Investigating a Semi-Solid Processing technique using metal powder bed Additive Manufacturing Processes

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The work reported investigates in-situ alloying using a semi-solid processing technique with metal powder bed Additive Manufacturing (AM); in this instance Selective Laser Melting (SLM) and Electron Beam Melting (EBM) were employed. This technique utilised customised powder blends that were processed at elevated temperatures. The selection of processing temperature considered specific alloy solidification ranges. As a result, parts with reduced residual stresses can be produced. In addition, the use of customised powder blends explored the feasibility of developing alloys specific to the process/application, thus increasing available material ranges for AM metal powder bed processes.

1 Introduction

Metal powder based Additive Manufacturing (AM) technologies such as Selective Laser Melting (SLM) also known as Direct Metal Laser Sintering (DMLS™), Electron Beam Melting (EBM) and Direct Metal Deposition (DMD) are being increasingly adopted with research conducted to enable processing of different materials. Extensive research and subsequent confidence in these technologies have resulted in quality parts that are directly used in various sectors such as automotive, aerospace, medical and other industries. Parts produced by metal AM have properties similar to parts produced using conventional methods; and are sometimes superior.

Metal powder bed technologies such as SLM and EBM share a similar basic working principle. The process begins with part design with computer aided design software. This 3D computer designed model is processed, additional supports are added (if required) and sliced into a number of layers before it is fed to the AM machine. In the machine a thin layer of metal powder is spread on a process table (movement constrained in vertical direction only). The high intensity energy source (laser beam in SLM and electron beam in EBM) scans and liquid phase sinters the desired area of the powder bed based on the sliced data from the file. On completion of a scan, the process table lowers by single layer thickness and fresh powder is spread. The beam scans the powder bed and process continues until a complete part is build. Figure 1 illustrates schematic of SLM and EBM technologies. SLM operates in an inert atmosphere of argon gas circulating in the processing chamber whereas the EBM is carried out under vacuum. The build rate in SLM is comparatively slower than EBM; however surface finish of parts produced in SLM is superior.

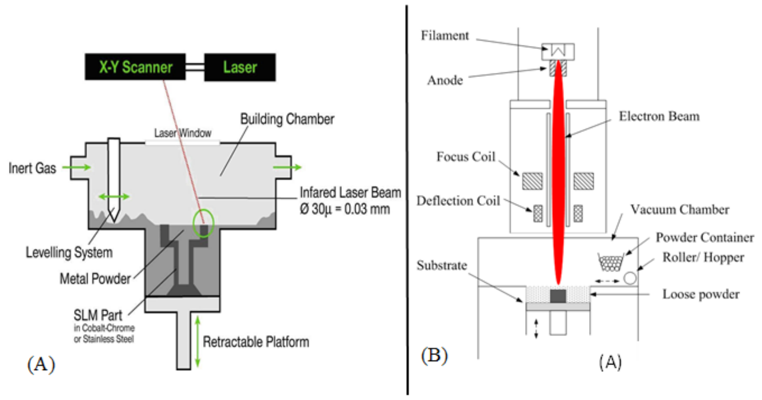

Figure 1 Schematic of metal powder bed AM processes; (A) Selective Laser Melting (Wehmoller *et al.* 2005) and (B) Electron Beam Melting

In SLM/EBM due to rapid heating and cooling of melt pools residual stresses are developed. These stresses result in consolidated layer(s) warping (bending upwards). To resist warping, supports/anchors are melted in place; anchoring layers to the substrate plate. The accumulated stress in the parts occasionally results in cracking or warping post separation from substrate plate. To reduce residual stress accumulation optimisation of processing parameters and preheating is often employed (Buchbinder D; *et al.* 2011a; Kruth *et al.* 2012). This helps to reduce the number of supports required however it does not eliminate the need for supports/anchors completely.

