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# Selective laser melting of Invar 36: microstructure and properties

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## Abstract

Invar 36 samples have been fabricated by selective laser melting at a constant laser power but with varied laser scanning speeds. Some samples were further heat treated or hot isostatically pressed (HIPed). The obtained microstructures were studied using optical and electron microscopes, X-ray diffraction and electron backscattered diffraction techniques and the properties evaluated through both tensile testing and thermal expansion measurement. It was found that the as-fabricated samples show very low porosity ( $<0.5\%$ ) when the laser scanning speeds are below 3200mm/s but show remarkably increased porosity above 3200mm/s (at 400W). Increased scanning speed also led to increasingly irregular-shaped laser scanned tracks together with an increased number of pores on sample surfaces and keyhole features within the samples, all indicative of increasingly unstable melt flow behaviour. The as-fabricated microstructure was dominated by columnar  $\gamma$  grains decorated by nanosized  $\alpha$  precipitates, resulting in development of texture. Heat treatment did not change microstructure significantly while HIPing closed the majority of pores but also caused pronounced coarsening of  $\alpha$  precipitates especially those located at grain boundaries during subsequent slow cooling. With the presence of elongated pores, the vertically built samples were found to show much lower elongation than horizontally built samples while in the absence of pores their ductility has been significantly improved but their tensile strengths are still lower than the latter. The vertically built samples generally failed in a transgranular mode while the horizontally built samples failed in an intergranular mode. HIPing greatly

degraded tensile properties due to the presence of coarse grain boundary  $\alpha$  precipitates weakening the bonding between grains. Irrespective of building orientations, the as-fabricated samples show low coefficients of thermal expansion below 300°C comparable to conventionally manufactured Invar 36.

**Key words:** Selective laser melting; Invar 36; microstructure; tensile behaviour; thermal expansion

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## 1. Introduction

Invar 36, Fe-36wt.%Ni alloy, is well known for its low coefficient of thermal expansion (CTE) below its Curie temperature (230°C) and excellent mechanical properties in cryogenic environment [1-5]. Due to these features, it has been widely used as highly reliable and high precision materials in components where high dimensional stability is required. The applications include space equipment, precision instruments, clocks, seismic creep gauges, television shadow-mask frames, valves in motors, liquefied natural gas storage tank and antimagnetic watches, etc. Conventionally, Invar 36 components are manufactured by machining which is expensive and difficult because the material is soft and gummy. Machining bulk Invar 36 materials into complex shapes is thus particularly challenging. Selective laser melting (SLM), due to its capacity to fabricate complex freeform geometries directly from computer-aided design (CAD) models, is considered as one of the best near-net-shape manufacturing technologies for processing metallic materials, especially for those that have difficulty with machining/tooling because it can produce shapes that require minimum machining. However, to the best of the authors' knowledge to date, the report on SLM of Invar 36 is lacking although there are a couple of reports on direct laser deposition (DLD) of Invar 36 which suggests that the residual stress could be reduced to some extent by using low CTE materials such as Invar 36 for laser deposition [6-7]. As a result, the structural integrity (defects such as porosity and cracking), microstructure, mechanical and thermal expansion properties of selectively laser melted Invar 36 are not well understood.

In this paper, we conducted a parametric study to investigate the influence of laser scanning speed on porosity and microstructural development of Invar 36 during SLM as well as the influence of post-SLM HIPing and heat treatment. The tensile properties and thermal expansion properties of the fabricated samples were also evaluated. Particularly, detailed

investigation on the influence of porosity and microstructure on fracture behaviour has been performed with the aim of better understanding anisotropic behaviour in as-SLMed samples.

## **2. Experimental**

The material used in this study is gas atomised Invar 36 powder supplied by TLS Technik in the size range of 25-50 $\mu$ m. A Concept Laser M2 Cusing SLM system which employs an Nd:YAG laser with a wavelength of 1075 nm, a maximum laser output power of 400 W and a maximum laser scanning speed of 4300mm/s has been used to prepare 10x10x10mm cubes and 70x10x10mm blocks for characterisation to understand the influence of laser scanning speed and sample size on porosity development. The samples were fabricated at a constant laser power of 400W but with different laser scanning speeds ranging from 1800mm/s up to 4300mm/s. An island scanning strategy which has been detailed elsewhere [8] was used to fabricate the current samples. All the samples were fabricated in argon atmosphere to minimise oxidation during processing.

