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## Research Paper

# Compression fatigue behavior of laser processed porous NiTi alloy

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#### ABSTRACT

Porous metals are being widely used in load bearing implant applications with an aim to increase osseointegration and also to reduce stress shielding. However, fatigue performance of porous metals is extremely important to ensure long-term implant stability, because porous metals are sensitive to crack propagation even at low stresses especially under cyclic loading conditions. Herein we report high-cycle compression–compression fatigue behavior of laser processed NiTi alloy with varying porosities between  $\sim$ 1% and 20%. The results show that compression fatigue of porous NiTi alloy samples is in part similar to metal foams. The applied stress amplitude is found to have strong influence on the accumulated strain and cyclic stability. The critical stress amplitudes associated with rapid strain accumulation in porous NiTi alloy samples, with varying relative densities, were found to correspond to 140% of respective 0.2% proof strength indicating that these samples can sustain cyclic compression fatigue stresses up to 1.4 times their yield strength without failure.

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#### 1. Introduction

NiTi alloys (Nitinol) are gaining importance due to their biocompatibility, shape memory effect and superelasticity (Shabalovskaya, 2002). These alloys also exhibit elastic moduli that closely match with that of human cortical bone (20 GPa) in the martensitic condition (28–41 GPa) (Shabalovskaya, 2002; Gyunter et al., 1995). Close matching of elastic modulus between load bearing metal implants and the living tissues can eliminate implant loosening following stress shielding of the bone (Robertson et al., 1976; Cameron et al., 1978; Head et al., 1995). Further, by introducing optimal porosity in Nitinol alloys the mechanical properties, cellular adhesion and growth

can be tailored (Bansiddhi et al., 2008; Balla et al., 2009, 2010). The rough surface morphology of porous implants further promotes bone tissue ingrowth into the pores (Xue et al., 2007; Bandyopadhyay et al., 2010) and provides anchorage for biological fixation (Xue et al., 2007; Bandyopadhyay et al., 2010; Schneider et al., 1989; Pillar, 1987; Clemow et al., 1981) ensuring uniform stress transfer between the implant and the bone, which has the potential to improve long-term stability (Bandyopadhyay et al., 2010; Schneider et al., 1989; Pillar, 1987; Clemow et al., 1981). Thus porous NiTi alloys are promising biomaterials for hard tissue replacements (Shabalovskaya, 2002; Gyunter et al., 1995; Bansiddhi et al., 2008). It is known that for load bearing metal implant applications,

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biomechanical properties of the porous NiTi alloys are very important for safety and reliability. Among the mechanical properties, fatigue performance of the porous alloys is especially crucial to the usability of porous NiTi alloy because porous metals are sensitive to crack propagation even at low stresses especially under cyclic loading conditions. Further, the pores act as stress raisers and concentrators in these structures leading to significant drop in fatigue resistance. In line with this, there have been some studies showing 50-75% drop in the fatigue strength of porous coated Ti alloy implants compared to their equivalent fully dense materials (Kohn and Ducheyne, 1990; Cook et al., 1984; Yue et al., 1984). In fact, our own study on high-cycle rotating bending fatigue response of porous NiTi alloys fabricated using Laser Engineered Net Shaping (LENS<sup>TM</sup>) showed that the presence of 10% porosity in NiTi alloys can decrease the fatigue failure stress up to 54% (Bernard et al., 2011). Therefore, fatigue behavior of porous metals for implant applications assumes significant importance, in particular compression fatigue due to its close relevance to in vivo loading condition. However, to the best of our knowledge very little work has been done on fatigue behavior of bulk porous metals, in particular on compression fatigue of NiTi alloys. Some earlier works (Barrabés et al., 2008; Arciniegas et al., 2007; Guo et al., 2009; Zhang et al., 2008) reporting compression cyclic testing of porous NiTi alloys provide some preliminary understanding of compression fatigue behavior of porous NiTi alloys but in some studies the tests were carried out up to a maximum of 200 cycles only (Guo et al., 2009; Zhang et al., 2008). In this work, we have fabricated dense and porous (up to 20% porosity) NiTi alloy samples using Laser Engineered Net Shaping (LENSTM)—a laser based additive manufacturing technology. The laser processed NiTi alloy samples were characterized for their mechanical properties in monotonic and cyclic compression loading conditions. Although in vivo mechanical loading conditions are highly complex involving static and cyclic tensile, bending, shear, and so forth loading, present investigation under compression loading conditions provide useful data to understand the cyclic mechanical behavior of laser processed porous NiTi alloy samples.