Semi-Solid Processing (SSP) is a novel technique developed to reduce the effect of residual stresses in part produced by metal powder bed AM processes. Researchers at University of Sheffield were able to produce parts without supports/anchors and providing freedom to produce parts which were not possible earlier as shown in Figure 2 (Mumtaz *et al.* 2011). The process began by mixing batches of custom elemental or pre-alloyed powders such that when melted together under laser or electron beam(s) would form an in-situ alloy in the powder bed. The alloy formed will have a wide solidification temperature that is lower than the respective parent materials. The complete SSP processing in SLM/ EBM was performed at elevated temperature; temperature selection was based on the solidification range of the respective in-situ alloy formed. Therefore the processed material remained in liquid-solid mushy state until the complete part was build and allowed to cool down. The selection of optimum elevated temperature resulted in minimum residual stress being induced during solidification.

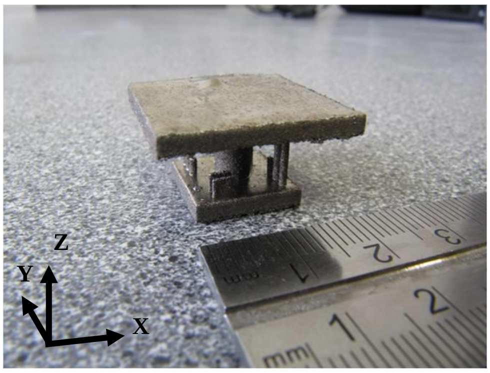

Figure 2 SLM parts produced with SSP (Material: Bi3Zn) (Mumtaz *et al.* 2011).

Several studies have looked at processing new materials, improving process capabilities and part properties. Different materials such as Aluminium alloys, titanium alloys, stainless steel, nickel alloys, and others are used to produce parts (Das 1998; Brinksmeier *et al.* 2010; Kruth *et al.* 2012; Sun *et al.* 2013). The desire to process aluminium and its alloys using metal powder bed AM processes has been well known (Louvis *et al.* 2011). Several studies have been published on processing cast and wrought aluminium alloys systems such as Al-Si, Al-Si-Mg, Al-Cu-Mg and Al-Mg-Zn (Buchbinder D. *et al.* 2011b; Louvis *et al.* 2011; Dadbakhsh *et al.* 2012). Common issues observed in processing aluminium with SLM were insufficient wetting of melt pool, high reflectively of aluminium powders, aluminium oxide causing degradation of part properties, etc. (Louvis *et al.* 2011).

Another high interest metallic alloy has been titanium and its alloys. Ti6Al4V (also known as Ti64) has been processed using SLM/EBM and widely used in aerospace and medical industry due to better mechanical properties and bio compatibility of the material (Li *et al.* 2006; Thijs *et al.* 2010). Like other metallic materials, warping in EBM is a common issue. Therefore the semi-solid processing method was tried using EBM with a custom metal powder mix. Previously attempts made to process Ti64 using EBM to benchmark unsupported overhanging features capability and unsupported features warped (Vora *et al.* 2012). A maximum of 2.7mm of warp height was measured for 5mm long and 2mm thick unsupported overhang geometry. Figure 3 shows warped geometry and corresponding warp height.

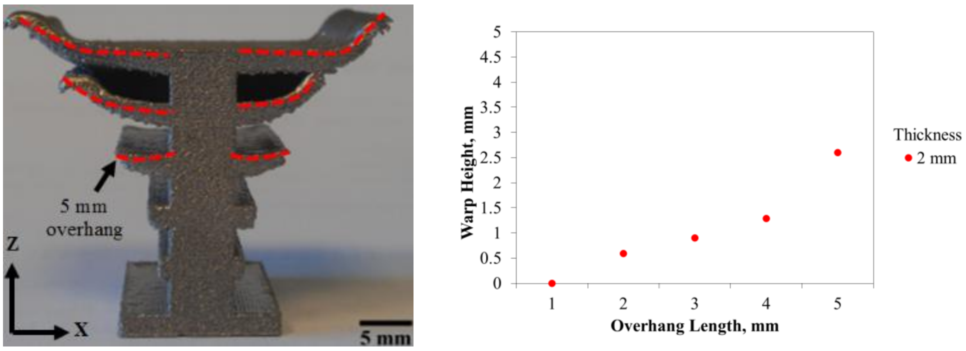

Figure 3 Benchmarking unsupported overhang capabilities with Ti64 in EBM; 2mm thick overhang geometries.

