



# Combining micro computed tomography and three-dimensional registration to evaluate local strains in shape memory scaffolds



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## ABSTRACT

Appropriate mechanical stimulation of bony tissue enhances osseointegration of load-bearing implants. Uniaxial compression of porous implants locally results in tensile and compressive strains. Their experimental determination is the objective of this study. Selective laser melting is applied to produce open-porous NiTi scaffolds of cubic units. To measure displacement and strain fields within the compressed scaffold, the authors took advantage of synchrotron radiation-based micro computed tomography during temperature increase and non-rigid three-dimensional data registration. Uniaxial scaffold compression of 6% led to local compressive and tensile strains of up to 15%. The experiments validate modeling by means of the finite element method. Increasing the temperature during the tomography experiment from 15 to 37 °C at a rate of 4 K h<sup>-1</sup>, one can locally identify the phase transition from martensite to austenite. It starts at ~24 °C on the scaffolds bottom, proceeds up towards the top and terminates at ~34 °C on the periphery of the scaffold. The results allow not only design optimization of the scaffold architecture, but also estimation of maximal displacements before cracks are initiated and of optimized mechanical stimuli around porous metallic load-bearing implants within the physiological temperature range.

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## 1. Introduction

Suitable mechanical stimuli govern tissue formation [1]. The presence of a dynamic mechanical stimulus alters, among other things, mesenchymal stem cell proliferation and differentiation and, hence, regulates bone formation [2–5]. Therefore, the appropriate cyclic mechanical loading of solid and porous implants gives rise to enhanced osseointegration [6–8]. The uniaxial compressive deformation of scaffolds, for example, leads to osteogenic effects in osteoblasts [9] and increases cell differentiation and extracellular matrix synthesis [10]. To achieve an increase in bone formation within scaffolds upon mechanical stimulation, strains between 0.5 and 1.5% are usually proposed [2,11–13].

Scaffolds for load-bearing applications usually consist of metals such as stainless steel, titanium and its alloys, tantalum or NiTi, because of the required mechanical strength [14]. NiTi belongs to well-established biomaterials with distinct mechanical properties, including comparatively low elastic modulus, high damping capacity, pseudoelasticity and the possibility for shape recovery after

deformation. It was hypothesized that pseudoelasticity should allow for cyclic mechanical stimulation of tissue in the proximity of a NiTi scaffold or implant, as an induced deformation can be recovered upon unloading [15]. Also, the shape memory effect might allow for mechanical cell stimulation, as a scaffold-shape change can be thermally induced [16]. Moreover, porous NiTi shows high osteoconductivity and supports osseointegration, which is achieved faster and with better bonding than for porous Ti [17,18]. The reasons behind this phenomenon might be the pseudoelasticity and the low elastic modulus of NiTi, as they mimic the mechanical properties of healthy bone better than other metallic alloys [17].

Several techniques for manufacturing porous NiTi scaffolds are on the market: space holder methods [19,20] including metal injection molding [21,22] or self-propagating high-temperature synthesis [23–25]. These techniques, however, only entail rounded/sponge-like pore geometries. Additive manufacturing techniques such as selective laser melting (SLM) are more flexible, as a focused laser beam fuses the metal powder according to the pre-defined design in micrometer-thin layers. In this way, one can produce complex-shaped NiTi scaffolds with micrometer-sized pores based on three-dimensional (3-D) units [16,26]. It was

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recently demonstrated that SLM-built porous NiTi scaffolds are suitable carriers for mesenchymal stem cells [27]. Furthermore, arranging the units in a periodic manner offers an easy way to tailor properties via unit cell adjustments such as varying strut thickness, unit dimensions or design. One can tune, for example, the mechanical properties and cell seeding efficacy of scaffolds [28,29]. The porous architecture of such scaffolds divides compressive load applied to the entire component into local compressive and tensile strains covering a broad range of amplitudes. The evaluation of the local strains is therefore essential for understanding cell–implant interactions and improving the scaffold architecture for tissue engineering in vitro and tissue integration in vivo.

The objective of the study is to measure the 3-D displacements and strain fields occurring upon scaffold compression in porous NiTi scaffolds via synchrotron radiation-based micro computed tomography (SR $\mu$ CT) and non-rigid 3-D registration. A pseudoplastic scaffold at room temperature was selected for the investigations, in order to determine the displacement fields during shape recovery. To this end, an in situ SR $\mu$ CT setup was used, enabling controlled heating for a continuous and well-defined shape recovery process of the deformed NiTi scaffold.

