the previous fused layer. The laser fuses the new layer, and the process is repeated until the part is completed.

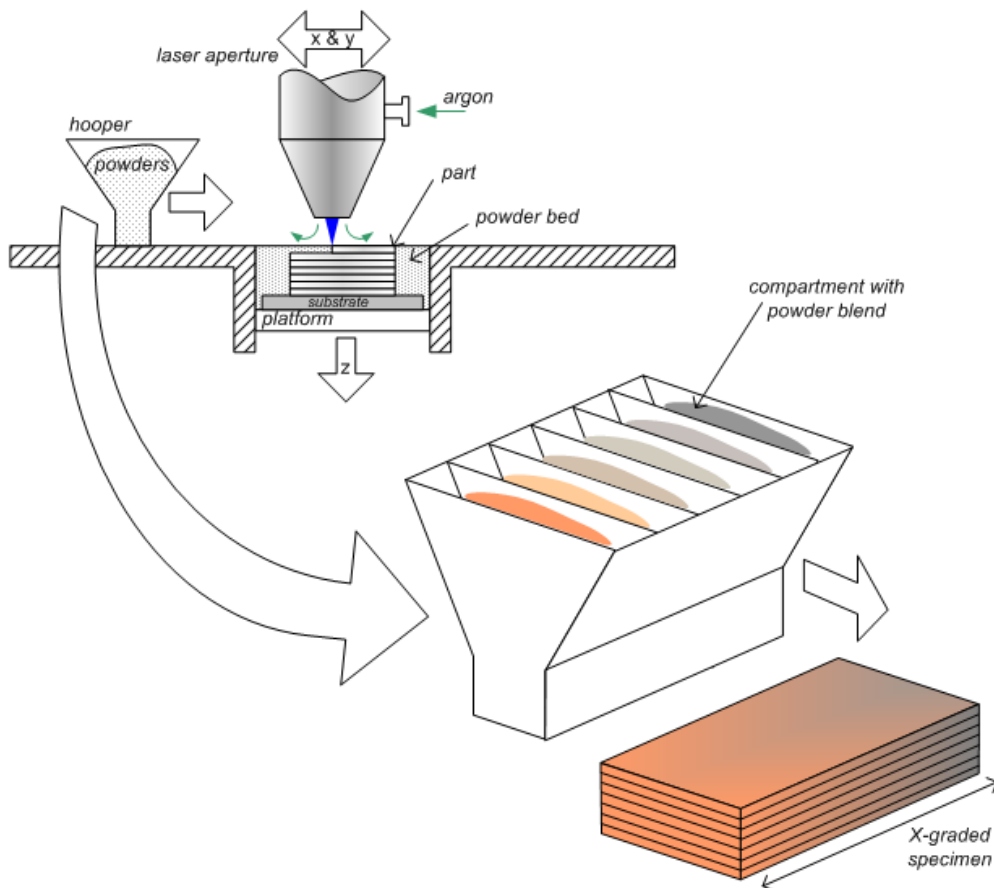


Figure 1. A schematic diagram of x-graded de-coater

The H13/Cu powders were mixed in five different compositions. The powder blends were tailored to achieve the particle size ratio of 7:1 to increase the apparent powder density. The powder mixture gradients from H13 to Cu were made by discrete increments of 12.5% until reaching 50% of Cu in H13 powder. The powder was sieved and particles in the range of 106 to 150 μm for H13 and Cu were mixed with particles below 38 μm and 22 μm of H13 and Cu respectively. The H13 tool steel powder was spherical and the Cu powder was more irregular and agglomerated. The H13 tool steel alloy composition was, in percentage weights: Fe 91.2%, C 0.4%, Cr 5.2%, Mn 0.4%, Si 1%, V 1% and Ni max. 1% and the copper was OFHC Cu (Osprey, 2003). Figure 2 shows images obtained in a Scanning Electron Microscope (SEM) from three of the five used mixtures/blends (H13, H13+25%Cu, H13+50%Cu).