The metal powders used in AM are commercially pure or occasionally a blend of powders (metallic and/or non-metallic) whereas most are used in form of pre-alloyed powders. These pre-alloyed metallic powders were traditionally designed for conventional powder based manufacturing process or casting. However these powders are occasionally found unfavourable for AM processes due to the non-equilibrium conditions during processing (rapid solidification phenomena) in metal AM, therefore affecting part properties and/or leading to in-processing issues in AM. Therefore it is required to develop optimised alloys in powder form for metal AM processes. Custom powder blends can be mixed and in-situ alloyed using SLM/EBM. Previous studies undertaken by Bartkowiak *et al.* (2011) and Sanz-Guerrero *et al.* (2008) investigated feasibility of developing in-situ alloying of Al-Cu / Al-Zn and Ti-Cu powder systems in SLM respectively. Both the studies showed potential in developing in-situ alloys under laser. In addition to the semi-solid processing, this work investigated in-situ alloying of elemental powder blends that can be used to design optimised alloys for SLM and EBM. This would result in faster development of materials that will satisfy industry demands and also be a cost effective approach allowing simpler supply chains for many material variants.

2 Experimental Setup and Methods

2.1 Powders Processed

The purpose of the study was to understand SSP using SLM and EBM. The new developed hardware for SLM and the EBM characteristic pre-heating themes motivated to compare both processes. A direct comparison between both the processes using same material would have been ideal. However due to health and safety issues on SLM for using titanium alloys and limitation on materials allowed to process on EBM at University of Sheffield, two different material system were selected. Al-Si eutectic system was processed on SLM and Ti64-Cu system on EBM machine.

Aluminium and Silicon powders were melted to perform in-situ alloying in SLM machine. The pure aluminium powder was a gas atomised powder with near to spherical morphology. The particle size distribution was in the range was 20-45µm. Pure silicon powder obtained was not gas atomised and thus had an irregular morphology. The as purchased powder had a wide particle size distribution; therefore the range was reduced by sieving (-45µm). Both the powders were mixed in a ratio of 88% by wt. of aluminium and 12% by wt. of silicon to effectively form a eutectic alloy; AlSi12 after laser processing. Ti64 and Cu were gas atomised powders with particle size range of 50-110µm. The powders were blended in a ratio of Ti64-90% by wt. and Cu - 10% by wt.

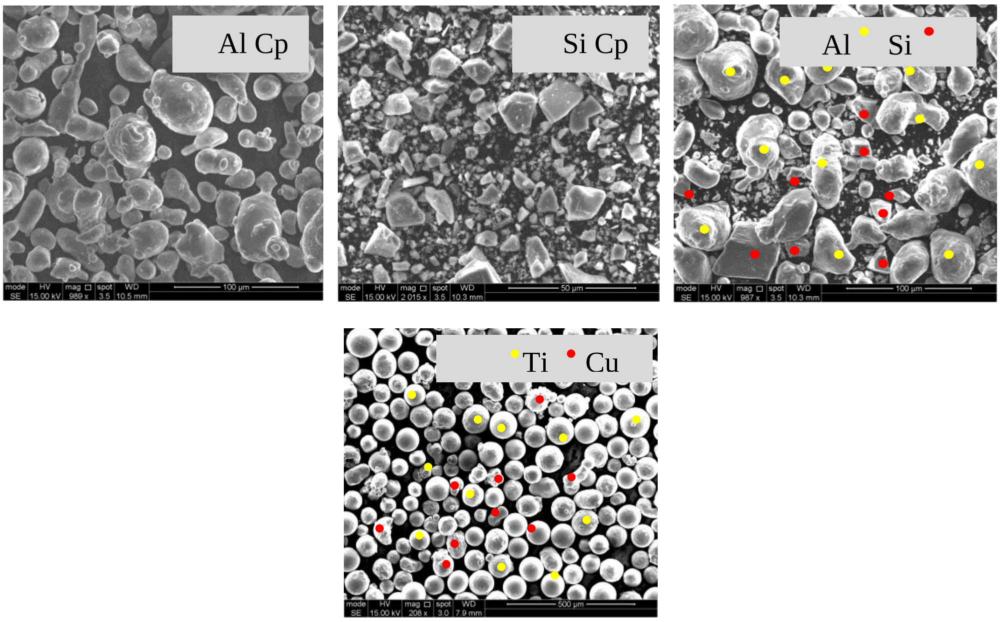

Figure 4 SEM Images of the powder processed.

