



## Pore control in SMA NiTi scaffolds via space holder usage

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### ABSTRACT

Porous NiTi shape memory alloy (SMA) was fabricated by sintering of compressed constituent elements pre-mixed with NaCl or urea spacer holders. Effect of spacer to metal volume-ratio ( $r_s$ ) on shape, size, distribution and openness of the voids was probed by optical metallography, X-ray diffraction (XRD) and scanning electron microscopy (SEM). Differential scanning calorimetry (DSC) was used to determine the SMA transformation temperatures. Controllable void geometry helping osteoblast proliferation and bone cell growth was gained by addition of the spacers. At  $r_s = 0.7$ , percentage of the open pores reached 52% while at  $r_s = 1.43$ , interconnected pores with 200 to 500  $\mu\text{m}$  diameter were produced. Although trace of microstructural chlorine was observed in NaCl treated samples, no remains were detectable in the urea made specimens. The latter had a greater advantage of being more homogeneous as well.

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### 1. Introduction

NiTi is an interesting bio-material capable of promoting osteoblast proliferation and semi-natural bone construction. This material possesses superelastic behavior, high corrosion resistance, low stiffness, shape memory effect and biocompatibility. It can, hence, be used in orthodontic teeth straightening and subtle orthopedic surgery [1].

Porous NiTi has evolved recently for bone replacement and implantation surgery. Open pores allow tissue propagation and defect remodeling [2]. This creates firm in-tissue fixation and healing expedition. NiTi exhibits desirable osteoconductivity and osteointegration when having appropriate porous structure [2,3]. Mechanical strength of the material decreases, however, with the pore size and content [4,5].

Different researchers have used alternative procedures like combustion synthesis, injection molding, spark plasma heating and conventional sintering for production of porous NiTi samples [2,6–10]. Partially hydrided titanium powders have previously been blended with Ni–Ti mixture to sinter porous SMA of desirable composition [10]. This has been described as a cost-effective simple procedure for production of low-impurity porous SMA. Biomedically-unsuitable pore specifications have, however, been a drawback diminishing biomedical application of the process.

Use of space-holder can help pore-geometry improvements [8]. Preferable pore dimensions range from 200 to 500  $\mu\text{m}$  for ordinary orthopedic applications [2,3]. Spacer-less samples yield less than

100  $\mu\text{m}$  blocked pore specimens [9]. Appropriate type, shape, size and amount of the spacer lead to an advantageous open pore structure. Pore-size increase resulting from larger spacer particles has, for example, been reported [5,11].

Pore controlling spacers should not affect on interactions of raw-materials [7]. Common spacers either are water soluble [12,13] like NaCl and NaF or heat decomposable [5,11] like urea and  $\text{NH}_4\text{HCO}_3$ . Selection of the most facilitating spacer for maximum controllability of pore-characteristics requires comparison of the effects of the spacers. Representative samples from each spacer group were, hence, selected and their effects on microstructure and characteristics of the pores were compared. Considering suitability for biomedical applications, morphology change from isolated to interconnected open-pores due to addition of the spacers was also carefully investigated.

### 2. Experimental procedure

Space-holders used in this research are depicted in Fig. 1. NaCl spacers look cube-like, in general. Urea globules have round-corner rectangular geometry with glassy surfaces. Both spacers were sieved between 300 and 500  $\mu\text{m}$  and then mixed with elemental metallic powders (titanium, 99.99% Alfa Aesar and 99.1% GER nickel Merck) with Ni/Ti atomic ratio of 49.2/50.8, for 1 h. Mixture volume-ratios used were:  $r_s = 0.1, 0.2, 0.4, 0.7, 0.8, 0.9$  and 1. The mixtures were loaded into cylindrical steel molds and compressed at 100 MPa pressure.

Compacts made with NaCl were divided into two sets. Members of the first set left inside the large beakers filled with distilled water separately overnight to dissolve all mechanically mixed spacers. Samples of the second set lost their salt when heated-up at the sintering

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