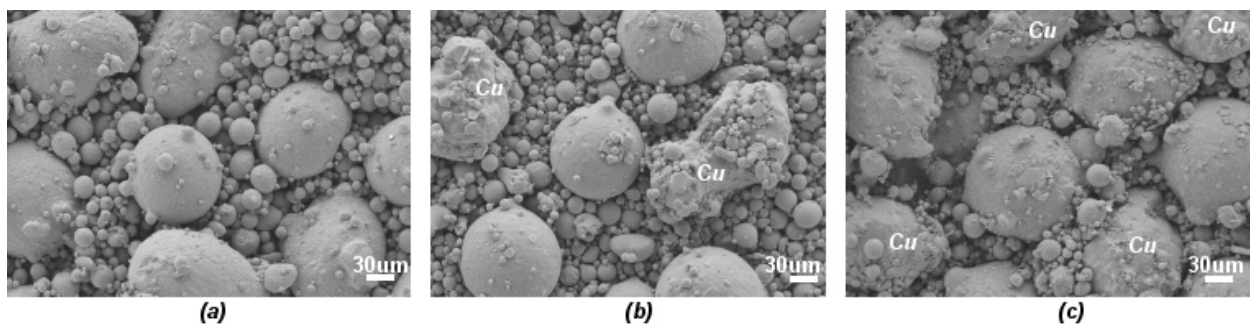


Figure 2. SEM images from the powder blends (a) H13, (b) H13 + 25%Cu and (c) H13 + 50% Cu. Note: Only large particles are indicated.

The powder mixture of H13/Cu was filled in each compartment of the hopper (see Figure 1) in sequence by increasing Cu percentage to the 50% in the middle compartment. The powder layer thickness was approximately 300µm and the substrate used was mild steel plate. From the work conducted previously at Loughborough University (Su, W., 2002), the refill laser scanning strategy (Figure 3) has been identified as an accurate building strategy. The strategy consisted of alternate scanning vectors followed by a refill of powder and scanning the vector-gaps. The use of this pattern, which is shown in Figure 3, is better explained in Su (2002). The pattern accounts for the shrinkage and contraction of powder during the laser processing. Some powder gets sucked to the melt pool due to capillary action and a fraction of material is lost due to spatter effect. This leaves the scanned layers un-even and voids areas that results in a pore during the fabrication of the whole cube. This effect can be minimised by the alternate scanning strategy and refill in between the scanning process.

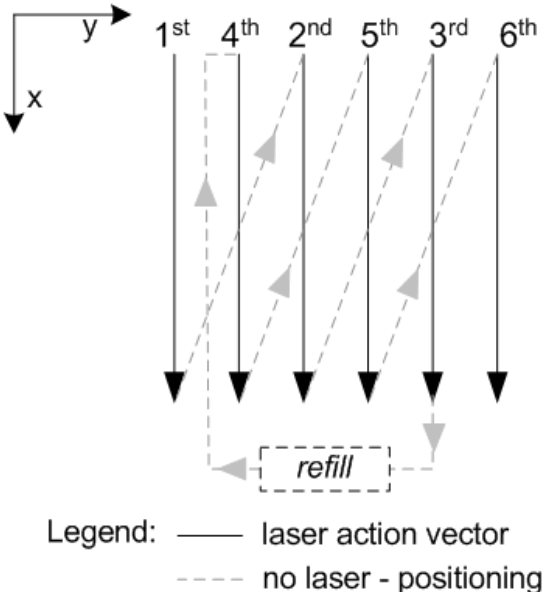


Figure 3. The refill scanning strategy

The specimens were built using a high power Nd:YAG pulsed laser (GSI Lumonics JK701H). The process parameters were optimised in the previous work (Su, W., 2002). The pulse energy used was 10J, repetition rate of 10Hz, pulse width of 5ms and the feed rate of 3.33mm/s. The laser spot diameter was 0.8mm. The average laser power was 100W with an energy density of 37.5J/mm² and the peak power of 2kW. The specimens were fused under room temperature and under a localized gas assist using argon to reduce the oxidation.

Specimens were built at 40x6x4mm dimension. The specimens were cross-sectioned, mounted, polished and etched with 2% of nitric acid solution. The microstructure and the microhardness analyses were performed. The dimensional accuracy analysis was performed on 10x10 mm square specimens. The microhardness test was performed along the X gradient with the spacing of 1mm using Vickers hardness system.

Results

The purpose-built X-graded recoater system’s performance was found to be good. The composition was distributed discretely over the powder bed. Figure 4, shows the distribution of the powder mixture and the fused layer of a specimen.

