



Porous low modulus Ti40Nb compacts with electrodeposited hydroxyapatite coating for biomedical applications

K. Zhuravleva^{a,b,*}, A. Chivu^{a,c}, A. Teresiak^a, S. Scudino^a, M. Calin^a, L. Schultz^{a,b}, J. Eckert^{a,b}, A. Gebert^a

^a Leibniz Institute for Solid State and Materials Research IFW Dresden, P.O. Box 270016, D-01171 Dresden, Germany

^b TU Dresden, Institute of Materials Science, D-01062 Dresden, Germany

^c Dept. of Materials Science and Engineering, Politehnica University of Bucharest, Romania

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ABSTRACT

Porous β -type non-toxic Ti40Nb alloy was prepared by compaction of mechanically alloyed powder mixed with NaCl or Mg particles as space-holder material. The compacts with porosity of 36–80% demonstrated a very low Young's modulus of ~ 1.5 –3 GPa and compression strength of ~ 10 –35 MPa, which is suitable for potential implant material application. Porous samples were electrochemically covered with hydroxyapatite. The influence of the deposition time and of the electrolyte concentrations on the morphology of the hydroxyapatite coating was studied. It is demonstrated that a homogenous coating of hydroxyapatite crystals with different shape and size can be obtained on the surface of the porous samples.

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1. Introduction

Ti and its alloys are already widely used as implant materials because of their comparatively high biological compatibility and good corrosion resistance [1–3]. Hard tissue replacement is a well-known surgical procedure with excellent short-term results, but the long-term results are not as encouraging. It can be related to biomechanical factors associated with implant material, body reaction and implant fixation method [4]. The significant stiffness mismatch, which is expressed in a large difference between the Young's modulus of a human bone (≈ 20 GPa for cortical bone) and that of a massive implant material (≈ 100 GPa for Ti), limits the mechanical biofunctionality of Ti alloys and can cause stress-shielding with the consequence of implant failure [5,6]. Therefore, the reduction of stiffness is a major aspect for the development of Ti alloys for biomedical applications.

In comparison with conventional dense Ti alloys, porous Ti alloys show some special advantages. The interconnected porous structure may facilitate the transportation of body fluids, further promote bone tissue ingrowth [7] and thus better fixation to the surrounding bone [8]. Porous materials have a large surface area, resulting in increasing bioactivity. Besides that porous structure also reduces the Young's modulus of the alloy. For example, porous Ti alloy samples with 30–80% porosity display similar low Young's modulus (12–17 GPa) as a human cortical bone [9].

Established routes for the creation of porous architectures are spark plasma sintering (SPS) [10], fiber sintering [11], melt gas injection into liquid metal, self-propagating high-temperature synthesis (SHS) [12] and selective laser melting [13]. Another promising way to prepare porous alloys is the compaction of Ti-based powders together with a fugitive phase. Since the porosity level, the pore size as well as the shape and the resulting mechanical properties are easily adjustable [14]. In this method, the biocompatible metallic powder is firstly mixed with a fugitive second phase powder (i.e. the space-holder material), which can be later removed by evaporation or by dissolution [15,16].

New generation of Ti-based implant materials is very low Young's modulus β -type alloys composed of non-toxic and allergy free elements. The main reasons for failure of implants are corrosion and inflammation, caused by ion release from implant [17]. Some of the common alloying elements used in implant alloys were reported to be cytotoxic, for example V, Ni and Al [18]. Many attempts to produce β -type non-toxic Ti alloys ended up with alloys with Young's modulus of 40–88 GPa [19], which is still high compared to that of a human bone. Further reduction of Young's modulus can be attributed to introduction of porosity into the alloy [20]. Various studies on Ti and TiAlV porous alloys demonstrated their improved properties compared to a bulk alloy [21–23]. In the work by J. Xiong [24] shape memory Ti26Nb porous alloy was produced, that demonstrated a Young's modulus of ~ 5 GPa. A. Nouri and colleagues worked with Ti–Sn–Nb alloy [25] but upon slow cooling $\alpha + \beta$ mixture was produced, that increased the Young's modulus [22]. The challenge for the further work was to optimize processing conditions to obtain a porous mostly β -phase Ti–Nb alloy with porous size in the order of 200–400 μm .

* Corresponding author at: P.O. Box 270016, D-01171 Dresden, Germany. Tel.: +49 3 51 46 59 0; fax: +49 3 51 46 59 542.

E-mail address: k.zhuravleva@ifw-dresden.de (K. Zhuravleva).

In the present paper we will demonstrate two successful approaches to produce porous low Young's modulus Ti40Nb compacts by employing NaCl or Mg as a space-holder material. It will be shown that by careful selection of pressing, sintering and quenching conditions the defined porous architecture can be obtained together with β -phase structure, which results in remarkable medical performance data.

A popular method for surface modification of biomedical materials is hydroxyapatite (HAP) coating [9,26]. Compared with physical methods, chemical treatment is believed to be more suitable with complex surface morphology such as porous structures, because it allows the liquid medium full access to the outer surfaces and inner surfaces [7,27]. In the present paper a suitable method for HAP coating of porous Ti40Nb compacts will be described.

2. Materials and methods

Porous Ti40Nb bodies have been produced from ball-milled nano-crystalline alloy powders with composition Ti40Nb (wt.%). For that, commercial Ti (Johnson Matthey GmbH, purity 99.4%, <100 mesh) and Nb (Johnson Matthey GmbH, purity 99.8%, – 325 mesh) powders were milled under a high purity argon atmosphere (<0.5 ppm H₂O, <0.5 ppm O₂) for 40 h at a speed of 200 rpm using a planetary ball

mill Retsch PM 400. 2 wt.% of NaCl powder was used as process control agent (PCA) in order to prevent powder sticking and to improve the powder yield. The final Ti40Nb powder particle size was about 10 μ m. All details of the alloying process for obtaining nearly pure β -type Ti40Nb powders are described elsewhere [28].

