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Selective laser melting of Invar 36: Microstructure and properties

Chunlei Qiu *, Nicholas J.E. Adkins, Moataz M. Attallah **

School of Metallurgy and Materials, University of Birmingham, Edgbaston, Birmingham B15 2TT, UK

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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 backscatter 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 3200 mm/s but show remarkably increased porosity above 3200 mm/s (at 400 W). 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 γ grains decorated by nanosized α 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 α 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 α 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.

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

* Corresponding author.
** Corresponding author.
E-mail addresses: c.qiu@bham.ac.uk (C. Qiu), m.m.attallah@bham.ac.uk (M.M. Attallah).

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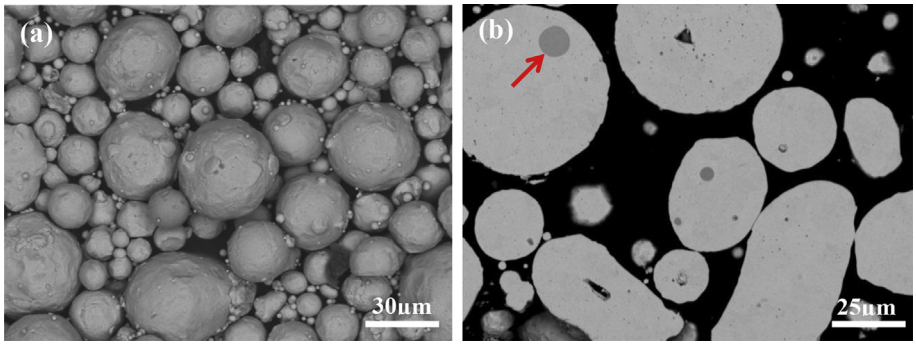

Fig. 1. Backscattered electron SEM micrographs of (a) as-received Invar 36 powder particles and (b) their cross section. The arrow shows the Fe-rich regions.

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 μm. 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 4300 mm/s has been used to prepare 10 × 10 × 10 mm cubes and 70 × 10 × 10 mm 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 400 W but with different laser scanning speeds ranging from 1800 mm/s up to 4300 mm/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 70 × 10 × 10 mm and the vertical ones are cylinders with a diameter of 10 mm and a length of 70 mm. 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 h prior to water quenching. The samples were then heated up to 570 °C and dwelt for 1 h before air cooling down to room temperature, which was followed by a final soaking at 95 °C for 48 h. The HIPing was performed at 950 °C and 100 MPa for 2 h 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 backscatter 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_3 and 35% H_2O. 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 1.0×10^{-3} 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 20 mm and a diameter of 8 mm between room temperature and 1300 °C using mechanical dilatometry techniques.

3. Results

3.1. Powder characterisation

Fig. 1a and b shows the particle size distribution and cross section of as-received Invar 36 powder, respectively. Most of the powder particles show a diameter between 25 μm and 50 μm 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 α and fcc Ni-rich γ 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 (10 × 10 × 10 mm) and elongated blocks (70 × 10 × 10 mm) as a function of laser scanning speed at a fixed laser power (400 W). It clearly shows that the porosity levels in the cubic samples are generally low when the laser scanning speeds are below 3200 mm/s (<0.5% in area fraction). However, above 3200 mm/s, the samples show a pronounced increase in porosity (0.7%–1.8%). Particularly, the sample fabricated at the highest scanning speed of 4000 mm/s shows widespread porosity, mainly elongated pores. The elongated blocks show generally a similar trend

Table 1
EDX analysis on the matrix regions and dark spots in the as-received powder particle sections.

At.%	Matrix-1	Matrix-2	Matrix-3	Dark spot-1	Dark spot-2
Fe	64.87	65.36	65.29	94.58	96.64
Co	0.63	0.59	0.63	0.52	0.56
Ni	34.50	34.06	34.08	3.64	1.58
Mn	–	–	–	0.76	1.22
Cr	–	–	–	0.49	–