Samples were also fabricated both vertically and horizontally for mechanical testing. The horizontal samples have a dimension of 70x10x10mm and the vertical ones are cylinders with a diameter of 10mm and a length of 70mm. Some of the SLMed samples were also heat treated and/or HIPed to study the influence of post-build heat treatment and HIPing on microstructure, mechanical and thermal expansion properties. A standard heat treatment for Invar 36 [2] has been used which includes heating of samples from room temperature up to 830°C at 5°C/min and dwelling at 830°C for 0.5 hour prior to water quenching. The samples were then heated up to 570°C and dwelt for 1 hour before air cooling down to room temperature, which was followed by a final soaking at 95°C for 48 hours. The HIPing was performed at 950°C and 100MPa for 2 hours with both ramping and cooling rates of 5°C/min.

Metallographic specimens were prepared using conventional methods and examined using optical microscopy (OM), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDX), and electron backscattered diffraction (EBSD) in a JEOL 7000 FEG-SEM microscope to reveal the size, distribution and morphology of pores together with microstructure and texture development. Tessellated micrographs each containing tens of frames were used to study the porosity distribution over large areas. The porosity was also quantified by image analysis using ImageJ. The top surfaces of the as-fabricated samples that are the result of laser melting of the final layer of powder were also investigated using SEM. To reveal microstructure, samples were etched in a solution containing 40% HCl, 25% HNO<sub>3</sub> and 35% H<sub>2</sub>O. Samples before and after SLM were also chemically analysed using a LECO TC436AR ANALYSER to study the chemical change during SLM.

Tensile tests were performed at room temperature using a computer-controlled electric screw driven Zwick/Z100 tensile testing machine on both vertically and horizontally built samples along their axis. The tests were conducted under strain control mode with a strain rate of  $1.0 \times 10^{-3} \text{ s}^{-1}$ . Tensile fracture surfaces and the longitudinal sections of the tested specimens were examined using SEM and EBSD. Thermal expansion measurement was performed on cylindrical samples with a length of 20mm and a diameter of 8mm between room temperature and 1300°C using mechanical dilatometry techniques.

### **3. Results**

#### **3.1 Powder characterisation**

Fig. 1a and 1b show the particle size distribution and cross section of as-received Invar 36 powder, respectively. Most of the powder particles show a diameter between 25µm and 50µm and a near-spherical morphology. The powder section reveals the presence of pores

within a few particles probably due to gas entrapment during atomisation. There are, however, a number of relatively darker spherical spots within each powder particle which are identified by EDX to be rich in lower atomic number element Fe but depleted in Ni (as shown in Table 1). This suggests that phase separation into bcc Fe-rich  $\alpha$  and fcc Ni-rich  $\gamma$  phases may have occurred within the powder particles during atomisation and solidification [9].

### 3.2 Porosity and surface structure development

Fig. 2 shows the variation in porosity within the as-SLMed small cubic samples (10x10x10mm) and elongated blocks (70x10x10mm) as a function of laser scanning speed at a fixed laser power (400W). It clearly shows that the porosity levels in the cubic samples are generally low when the laser scanning speeds are below 3200mm/s (<0.5% in area fraction). However, above 3200mm/s, the samples show a pronounced increase in porosity (0.7%–1.8%). Particularly, the sample fabricated at the highest scanning speed of 4000mm/s shows widespread porosity, mainly elongated pores. The elongated blocks show generally a similar trend in porosity with laser scanning speed below 3200mm/s. However, at 3200mm/s, the elongated sample was found to show much higher porosity (0.75%) than its small cubic counterpart (0.25%). A similar phenomenon was observed in our previous study where scaling-up of Ti-6Al-4V samples also resulted in an increased porosity level under a certain processing conditions [8].