Mg (350–500 μ m) or NaCl particles (125–300 μ m) were used as space-holders for the production of the porous materials. The selection of the size ranges of the space-holder particles, corresponding to the anticipated pore sizes, was made according to the recommendations for implant materials [29].

Three different types of samples were prepared: a highly dense Ti40Nb sample without space-holders, which is used as a reference sample, and samples with Mg or NaCl space-holders. The Ti40Nb powder was consolidated at room temperature to a green compact by using a pressure of 700 MPa for 2 h. The green compact was then sintered in an argon atmosphere at 1273 K for 2 h and finally water quenched. For the preparation of the porous samples with Mg space-holder, Ti40Nb powder was thoroughly mixed with 50 vol.% Mg powder and then this mixture was uniaxially pressed for 2 h at a pressure of 700 MPa to form green bodies. The green bodies were then heat treated to sinter the samples and to evaporate the Mg particles. For that the samples were sealed in argon filled quartz tubes, heat treated at 1273 K (heating

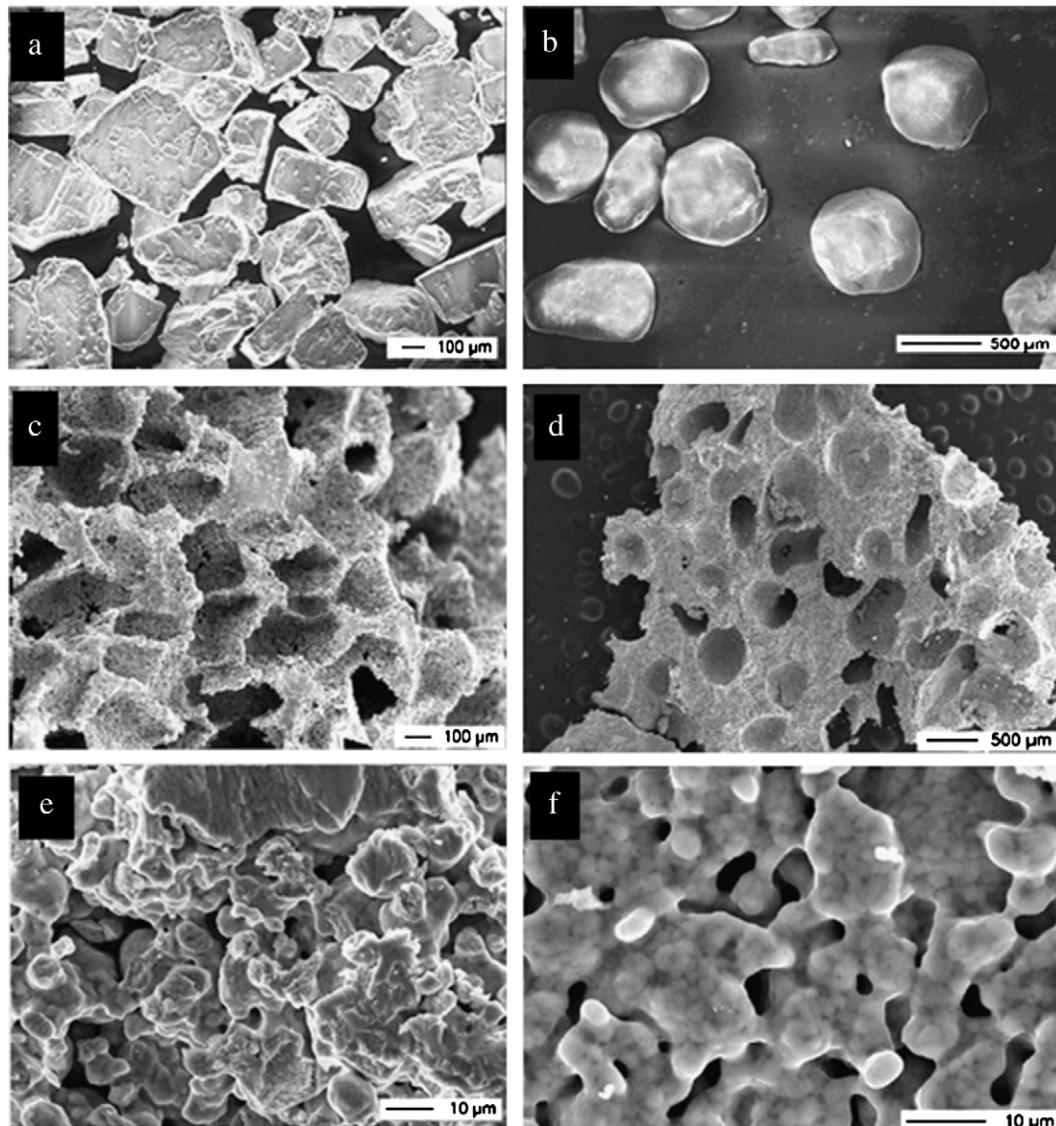


Fig. 1. SEM images of a) NaCl space holder particles, b) macro-pores, c) micro-pores of an uncoated Ti40Nb sample, prepared with NaCl particles as a space-holder, d) Mg space-holder particles, e) macro-pores, and f) micro-pores of an uncoated Ti40Nb sample, prepared with Mg as a space-holder.

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