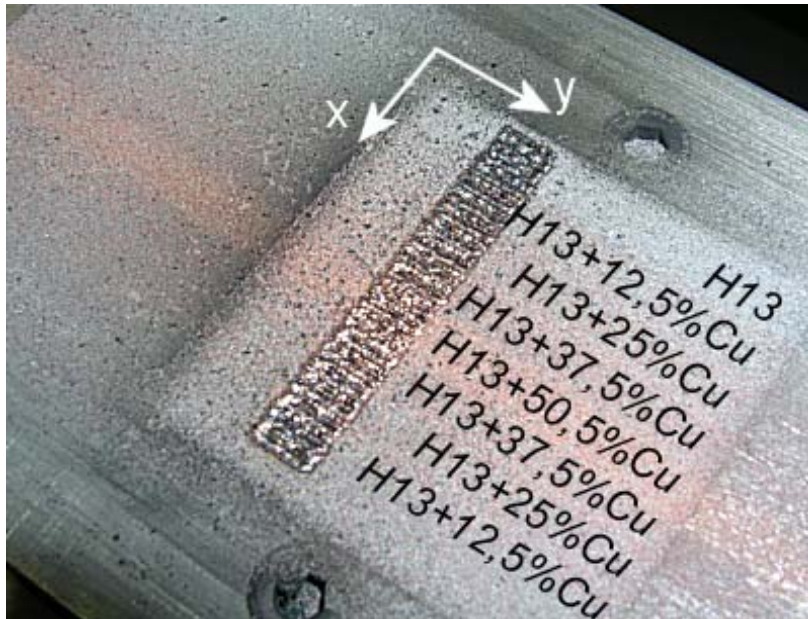


Figure 4. Image of the laser fused X-graded layer and the material composition.

The surface quality was similar despite the variation in the H13/Cu compositions. The specimen's top layer was rough and irregular. The surface could be re-melted by using lower laser intensity. However, this is not performed in this work as the focus is to optimise the parameter and achieve the optimal mechanical and microstructure properties. It also can be seen that there is no distinct boundaries between the various compositions of H13/Cu mixtures.

The dimensional accuracy measurements were performed on six specimens. The average error obtained in the X and Y directions are 0.8% and 1.3% respectively and the standard deviation was 0.5. This results show that the process parameter used produces accurate parts in those directions. However, as the powder layer deposition is partially automated and powder layer thickness is not accurately controllable, the average error in the z direction was found to be -6.7% with the standard deviation of 1.2.

Figure 5 shows one of the specimens and the longitudinal cross-section image. The cross sections along the specimens indicated the presence of pores in all formed alloys. Cracks were strongly present in the 12.5, 25 and 37.5%Cu blends. These cracks could have been formed due to the high cooling rate achieved by the Cu addition to the H13. The formation of the cracks was found to be similar to the cracks in laser welded parts. These cracks could be formed in the middle of the laser beam and in the heat affected zone (HAZ).

Figure 6 shows the cracks and porosity for the H13 and 12.5%Cu region. The cross section of the specimens showed relatively low porosity. The porosity level was measured and was found to be 2.35% at pure H13 region. In the region of H13+25%Cu and H13+50% Cu the level of porosity increased tremendously to 2.89 and 6.80% respectively. However, the percentages of porosity found in these specimens are considered to be low compared to the conventional powder metallurgy (German, 1994).