2.2 Selective Laser Melting

A Renishaw SLM 125 machine was used to produce parts. The SLM 125 has a build volume of 125mm X125mm X 125mm. The laser melting process was performed under an argon atmosphere of 3-4 mbar. The system was equipped with 200W modulated yttrium- fibre laser. For SSP processing, new hardware was employed to raise the powder bed temperature during laser processing. This hardware was developed with support from the machine supplier (Renishaw plc.). The new build area of the heated bed was Ø50mm X 50mm. The new hardware was designed to reach a steady state temperature of 600°C. The hardware used bidirectional heating to the powder bed; cartridge heaters were placed below the substrate plate and a high resistive conduction heating element was installed on the side walls of the build envelope thus heating the build volume. Laser power of 200W, powder layer thickness of 50µm, and apparent scan speeds in the range of 800 mm/s, 900 mm/s and 1110 mm/s were used to build parts. The selection of preheating temperature was based on the specific alloy solidification range, in this case for Al-Si alloy the bed temperature was in the range of 350-570°C.

2.3 Electron Beam Melting

An Arcam EBM S12 machine was used to build parts. The EBM S12 has a build volume of 250mm X250mm X180mm. In this work, a beam current of 17mA, focus offset of 19mA and average beam velocity of 500 mm/s was used for volume melting. Various speed functions were used to produce parts; 30, 36 and 50. Speed function is function of beam current and beam speed for optimum process control in EBM. Parts were built on a preheated substrate plate (start plate) at 750°C and each individual powder layer was also preheated by multiple electron beams (Multibeam™) technology. For preheating, a beam current of 30mA with focus offset 50mA was used. Parts were produced with layer thickness of 70µm.

2.4 Microscopic Characterisation

Optical microscopy was performed on the samples produced. The samples were cross-sectioned and mounted in the x-z plane i.e. perpendicular to build direction in order to analyse the melt pool. After grinding with progressively finer grits, the samples were polished using 6µm and 1µm diamond suspension fluid. Prior to metallographic analysis, all the samples were etched based on standard practices recommended by ASTM standard E407-07. Non-hydrofluoric based etching agents were used for etching both Al-Si and Ti64-Cu samples.

2.5 Part design

Figure 5 shows the part geometry produced. 'T' geometries were manufactured with overhanging feature length in the range of 2-5 mm. To compare the effect of residual stresses in 'T' geometries, one side was supported and other was unsupported. Based on the previous benchmarking study (Vora *et al.* 2012), a clear visible warping was observed in geometries with 2mm thick overhangs. Therefore 2mm thick overhangs were considered for the purpose of this study.

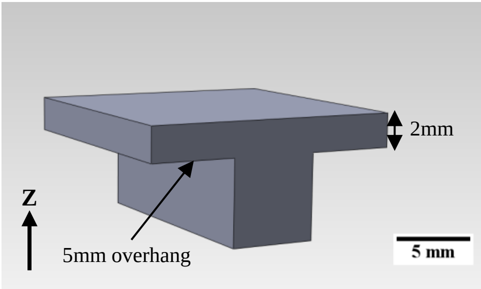

Figure 5 Supported and unsupported 'T' geometry for SSP with SLM and EBM processing.

3 Results and Discussion

3.1 Parts Produced

Figure 6 shows parts produced with SSP processing technique using SLM and EBM. This length of unsupported feature would typically warp without supports causing process failure. Processing with SSP allowed the production of complete overhanging features with reduced or negligible warp. As seen in Figure 6 (A and B) flat top surfaces of the parts were obtained.

