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# Micro porosity analysis in additive manufactured NiTi parts using micro computed tomography and electron microscopy

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ABSTRACT

Long-term fixation of biomedical implants is achievable by using porous materials. These kinds of materials can produce a stable bone-implant interface. A critical aspect in production of porous implants is the design of macro and micro pores. In this research, a micro direct metal deposition process, newly developed as a potential method for implant production, was used to fabricate porous NiTi parts. The effect of process parameters on formation and distribution of micro pores was analysed using micro computed tomography and scanning electron microscopy. The analysis showed that, by increasing the laser scanning speed, inherent micro porosity increases. Also, it was found that there is an optimum temperature to achieve minimum inherent micro porosity by micro direct metal deposition which is 300 °C for NiTi powder.



## 1. Introduction

Porous metallic implants are promising due to their inherent low elastic modulus which is close to the stiffness of real bone. Moreover, porous structure promotes osseointegration in fabricated implants [1]. Generally, orthopaedic porous scaffolds should mimic the morphology, properties and microstructure of real bone which includes micro (<20 μm) and macro (>100 μm) pores [2]. Different methods exist for porous materials fabrication. Some methods such as casting, vapour deposition and especially additive manufacturing, allow a greater control over pore size and distribution. By these methods, open-cell porous structures are possible to be fabricated. On the other hand, there are other methods such as decomposition of foaming agents in either molten or powder metal matrices, which allow lower control over the characteristics of pores. Using these methods result in close-cell porous structures [3]. Many works have been conducted on production of porous scaffolds with macro pores using additive manufacturing methods such as selective laser melting (SLM) [4,5] and laser engineered net shaping (LENS) [6,7]. In additive technologies, macro pores are possible to implement using computer-aided design (CAD). In addition, two different categories of micro pores can be obtained in laser melted parts. Melting process can cause the formation of the first category of micro pores. We use the term *inherent micro porosity* for the micro pores that are formed by melting process. This kind of porosity is non-controlled and substantial. Also, we use the term *controllable micro porosity* for pre-defined micro pores which can be manipulated by change in hatch distances or strategy of scanning as two main important process parameters in laser melting. Micro direct metal deposition (μDMD) is an additive manufacturing technique that produces parts with micro features from metal powders, using direct metal deposition (DMD) concept [8]. μDMD uses low energy laser beam to promote melting in an inert environment within a chamber. Orthopaedic implants with inherent and controllable micro porosities can be fabricated by μDMD due to the specific process parameters of this technique. Control over the architecture of micro pores is essential for porous scaffolds to serve as a bone implant [9,10]. An evolution from random distribution of micro pores to pre designed porosity based on specific process parameters is important to control the functional behaviour of porous scaffolds. Regular architecture of micro pores permits cells to be seeded in the core much more readily than random architecture scaffolds [11]. Porosity, spatial distribution and morphology of micro pores have impact on mechanical and biological properties of porous implants. Design of micro pores inside the materials for biomedical application is still under research. It is possible to control micro pore's percentage by adjusting of process parameters. Despite there is no direct control on inherent micro pore's shape, it is possible to indirectly control the predefined micro pore's shape by changing process parameters such

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as hatch distance and scanning strategy as demonstrated in this research. In this paper, the effects of μDMD process parameters in obtaining fully dense NiTi were investigated. Moreover, the effects of μDMD process parameters on porosity, micro pore's shape and distribution were analysed.

## 2. Materials and methods

Mechanically alloyed nickel–titanium powder produced by MBN Nanomaterialia (Italy) with a Ni:Ti ratio of 50.8:49.2 (atomic %) was used in this research as a starting material. Characteristics of the powder are given in Table 1 and described in more details elsewhere [8]. MSL50 micro laser sintering machine (Manudirect Company, Italy) with a continuous fibre laser (YLM-100-WC, IPG, wavelength: 1070 nm), maximum power of 100 W and 30 μm minimum laser spot diameter was used to implement single tracks, thin walls and small cubes. A schematic representation of the system is shown in Fig. 1. Using a heating stage and Argon filled chamber, it is possible to increase and control the temperature of parts during the process. Fabrication of thin walls with different process parameters is the easiest way to investigate the effect of input energy on inherent micro pore's morphology and distribution in laser melted parts. To achieve the thickness of each layer produced by a single laser scan, a series of single tracks were processed using different scanning speeds (V), powder feeding rates ($RF_n$) and substrate temperatures (T) as shown in Table 2 and Fig. 2(a). The heights of single tracks were measured using a state-of-the-art 3D optical profiler (Sensofar Plu Neox, SENSOFAR-TECH, SL, Spain) operating in confocal mode [12] by calculating of arithmetic mean values of heights from horizontal sections on each single track (Fig. 2(b)).