#### 2. Materials and methods

Equiatomic (50:50 at%) NiTi alloy powder (Crucible Research, PA) with particle size in the range of 50–150  $\mu$ m was used in this study. Dense and porous NiTi alloy samples with different amount of porosities were fabricated using LENS<sup>TM</sup>-750 (Optomec, Albuquerque, NM, USA) equipped with 0.5 kW continuous wave Nd:YAG laser, on a substrate of 3-mm thick

rolled commercially pure Ti plates. The samples were fabricated in a glove box containing argon atmosphere with O2 content less than 10 ppm to limit oxidation of the NiTi alloy during processing. The LENS<sup>TM</sup> process uses a Nd:YAG laser, up to 4 kW power, focused onto a metal substrate to create a molten metal pool on the substrate. Metal powder is then injected into the metal pool, which melts and solidifies. The substrate is then scanned relative to the deposition head to write a metal line with a finite width and thickness. Rastering of the part back and forth to create a pattern and fill material in the desired area allows a layer of material to be deposited. Finally, this procedure is repeated many times along the Z-direction, i.e. height, until the entire object represented in the three-dimensional CAD model is produced on the substrate (Balla et al., 2009). A series of samples were processed using different laser powers, scan speeds, and powder feed rates as shown in Table 1. The bulk density of the samples was calculated from measured dimensions and mass of the samples. The relative density of the samples was determined using the ratio of the measured bulk density of the porous samples and the theoretical density (6.45 g/cc) of the NiTi alloy.

Cylindrical samples with  $\emptyset$  7 mm were used for monotonic and cyclic compression testing using a servo hydraulic materials test system (axial/torsion) machine with 250-kN capacity. Elastic modulus and 0.2% proof strength of the samples were determined from the stress-strain plots derived from load-displacement data recorded during monotonic compression testing. Compression-compression fatigue tests were carried out on porous NiTi alloy cylindrical samples, having  $\sim$ 1%, 10% and 20% by volume porosity (relative density  $\sim$ 100%, 90% and 80%), up to failure or 10<sup>6</sup> cycles (whichever occurred first) at 15 Hz with a stress ratio (R) of 0.1. Each sample with varying porosity was stressed at several values of cyclic maximum compressive stress, i.e., 100%, 120%, 140% and 150% of respective 0.2% proof strength of NiTi alloy. The maximum compressive stresses and corresponding stress amplitudes used in the present work are summarized in Table 2. The load displacement data recorded during compression-compression fatigue tests were analyzed to determine accumulated strain at the maximum compression stress as a function of number of cycles. For microstructural analysis, samples were prepared following standard mechanical polishing procedures using up to 1 µm alumina powders, and then electrolytic polishing was carried out in 1:4 H<sub>2</sub>SO<sub>4</sub>:methanol electrolyte at 5 V, at 20 °C. Finally, these polished samples were etched for a period of 10 min using a solution consisting of 20 mL distilled water, 45 mL glycerin, 25 mL HNO<sub>3</sub>, and 1 mL HF. Microstructural analysis of the samples was performed using light microscope and fieldemission scanning electron microscopy (FESEM) (FEI Sirion,

Table 1 – Laser parameters used in the present work and mechanical properties of NiTi alloy samples.							
Laser power (W)	Scan speed (mm/s)	Powder feed rate (g/min)	Energy density (J/mm²)	Relative density (%)	Porosity (100-relative density) (%)	0.2% Proof stress (MPa)	Elastic modulus (GPa)
350	15	10	19.8	98±1.6	1	971±20	30±2
200	10	15	16.9	$90 \pm 1.3$	10	$656 \pm 84$	$21\pm1$
150	20	20	6.4	$80\pm1.2$	20	368±60	14±1

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