Since the porosity development is believed to be associated with melt flow behaviour according to our previous work [10-11], the top surface structure of the as-fabricated samples which contains information about the melt flow traces was also investigated and the result is shown in Fig. 3. It can be seen that the elongated sample fabricated at 1000mm/s show laser scanned tracks that are evenly lined up and regularly overlapped with neighbouring tracks

whereas at 3200mm/s the tracks become increasingly irregular-shaped and even contain open pores with some obviously being due to discontinuity in the laser scanned track and the rest (cave-like pores) due to the development of overhangs or bridges above a previous layer at some localised sites. In contrast, the cubic sample that was also fabricated at 3200mm/s shows generally regular laser tracks (Fig. 3c) except for some regions where the laser scanned tracks are irregular-shaped and are associated with the development of pores (Fig. 3d). At higher laser scanning speed such as 4000mm/s, more irregular-shaped laser scanned tracks and more pores could be seen (Fig. 3e-f). These open pores are extremely harmful for porosity development within bulk samples since they are likely to remain as closed pores in the subsequent building particularly when the layer where they are located cannot be fully remelted in the following building. Given that the laser scanned tracks are the traces of melt flow during SLM, the results suggest that the melt flow has become unstable at high laser scanning speed.

### **3.3 Microstructural development**

Fig. 4 show the microstructure of the samples fabricated at 1000 mm/s and 3200 mm/s. The bottom regions (around 2 mm from the substrate) of the samples are generally dominated by large vertical columnar grains (45  $\mu\text{m}$  in width on average) which have grown through a number of layers. In contrast, in the upper regions, the grain structure is more complicated. While they still contain some large vertical columnar grains (Fig. 4b), there are also a number of fan-like clusters of grains which are much smaller (20 $\mu\text{m}$  in width) (see Fig. 4c and e). The laser scanning speed does not seem to affect the microstructure significantly but does greatly influence the porosity and crack development. In the sample fabricated at 1000mm/s, there is no obvious porosity present whereas in the sample fabricated at 3200mm/s a number of elongated pores that are mainly located at the inter-layer boundaries could be observed (as indicated by the arrows shown in Fig. 4e), consistent with the above porosity observation and



measurement on as-polished samples. Moreover, it is noted that there are a number of large keyhole weld beads which have penetrated down through several previous layers throughout the samples fabricated at or above 3200mm/s (Fig. 4e-f). These weld beads consist of fan-like columnar grains with some being associated with cracking along their central lines and with some spherical pores that could be attributed to keyhole pores.

Fig. 5 shows the SEM micrographs of detailed microstructural features in as-SLMed sample. A typical fan-like grain cluster was found to generally contain columnar grains at the lower side and some equiaxed grains on the upper side of a weld bead (Fig. 5a-b). In many other weld beads, columnar grains were found to have grown epitaxially beyond several layers (Fig. 5c-d). At the sites of pores, very fine equiaxed grains were developed on the top region of the pores upon which large columnar grains were further formed (Fig. 5e).

EBSD was further performed to study the microstructure and texture development of as-SLMed samples. The results are shown in Fig. 6 and further confirm that the microstructure is dominated by columnar grains. Texture could be observed near  $\langle 001 \rangle$ . The as-fabricated samples were also found to be dominated by  $\gamma$  phase (around 90% area fraction) decorated by  $\alpha$  precipitates (around 5% in area fraction); see Fig. 6d.

Fig. 7 shows the microstructure of SLMed samples after HIPing and/or heat treatment. There is no obvious change in grain size and structure after HIPing and/or heat treatment. All the samples are dominated by a mixture of vertical columnar grains and fan-type columnar grains together with a small number of equiaxed grains. The samples that have been heat treated were found to occasionally show cracking at the interfaces between some neighbouring grains, particularly at the interface of two layers of grains (see Fig. 7e-f). The cracking is believed to be formed due to the internal stress developed during water quenching after the heat treatment at 830°C. HIPing obviously has closed the majority of porosity present in the