288 thin walls were produced according to Table 3. The overlap was considered constant for all successive layers in each wall and was driven from Eq. (1).

$$\text{Overlap\%} = \frac{\text{Overlap length}}{\text{Layer thickness}} \times 100\% \quad (1)$$

Fig. 3 schematically shows the overlap between the successive layers. The heights of thin walls were measured by 3D optical profilometry, using the same procedure of single tracks.

Micro X-ray computed tomography (μCT) is a three-dimensional (3D) non-destructive imaging technique that allows the investigation of internal and external structures of parts and small features, with micrometric resolution [13,14]. In this research, μCT is used to analyse the porosity in 3D volume of laser melted parts. Such information can be used to improve the μDMD process; for example, to minimize porosity by varying μDMD parameters. μCT can also be used for quality control in laser melting process. The samples were scanned using a metrological μCT system (Nikon X-Tek MCT225, Nikon Metrology/X-Tek Systems Ltd., UK) with 225 kV micro-focus X-ray source, 3 μm focal spot resolution, flat panel detector with 2000 × 2000 pixels (16 bit), and temperature-controlled cabinet. The samples were placed on a rotating stage and a number of two-dimensional (2D) X-ray projections were acquired at various angular positions. Such projections were used to reconstruct a 3D model, by means of a filtered back-projection algorithm. The used CT scanning parameters are listed in Table 4. Distribution and volume of internal pores were analysed using commercial volume imaging software (VG Studio MAX, Volume Graphics GmbH, Germany). The porosity percentage related to the central part of each wall (shown in red in Fig. 4) was provided by calculating the total volume of pores against the total selected volume.

**Table 1**
Characteristics of initial powder.

| | Nickel (%) | Titanium (%) | Particle size [μm] |
|---|---|---|---|
| NiTi powder | 50.8 | 49.2 | 28–35 |

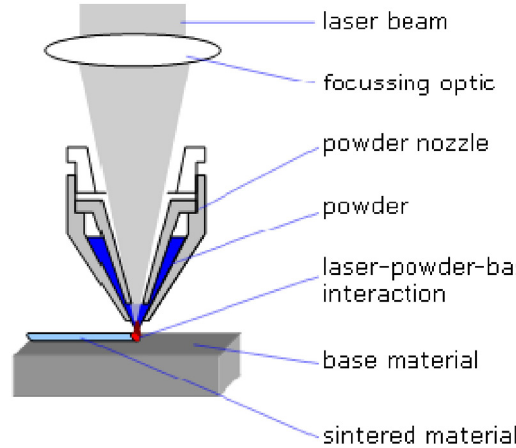


**Fig. 1.** Schematic representation of micro direct metal deposition system.

Controllable micro pores were implemented by varying μDMD process parameters. To analyse the effect of scanning strategy on controllable micro pore's morphology and distribution, different strategies based on the angles between tracks ($\theta_T$) and layers ($\theta_L$) were examined in this research to produce NiTi cubes. According to Table 5, five different strategies were employed to produce 5 NiTi cubes on titanium substrates (Fig. 5). Shape, sizes and distribution of micro pores in NiTi cubes were analysed using a scanning electron microscope (SEM) (Quanta 450, FEI). Porosity measurements were conducted on polished specimens, by performing image analysis of SEM micrographs. Also, the effect of hatch distance ($d_H$) on micro pore's morphology was investigated using this technique. To investigate the surface quality of laser melted NiTi cubes, roughness was measured using 3D optical profilometry.

## 3. Results and discussion

### 3.1. The effects of μDMD process parameters on geometry and inherent micro porosity of thin walls

In this chapter the effects of layer overlap, scanning speed and substrate temperature on the height of single tracks and thin walls are presented. Moreover, the effects of these parameters on quantity and distribution of inherent micro pores are described. Total input energy produced by μDMD can be calculated using Eq. (2). [15]:

$$E_L = \frac{P}{V \times \Delta Z \times d} \quad (2)$$

where $P$ is power of laser, $d$ is diameter of laser focus spot; V is the scanning speed and ΔZ is the pre-defined thickness of each layer. Increase in layer thickness and scanning speed as main μDMD process parameters results in decrease of input energy in laser melting according to Eq. (2). Low input energy causes discontinuous single tracks and inhibits the formation of 3D parts. On the other hand, at very high level of input energy, the temperature of melt pool

**Table 2**
μDMD process parameters used to produce NiTi single tracks.

| Laser power (W) | Scan speed (mm/min) | Powder feeding rate (mg/min) | Substrate temperature (°C) | Internal energy density (kJ/mm$^2$) |
|---|---|---|---|---|
| 30 | 70, 100, 150, 200 | 0.76, 1.26 | 200, 300, 400 | 0.3, 0.4, 0.6, 0.85 |