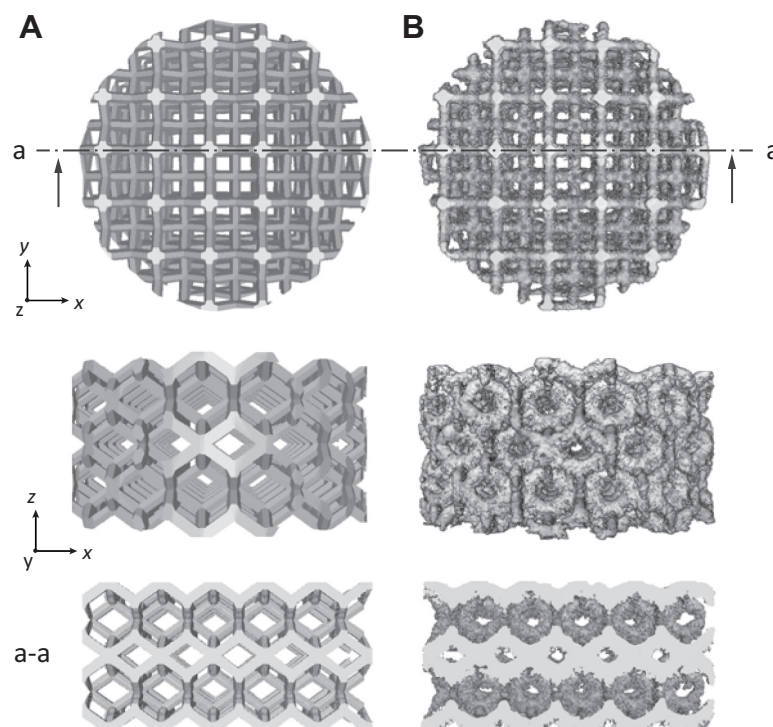
## 2. Materials and methods

### 2.1. Specimen preparation

Selective laser melting (SLM) (SLM Realizer 100, SLM-Solutions, Lübeck, Germany) served for the fabrication of cylindrical NiTi scaffolds of height 4 mm and diameter 8 mm (cf., Fig. 1). The cubic unit cell of the scaffolds with a volume of (2 mm)<sup>3</sup> was based on a rhombic dodecahedron. As the cylindrical scaffolds were built from cubic units, the cells at the fringe of the cylinder were truncated. Two layers of six complete and nine truncated unit cells were stacked upon each other, i.e. the bottom layer reached from 0 to

2 mm of the scaffold height, the top layer from 2 to 4 mm. An integrated layer of seven complete and ten truncated unit cells, staggered about a half unit cell compared with the bottom and top layer cells, was formed in the center of the scaffold. This center layer ranged from 1 to 3 mm of the scaffold height. The struts of the architectural elements were 300  $\mu$ m thick. The pre-alloyed NiTi powder (MEMRY GmbH, Weil am Rhein, Germany) with a nominal Ni-content of 55.96 wt.% consisted of spherical particles 35–75  $\mu$ m in diameter. After SLM processing, the specimens exhibited phase transformation temperatures of (10  $\pm$  9) °C for martensite start  $M_s$ , (−87  $\pm$  6) °C for martensite finish  $M_f$ , (−44  $\pm$  7) °C for austenite start  $A_s$  and (3  $\pm$  3) °C for austenite finish  $A_f$ . The broad temperature difference between martensite start and finish might be an indication that there is also an R-phase transformation included. Ni evaporation during SLM processing was calculated taking the well-known relationship between the austenite finish temperature and the Ni content as well as the impurity content of the scaffolds into account [30,31]. The preferential evaporation of Ni with respect to Ti amounted to a concentration shift of  $\sim$ 1 wt.% with respect to the starting material prior to atomization.

In order to obtain specimens with pseudoplastic behavior around body temperature, scaffolds were post-treated by annealing to a temperature of 500 °C for a duration of 60 min under an Ar atmosphere, followed by cooling to room temperature within 140 min. Even though exact adjustment of transformation temperatures requires further optimization of thermal treatment conditions, applied parameters were found to shift the phase transformation temperatures to the desired range. The resulting phase transformation temperatures of the scaffold investigated referred to 45 °C for R-phase start  $R_s$ , 14 °C for R-phase finish  $R_f$ , −18 °C for  $M_s$  and −39 °C for  $M_f$  upon cooling and to 25 °C for  $A_s$  and 41 °C for  $A_f$  upon heating. The phase transformation temperatures were determined by means of differential scanning calorimetry (DSC; DSC 30, Mettler-Toledo) using a heating rate of 10 K min<sup>−1</sup>.



**Fig. 1.** Three-dimensional views of the scaffold (8 mm in diameter, 4 mm in height): column A, CAD-file used for scaffold fabrication; column B, 3-D rendering of tomography data of the SLM-built NiTi scaffold. The cross section indicated in the top view is applied for the representation of the global and local displacement fields (cf., Fig. 3).

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