sample fabricated at 3200mm/s (Fig. 7c). XRD analysis was also performed and the results suggest that the current HIPing and heat treatment conditions do not seem to affect microstructure significantly. All the samples analysed are dominated by  $\gamma$ -FeNi phase.  $\alpha$  phase is not detected by the current XRD analysis, suggesting that the volume fraction of  $\alpha$  phase is around or below 5% which is the current XRD resolution limit, consistent with the above EBSD analysis. The only obvious microstructural difference, however, was found by high magnification backscattered electron SEM imaging where the samples that have been HIPed show much coarser  $\alpha$  precipitates than the rest of the samples (Fig. 8). The  $\alpha$  precipitates in the SLMed+HIPed samples show an average diameter of 330nm whereas those in the as-SLMed samples have an average diameter of only 75nm. Heat treatment leads to coarsening of some of the precipitates but the majority remain almost unchanged, giving rise to an average diameter of 110nm. Moreover, the coarse precipitates within the samples that have been HIPed were found to be mainly located along grain boundaries while the rest of the samples show generally a homogeneous distribution of  $\alpha$  precipitates throughout them.

### **3.4 Tensile behaviour**

Fig. 9-10 and Table 2 show the tensile testing results for Invar 36 samples fabricated at 3200mm/s and 1000mm/s, respectively, two conditions that yield different porosity levels. In terms of the samples fabricated at the higher scanning speed which contain a high porosity level, the horizontally built samples were found to exhibit not only superior tensile strengths (both 0.2% yield strength (YS) and ultimate tensile strength (UTS)) but also much better elongations as compared with the vertically built samples, resulting in a pronounced anisotropy in tensile properties. The vertically built samples also show a great scatter in tensile strengths and elongation with some of the samples even failing prior to yielding. The horizontally built samples generally show a slant fracture feature with a certain necking whereas the vertically built samples tend to show flat fracture surfaces with no obvious

necking (see Fig. 9b). HIPing that has closed the majority of pores present in the samples, however, did not lead to improvement of tensile properties as expected but instead even degraded the properties. The SLMed+HIPed samples all failed without developing any necking indicating a brittle fracture.

As for the samples fabricated at 1000mm/s which contain low porosity, the horizontally built samples again show excellent tensile strengths and ductility but see no obvious improvement as compared with their counterparts that were fabricated at high laser scanning speed. The vertically built samples, however, show significant improvement in ductility and consistency of tensile properties at the absence of elongated pores. Their elongations are even better than the horizontally built samples although the strengths are still lower. Heat treatment was found not to cause significant change to the tensile properties although it led to development of some cracking in the samples as shown in Fig. 7b. All the tested specimens fabricated at 1000mm/s show necking to a varied extent (Fig. 10b).

To elucidate the origin for the difference in tensile properties among different samples, the fracture surfaces of the tensile tested specimens have been investigated and the results are shown in Fig. 11 and Fig. 12. From Fig. 11 which shows the fracture surfaces of samples fabricated at 3200mm/s, one can see that the as-fabricated samples built along different directions show very different fracture features. The vertically built samples actually show a number of large opened-up pores on their fracture surfaces. These pores show very smooth surfaces and are usually associated with un-melted or partially melted powder particles, suggesting that they were formed due to insufficient remelting of previous layer and incomplete melting of some powder particles [12-13]. The horizontally built samples, however, do not show widely opened-up pores but instead show relatively elongated and squeezed pores. The difference in the fracture feature between the samples built along different orientations is believed to be due to the difference in loading mode, i.e., the loading

direction relative to the orientation of the planar lack-of-fusion pores (could be considered as cracks). Thus, in the case of vertically built samples, the loading direction is normal to the orientation of the pores, which corresponds to the opening or tensile mode (Mode I) where pore surfaces move directly apart whereas in terms of the horizontally built samples, the loading direction is nearly parallel to the planar pores corresponding to the sliding or in-plane shear mode (Mode II) where the pore surfaces slide over one another in a direction that is parallel to the orientation of the elongated pores [14]. The opening/tensile mode obviously leads to earlier failure as compared with the in-plane shear mode.