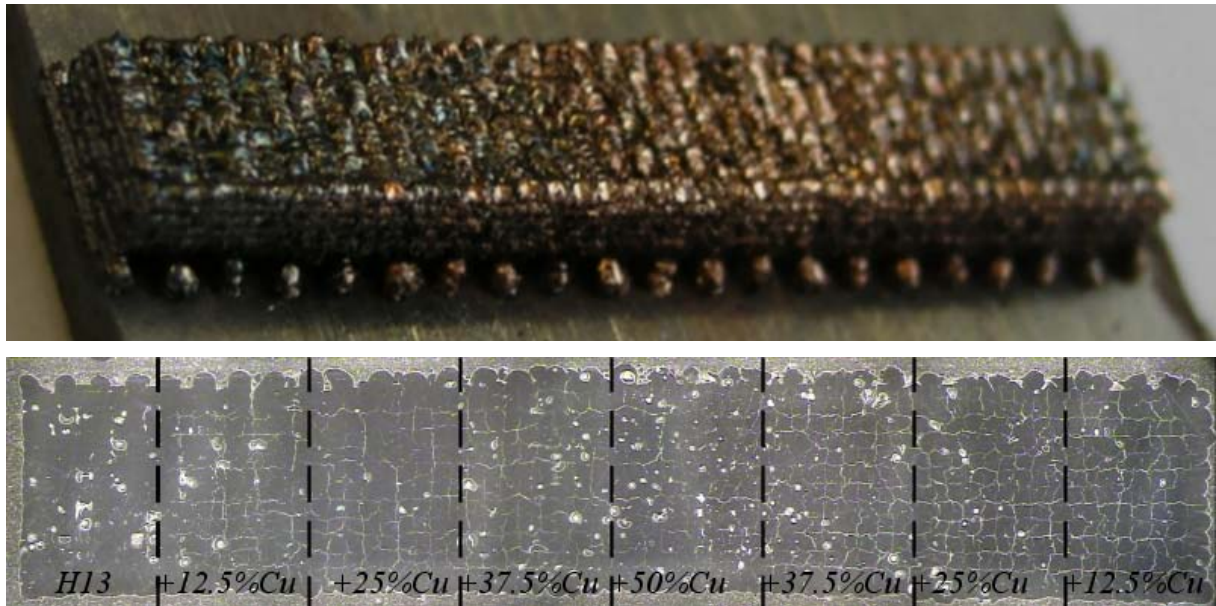


Figure 5. The overall view of the specimen (top) and the longitudinal cross-section of fused part with various H13/Cu mixtures (bottom).

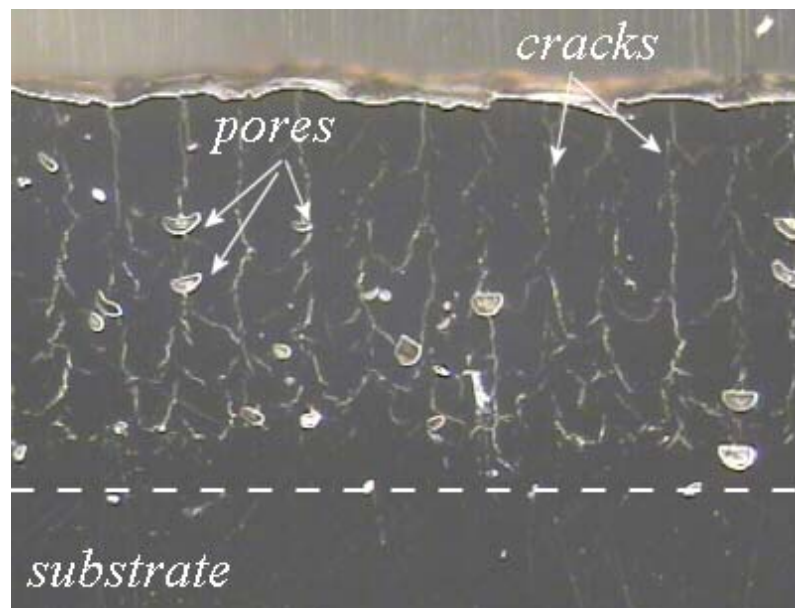
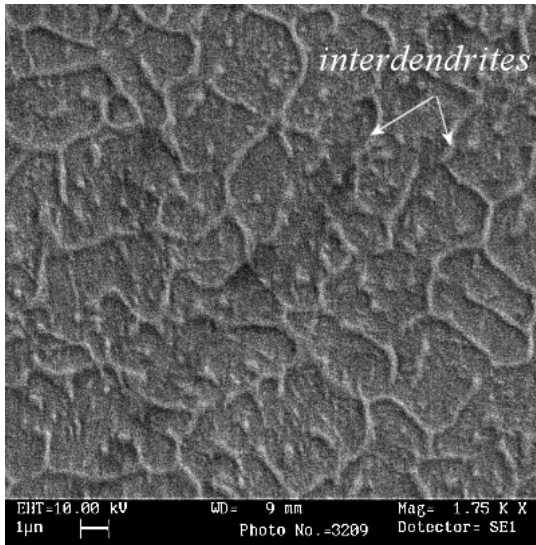
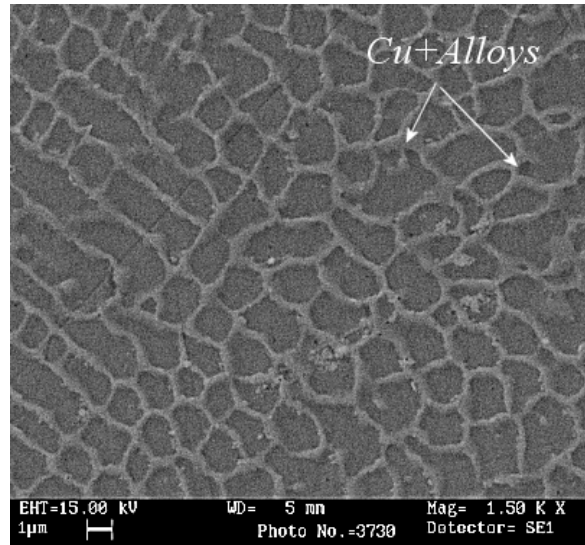


Figure 6. Cracks and pores in the 12.5%Cu section.