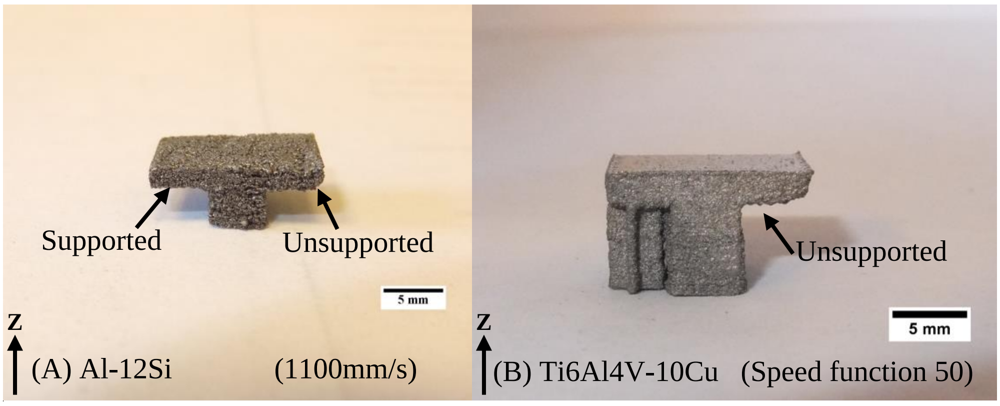

Figure 6 Parts produced by SLM and EBM- SSP processing

Due to irregular deposition of powder and poor powder flow properties of Al-Si powders, a poor surface finish was obtained on SLM parts. The irregular morphology of silicon powder further reduced flow properties of the blended powder. In parts produced by EBM, poor edge retention was observed on unsupported overhang. Buchbinder D; *et al.* (2011a) observed similar poor edge retention in 'T' geometries produced with AlSi10Mg in SLM. It was suggested this could be due to shrinkage of the first layer and then shrinkage progressively reducing over the thickness and resistance from previously consolidated layers. The higher shrinkage in first layer could be result of freedom of movement because of the absence of supports. However by employing the SSP technique, the shrinkage in layer did not cause layer to warp. This could be improved by raising the bed temperature to further reduce shrinkage and stress formation.

3.2 Metallographic Analysis

Figure 7 shows a micrograph of in-situ alloyed AlSi12 processed with SLM. A dendritic structure can be seen which is typical of aluminium-silicon system. The white structures (dendrites) are aluminium solid solution and the darker regions are Al-Si eutectics or silicon. Due to rapid solidification, SLM shows finer structures. It can be seen the dendrites in a few areas are coarse, suggesting slow cooing. However the eutectic structure seems to be reasonably uniform, suggesting successful in-situ alloying of Al-Si powders.