In contrast, the samples fabricated at 1000mm/s, irrespective of building directions, all show almost no open pores on the fracture surfaces but instead show fairly ductile fracture mode characterised by the presence of massive fine dimples associated with fine  $\alpha$  precipitates; see Fig. 12a-b. Heat treatment shows no obvious change to the ductile fracture mode of the samples (Fig. 12c), consistent with the observation of their insignificant influence on tensile properties. HIPing, however, is shown to show faceted fracture surfaces associated with separation of materials along large  $\alpha$  particles without development of ductile dimples, characteristic of highly brittle fracture nature. The longitudinal sections of the tested specimens were further investigated by SEM and EBSD and the results are shown in Fig. 13 and Fig. 14, respectively. It is clear that in the as-fabricated samples which contain low porosity the longitudinal sections are generally dense with secondary cracks occasionally observed at the interface between two layers in the horizontally built sample (see Fig. 14b) whereas for the sample that has been HIPed a number of secondary cracks could be observed mainly associated with separation of materials along large  $\alpha$  precipitates at GBs consistent with the observation on fracture surfaces. Moreover, it is noted from Fig. 14 that the vertically built samples generally failed in a transgranular mode with columnar grains being sheared in a slant angle whereas the horizontally built samples failed in an intergranular

mode. The columnar grains in the horizontally built samples which were nearly normal to the sample axis (or loading direction/Z) have been found to be bent to such an extent (probably due to the necking) that they become parallel to the slant fracture surface prior to intergranular fracture during tensile testing (Fig. 14b).

### **3.5 Chemical analysis and thermal expansion behaviour**

Since the coefficient of thermal expansion of Fe-Ni alloy system is highly sensitive to the fraction of Ni, with 36wt.% Ni content giving the lowest coefficient of thermal expansion [11], the chemical control during SLM is significant for achieving low CTE. Thus, in the present work, chemical analysis has been conducted on Invar 36 before and after SLM to investigate the chemical change. The results are shown in Table 3. It is clear that Ni content has almost remained unchanged during SLM, suggesting that SLM is a reliable process to fabricate Invar 36 samples without causing significant chemical change. The oxygen pickup is also very limited, thanks to the argon protective atmosphere.

Thermal expansion measurement was further performed on as-SLMed and SLMed+heat treated Invar 36 and the results are shown in Fig. 15. It is obvious that all the three samples generally show a similar trend in the change of sample length with temperature, all showing very small displacement below 300°C but exhibiting a dramatic increase of displacement when temperature is above 300°C. Despite the similarity in the trend of displacement with temperature, the three samples show some difference in total displacement at a specific temperature. Particularly, the horizontally built sample shows minimum displacement below 300°C while the vertically built sample shows the maximum displacement. In addition, the sample displacement with temperature within 100°C-200°C was found to be nearly linear, giving rise to a slope (which corresponds to the CTE) of  $2.0 \times 10^{-6} \text{ K}^{-1}$ . According to Fig. 15b, the slope/CTE between 22°C and 100°C is even lower than  $2.0 \times 10^{-6} \text{ K}^{-1}$  while the CTE

between 200°C and 300 °C is slightly higher. Assuming that the thermal expansion below 300°C is nearly linear, then the approximate CTE below 300°C is  $3.5 \times 10^{-6} \text{ K}^{-1}$  which is still much lower than that above 300°C which is  $2.0 \times 10^{-5} \text{ K}^{-1}$  (almost one order of magnitude higher). The CTEs of the current samples are comparable to those of conventionally manufactured (cast+forged) Invar 36 which usually show a CTE around  $2.0 \times 10^{-6} \text{ K}^{-1}$  below 300°C [2].