The pure H13 formed fine dendrites due to the effect of laser fusion of the material above the supercritical liquidus line and the rapid cooling. The alloying elements were concentrated in the interdendrite spaces (see Figure 7a). The dendrites can be formed by austenite or martensite depending on the region of the part. According to Brooks et. al. (1999) the re-melting and re-heating cyclic effects of the laser iteration with the addition of new layers cause this microstructure change. With the addition of the Cu, a new alloy formed and the microstructure remains the same, however with the segregation of the Cu in the interdendrite spaces (see Figure 7b).



(a) Pure H13 (mag. 1.75k X)



(b) H13+25%Cu (mag. 1.50k X)

Figure 7. SEM picture of pure H13 and H13+25%Cu microstructures.

With the higher percentages of Cu, in the region of 37.5% and 50% composition, a two phase material was characterized, especially in the 50% Cu region. Figure 8 shows the SEM picture where the two phase material was clearly perceptible in the 50% Cu region. Part of the Cu segregated forming isolated areas of Cu. The other part of the Cu was found in the interdendrite spaces as in the other regions. Spherical structures of the H13 and Cu could be found dispersed inside pure Cu region. X-ray diffraction analysis showed that these particles were not from non-fused H13 particles and contained approximately 23% of Cu.

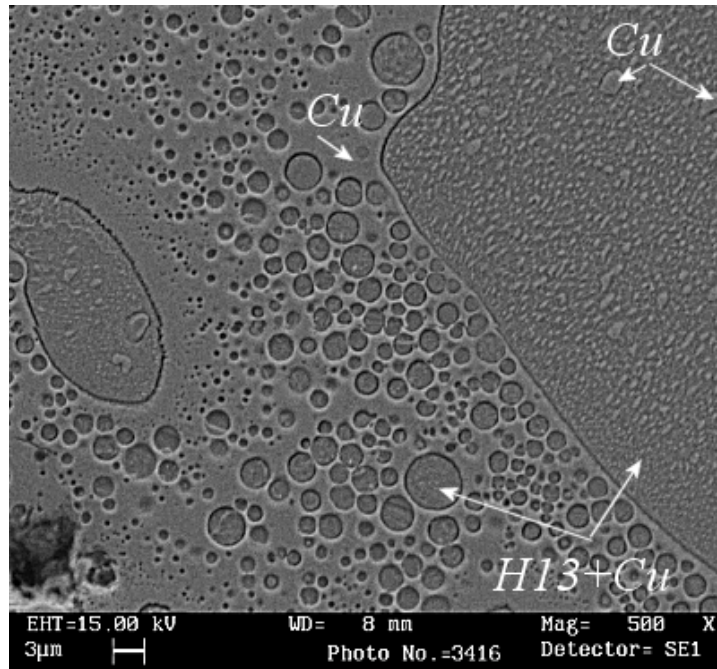


Figure 8. SEM picture of H13+50%Cu region (mag. 500 X).

The microhardness taken along the X-graded specimens shows the variation in hardness along the various regions. Figure 9 shows the microhardness results obtained from a single specimen. The graph shows that each region of the powder composition is 4mm long. The

variation of the hardness shows that the hardness reduces by increasing the Cu percentage. The variation of the hardness was observed to be high at H13+50%Cu mix region. This is mainly due to the two phase region formation as observed in the microstructure analysis. From this observation, the saturation of Cu in the H13 matrix is between 37.5 and 50%.