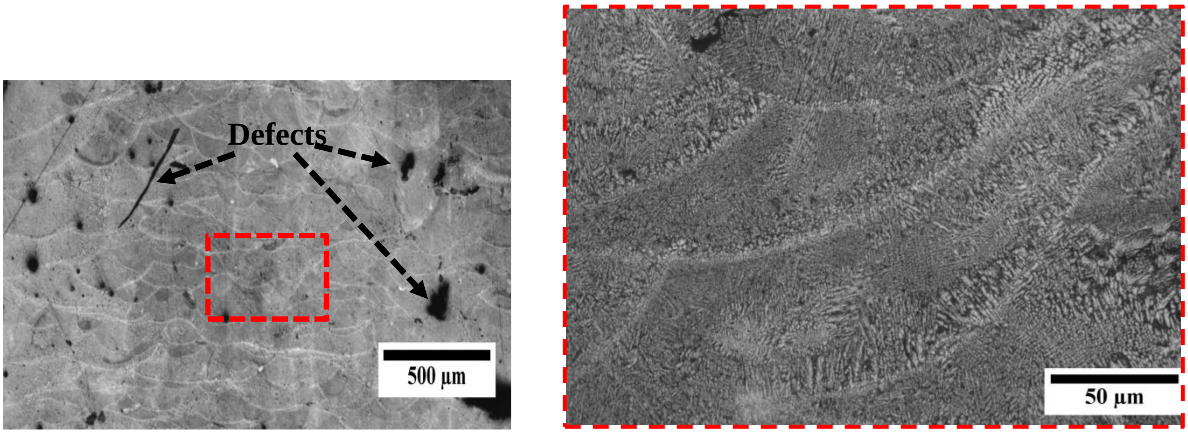

Figure 7 Microstructure of in-situ alloyed AlSi12 processed with SLM

Figure 8 shows the microstructure of Ti64-10Cu processed with EBM. It is well known that titanium undergoes allotropic phase transformation when heated up to 882°C. A similar transformed microstructure of acicular α-Ti (needle like structures) from the grain boundaries of β-Ti can be seen. A unique spread of red colour microstructure was observed. The colour suggested a Cu rich phase. Further analysis using Energy Dispersive X-ray spectroscopy (EDX) would help verify the hypothesis.

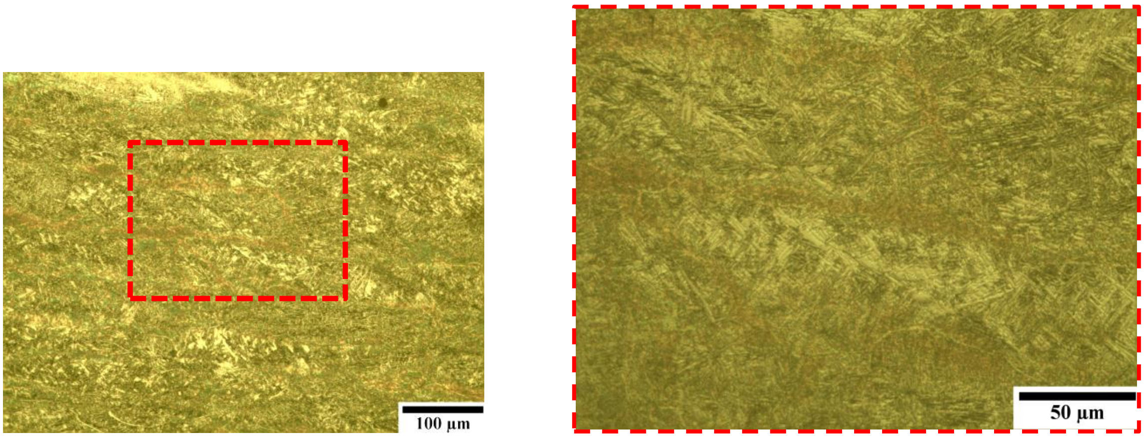

Figure 8 Microstructure of in-situ alloyed Ti64-10Cu processed with EBM

3.3 Effect of residual stress (by visual method- warp measurement)

Figure 9 shows a plot of warp measurement of in-situ alloyed AlSi12 processed by SSP technique with SLM. The parts showed no warp on the unsupported edges. The parts were built at an elevated temperature of ~380°C. The SLM parts produced at scan speed of 1110 mm/s had high part density of 91% compared to other scan speeds.