## **4. Discussion**

### **4.1 Porosity development**

The current experimental results demonstrate that Invar 36 has a wide processing window for acquisition of low porosity when laser power is high during SLM. However, when laser scanning speed is above a certain value (in the current case 3200mm/s) the porosity could be greatly increased. The porosity development is believed to be associated with the change of melt flow behaviour as evidenced by the observation on top surface structure. Thus, with increasingly irregular-shaped laser scanned tracks indicative of more unstable melt flow, the internal porosity level is higher. Laser scanning speed was found to greatly affect the surface structure and porosity development with high laser scanning speeds generally leading to more irregular-shaped laser scanned tracks and more pores being developed on both sample surfaces and within the samples. A similar phenomenon was observed in our previous work on SLM of Ti-6Al-4V where increase of laser scanning speed increased flow velocity and instability of the melt pool [10-11]. Unstable melt pool tends to lift up material away from the build surfaces rather than spread steadily along laser scanning direction [10], which is liable for development of cave-like pores (Fig. 3b, d and f) and elongated pores at the interfaces between layers as observed in Fig. 4e. Moreover, with a constant laser power but increased laser scanning speed, the input energy density would become smaller and it is more likely that the previous layer is not sufficiently remelted and bonded with the fresh layer, which is

evidenced by the observation on fracture surfaces of the samples fabricated at 3200mm/s where un-melted or incompletely melted powder particles could be observed (Fig.5e and Fig. 11). The dependence of porosity on sample size at a high laser scanning speed (3200mm/s) is believed to be due to the different thermal histories they have gone through. The smaller cubic samples contain smaller cross sections as compared with the elongated blocks and thus would be hotter during processing because the time for laser beam to come back to the same locations will be shorter. The hot surfaces may be beneficial for the wetting and spreading of molten material and thus for suppressing development of open pores such as cave-like pores on the top surfaces which are believed to be extremely harmful for porosity development within bulk samples. This is supported by the observation of smoother laser scanned tracks on the top surface of the smaller sample fabricated at the same laser scanning speed (see Fig. 3c).

Although the majority of pores present in the samples fabricated at high laser scanning speeds show elongated morphology, there are also some large spherical pores present (around 20 $\mu$ m in diameter) which are obviously due to keyhole welding (Fig. 4e). The increased number of keyhole welding (which is usually the result of violent interaction between heat source and melt pool) with increased laser scanning speed again suggests that the laser-melt interaction and melt flow behaviour have become increasingly violent and unstable. Cracking development along the central lines of the keyhole welds was also observed in the sample fabricated at high laser scanning speed, which is believed to be the result of shrinkage during solidification and cooling of keyhole welding melt pools. The central line happens to be the boundary of two sets of columnar grains on two sides of a keyhole weld bead and could be a vulnerable site for stress concentration and material separation.

## 4.2 Microstructural development

The current experimental results demonstrate that the as-SLMed microstructure is generally dominated by columnar  $\gamma$  grains with the bottom region containing large vertical columnar grains that grow along the building direction while the upper region consists of a mixture of vertical columnar grains and fine fan-like columnar grains constrained within weld beads. The microstructural development is believed to be associated with the thermal history that different regions may have experienced. Thus, the cooling rate at the bottom region is generally faster than those at the upper region since it is closer to the cold substrate, which is favourable for development of large columnar grains. The hotter upper region shows smaller thermal gradient which is thermodynamically unfavourable for growth of columnar grains beyond several layers vertically. It also makes the grain development more localised like those constrained within weld beads (Fig. 5b). Due to the presence of columnar grains, texture has been developed around  $\langle 001 \rangle$  (Fig. 6b and c). A high population of very fine  $\alpha$  precipitates were also present in the as-SLMed samples. Their size is even smaller than 100nm in diameter, probably due to the generally rapid cooling associated with the SLM process.

Post-SLM heat treatment was found not to change the microstructure significantly except for the development of some cracking throughout the samples probably due to the involvement of water quenching. The microstructure still consists of  $\gamma$  grains and nanosized  $\alpha$  precipitates. HIPing closed the majority of pores but led to coarsening of  $\alpha$  precipitates especially those at GBs. The difference in the effect of heat treatment and HIPing on  $\alpha$  precipitate size is unlikely to be due to the difference in the temperatures used for heat treatments (830°C and 570°C) and HIPing (950°C) given that the  $\gamma \rightarrow \alpha$  transformation temperature is actually below 500°C [4]. Instead, it should be attributed to the difference in cooling rate after heat treatments and HIPing. The former experienced rapid cooling (either water quenching or air



cooling) while the latter underwent very slow furnace cooling (around 5°/min). The growth of  $\alpha$  precipitates may have been suppressed by rapid cooling but favoured by slow cooling. The size and distribution of  $\alpha$  precipitates were found to influence tensile properties, which will be discussed in the following section.