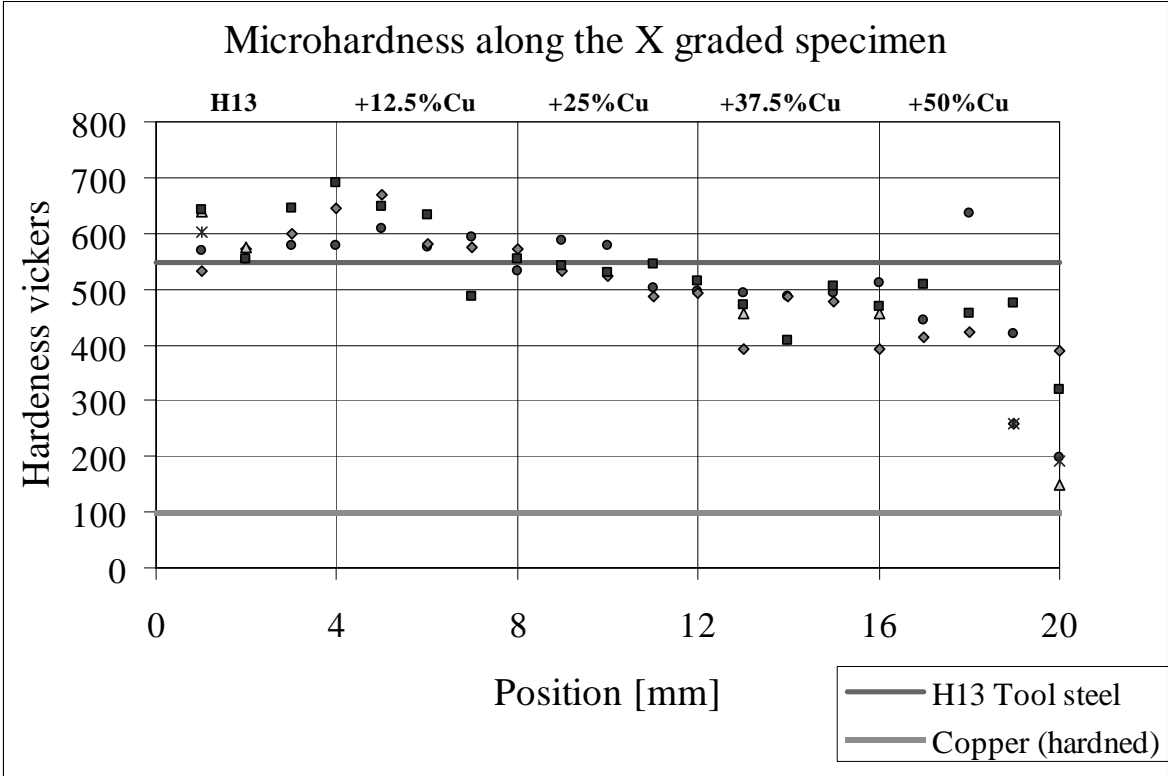


Figure 9. Microhardness along the X-graded specimen.

Conclusions

Producing FGM parts using direct metal laser melting is a complex task as it involves many areas of expertise. The work being carried out at Loughborough University is promising. The knowledge in laser processing and the associated process issues could be well utilised in producing complex FGM parts. However, there are many other areas that still need to be tackled in order to produce complex FGM shapes: powders deposition integration, dimensional accuracy and CAD/CAM interfaces.

In this work, the performance of the X-graded powder deposition system was found to be satisfactory. However, the gradient is discrete and voids are limited by the powder flow ability. Smaller particles could be used but also can reduce the flow of the powder. These issues could be solved with vibratory hopper and other mechanisms as being investigated by other researchers.

The porosity found in the FGM specimens was considered low. However, the cross-section image shows the formation of cracks in the H13 and Cu mixed parts. It is necessary to optimize the laser processing parameters to moderate the cooling rate of the material and hence reduce or eliminate the crack formation. Future work will involve in determining the best laser processing parameters for each blend according to the Cu percentages. The use of

pulse shaping and appropriate shield atmosphere and preheating could also help in reducing the cracks.

The two phase material formation could be expected as the percentages of added Cu are high. The saturation of the Cu in isolated areas is evident from the variation in the microhardness results. This saturation is high in the region where the Cu percentage is between 37.5% and 50%.

The surface quality and dimensional accuracy is not the major focus of this work. However, the dimensional accuracy in the x-y directions was found to be accurate, 0.8 and 1.3 percent of error respectively. At this stage, the surface quality is found to be poor, however, the parts could be machined or a laser re-melting technique could be used to smooth the surface.

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References

3D Systems Website, *LaserForm datasheets*, <http://www.3dsystems.com>, Accessed on February 2004.