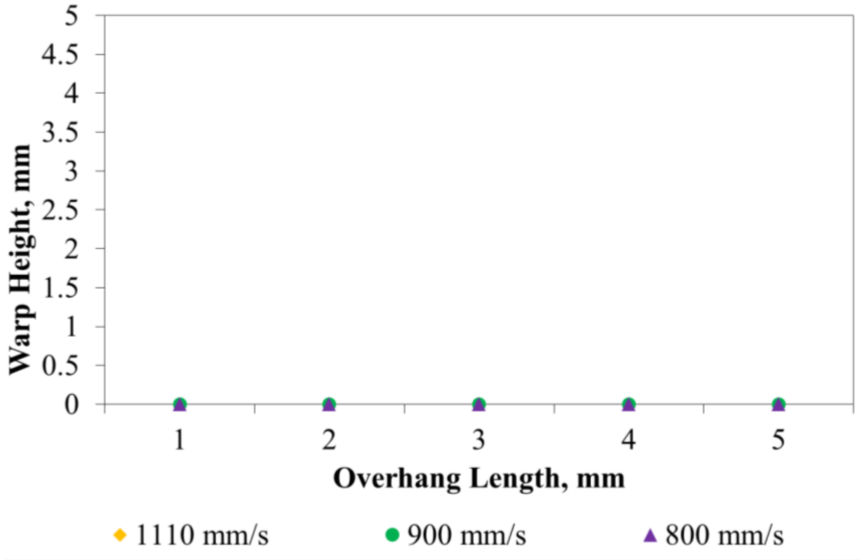

Figure 9 Warp measurement of in-situ alloyed Al-12Si samples produced by SLM

Figure 10 shows a plot of warp measurement of Ti64 and in-situ alloyed Ti64-10Cu samples produced by EBM. 2.7mm warp was measured in an unsupported overhang feature (5mm long and 2 mm thick) produced by Ti64. The SSP processed Ti64-Cu overhangs showed a maximum of warp height of 0.4 for a 5mm long unsupported feature and the minimum of 0.3mm was measured. The minimum warp height of unsupported feature was observed in part with higher scan speed.

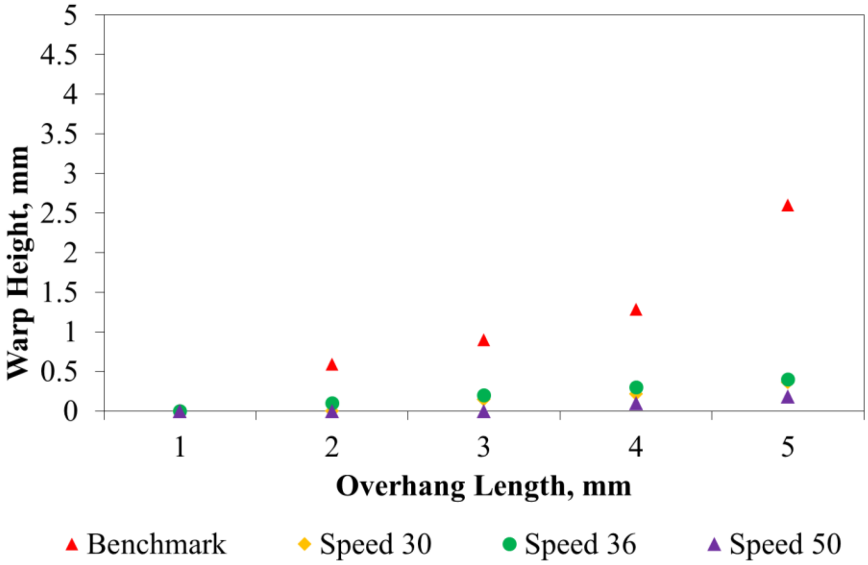

Figure 10 Warp measurement of in-situ alloyed Ti64-10Cu samples produced by EBM

The faster scan speed enabled control of the size of melt pool of the in-situ formed alloy. Compared to Ti64, the addition of copper increased the freezing range; which was also directly proportional to copper concentration. Thus the alloy exists in a liquid-solid mushy state over a wide range of temperature (Dahle *et al.* 1998) thus acting as a stress reducer and producing parts without substantially reduced warping.

4 Conclusions and Further Work

Al-Si and Ti64-Cu alloy systems were successfully processed and alloyed in-situ using Semi-Solid Processing technique within the SLM and EBM equipment. Unsupported overhang geometries of

5mm were built without warping, which we believe has never been achieved before. This work demonstrated new capabilities of SLM and EBM processes to build parts without supports using the blended powder approach. The SLM parts showed no signs of warping. Compared to benchmarking results with Ti64 on EBM, warp height was found reduced by 88% from 2.7mm to 0.3mm. Further analysis and optimisation work will be performed on parts produced. Work in areas of parameter optimisation to obtain optimum density and better surface finish, analysis on mechanical properties of parts and residual stress analysis will be conducted. This work also demonstrated new capabilities of SLM system to process material at higher temperature (up to 600°C) using the developed heated bed hardware. New materials will be designed and processed using SSP technique. In addition multi-layer parts with blended powders were manufactured. This shows the possibility of in-situ alloying of powder systems in SLM and EBM, therefore demonstrating a new approach in developing new materials quicker and optimised for metal powder bed AM processes.

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