#### **4.3 Tensile properties and fracture behaviour**

The current work suggests that with the presence of a high population of porosity (Fig. 2e and Fig. 11), the mechanical anisotropy especially in elongation will be significant. The vertically built samples show much lower elongation than the horizontally built samples due to that the tensile loading on them is easier to lead to tearing of the elongated pores present in the samples and thus to earlier failure. As for the samples that contain minimum porosity, the vertically built samples show significant improvement in elongation and consistency of tensile properties, further confirming the harmful effect of porosity on plasticity of vertically built samples. The horizontally built samples, however, are much less sensitive to the presence of elongated pores as the samples containing a number of pores were found to show tensile properties that are comparable to those containing minimum porosity. The results suggest that it is the loading mode (i.e., the loading direction relative to the orientation of defects such as pores/cracks) that really matters to the ductility of as-SLMed samples as long as they contain elongated pores or cracks.

In the absence of elongated pores like the samples fabricated at 1000mm/s, the as-fabricated samples still demonstrate anisotropy in both tensile strengths and elongation (see Fig. 10a). This is believed to be associated with the presence of texture and the difference in loading direction relative to the columnar grain orientation/texture within vertically and horizontally built samples. The observation on the fracture modes of the two types of samples revealed that the vertically built samples failed in a transgranular fracture mode with columnar grains

being sheared in a slant angle while the horizontally built samples failed in an intergranular mode involving rotation and bending of columnar grains prior to fracture (Fig. 14). The former suggests relatively easier deformation than the latter and thus leads to better ductility. However, detailed investigation on the deformation mechanisms of these samples is required to fully understand the anisotropy due to texture.

The post-SLM heat treatment was found not to cause significant influence on tensile properties obviously due to their insignificant influence on microstructure. HIPing, however, led to serious degradation in both tensile strengths and plasticity due to the coarsening of  $\alpha$  precipitates (mainly along GBs) which may have weakened the bond between grains, although it closed the majority of pores.

#### **4.4 Thermal expansion properties**

The magnitudes of thermal expansion for the samples investigated were found to be comparable to each other, probably due to the fact that the compositions of the samples after SLM remain almost unchanged. The samples fabricated before and after SLM all contain around 36 wt.% Ni which is essential for acquisition of low CTE. Nonetheless, there is still small difference in absolute thermal expansion displacement at each temperature among different samples. Particularly, at low temperature regime (below 300°C) which is crucial for application of this material, the horizontally built samples demonstrate smaller displacement than the vertically built samples at each specific temperature, which could be attributed to the fact that the horizontally built samples show higher tensile strengths than the vertically built samples. Heat treatment improves the thermal expansion property at low temperatures but not significantly.

## **5. Conclusions**

1. At a high laser power, Invar 36 has a wide processing window during SLM to achieve significantly low porosity ( $<0.5\%$ ).
2. At a constant high laser power, increased laser scanning speed is liable for development of increasingly irregular-shaped surface structure, increased porosity on sample surfaces and within samples as well as increased keyhole pores and cracking.
3. The as-fabricated microstructure of Invar 36 was dominated by columnar  $\gamma$  grains decorated by  $\alpha$  precipitates, leading to development of texture.
4. The presence of elongated pores is particularly harmful for the ductility of vertically built samples due to the tensile/opening loading mode incurred.
5. In the absence of elongated pores, the vertically built samples failed by shearing through columnar grains gaining significant improvement in ductility while the horizontally built samples failed in an intergranular mode with higher strengths but lower elongations.
6. HIPing followed by slow cooling led to coarsening of  $\alpha$  precipitates at GBs which weakened the bonding between grains, resulting in significant degradation in tensile properties.
7. The selectively laser melted samples show low coefficient of thermal expansion below  $300^{\circ}\text{C}$  that is comparable to conventionally manufactured Invar 36.

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