ATWOOD, C., GRIFFITH, L.H., SCHLIENGER, E., ENSZ, M., SMUGERESKY, J., ROMERO, T., GREENE, D., RECKAWAY, D., 1998, Laser engineering net shaping (LENS): A tool for direct fabrication of metal parts. Proceedings of ICALEO '98, Orlando, FL, USA.

BROOKS, J., ROBINO, C., HEADLEY, S., GRIFFITH, M., 1999, Microstructure and property optimization of LENS deposited H13 tool steel. Solid Freeform Fabrication Symposium, Austin, Texas, USA.

CHO, W., SACHS, E. M., PATRIKALAKIS, N. M., CIMA, M. J., LUI, H., SERDY, J., STRATTON, C. C., 2002, *Local composition control in solid freeform fabrication*, White Paper, MIT – Massachusetts Institute of Technology, EUA.

DAWES, CHRISTOPHER, 1992, *Laser Welding – A practical guide* (Cambridge, England: Abington Publishing), ISBN 1855730340.

ENSZ, M., GRIFFITH, M., RECKAWAY, D., 2002, Critical issues for functionally graded material deposition by laser engineered net shaping (LENS). MPIF – Metal Powder Industries Federation Report, USA.

GERMAN, R. M., 1994, *Powder Metallurgy Science*, 2nd Edition (New Jersey, USA: MPIF), ISBN 1-878954-42-3.

JEPSON, L., BEAMAN, J., BOURELL, D., JACKSON, B., Mc ADANs, D., PEREZ, J., WOOD, K., 2003, *Multi-material selective laser sintering: empirical studies and hardware*

development, White Paper, Laboratory for Freeform Fabrication, The University of Texas, Austin, USA.

KUMAR, P., SANTOSA, J. K., BECK, E., DAS, S., 2004, Direct-write deposition of fine powders through miniature hopper-nozzles for multi-material solid freeform fabrication. *Rapid Prototyping Journal*, MCB University Press. ISSN 1355-2546, Volume 10 – No 1, 14-23.

LÜ, L., FUH, J., WONG, Y.-S., 2001, *Laser-induced materials and processes for rapid prototyping* (Norwell, USA: Kluwer Academic Publisher). ISBN 0792374002.

MORGAN, R., SUTCLIFFE, C. J., O'NEILL, W., 2001, Experimental investigation of nanosecond pulsed Nd:YAG laser re-melted re-placed powder beds. *Rapid Prototyping Journal*, MCB University Press, ISSN 1355-2546, Vol. 7 No 3, 159-172.

YANG, S., EVANS, J. R.G., 2004, Acoustic control of powder dispensing in open tubes. *Powder Technology*, Number 139, 55-60.

MATWEB Material Property Data, *AISI Type H13 Hot Work Tool Steel, air or oil quenched from 995-1025°C*, <http://www.matweb.com>, Accessed on March 2004.

MENGES, G., MOHREN, P., 1993, *How to Make Injection Molds*, 2nd Edition (Munich, Germany: Hanser). ISBN 0-13-555475-6.

NORTON, R. L., 1996, *Machine Design: An Integrated Approach* (New Jersey, EUA: Prentice-Hall Inc). ISBN 0-13-555475-6.

OSPREY Powders Ltd. *Microfine Powder – H13 Datasheets*, <http://www.ospreymetals.co.uk>, Accessed on November 2003.

POGSON, S. R., FOX P., SUTCLIFFE, C. J., O'NEILL, W., 2003, The production of copper parts using DMLR. *Rapid Prototyping Journal*, MCB University Press, ISSN 1355-2546, Vol. 9, No 5, 334-343.

SMITH, W. F., 1993, *Structure and properties of engineering alloys*, Second Edition (USA: McGraw-Hill Inc). ISBN 007591725. 1993.

STORCH, S., NELESSEN, D., SHAEFER, G., REITER, R., 2003, Selective laser sintering: qualifying analysis of metal based powder systems for automotive applications. *Rapid Prototyping Journal*, MCB University Press, ISSN 1355-2546, Volume 9, Number 4, 240-251.

SU, W., 2002, Layered Fabrication of Tool Steel and Functionally Graded Materials with a Nd:YAG Pulsed Laser, PhD. Thesis (Loughborough, UK: Loughborough University).

SURESH, S., MORTENSEN, A., 1998, *Fundamentals of functionally graded materials* (London, UK: Institute of Materials), ISBN 1-86125-063-0.