Int. Journal of Refractory Metals and Hard Materials 53 (2015) 51–55

Contents lists available at ScienceDirect

Int. Journal of Refractory Metals and Hard Materials

journal homepage: www.elsevier.com/locate/IJRMHM

Tantalum foam made with sucrose as a space holder

Grzegorz Adamek *, Jaroslaw Jakubowicz

Poznan University of Technology, Institute of Materials Science and Engineering, Jana Pawla II no 24, 61-138 Poznan, Poland

ARTICLE INFO

Available online 15 April 2015

Keywords:
Tantalum scaffold
Metallic foam
Sucrose space holder
Porous biomaterials

ABSTRACT

The paper shows a promising method of tantalum foam biomaterial preparation with sucrose crystals as a space holder material, which is dissolved further in the process, as well as a high frequency induction heating sintering of the remaining Ta scaffold. Sucrose crystals of quasi-spherical shape have been used. The influence of the amount of space holder on the Ta scaffold porosity, phase composition and mechanical properties has been investigated. Because different Ta/sucrose ratios were applied, the Ta foams have been made with porosities of approx. 50, 60 and 70%. The space holder material has a strong influence on the phase composition of the foam surface. The research shows tantalum carbide formation during sintering. The increase of porosity leads to a deterioration of the mechanical properties. The metallic scaffold of the porosity of 50% shows the compressive strength and Young's modulus similar to that of a cancellous bone. The results show a great potential of sucrose applications in tantalum foam formation and their potential applications in medicine.

1. Introduction

The form of a material can be decisive in tissue cell proliferation and osseointegration rate, which is why not only bulk materials are appropriate for medical applications. Porous foams (void metal composite — VMC or scaffold) made from biocompatible metals seem to be a much more useful form. In this type of material, the open spaces (pores, voids) provide very useful paths for tissue cell proliferation and attachment. The porous implant is much more strongly bonded with the bone in comparison to the bulk implant [1–5].

There are many techniques of metallic foam preparation: foaming of the liquid metal [6], sintering with the space holder particles [7] or selective laser sintering [8]. Recent works [9–12] present the possibility of Ti–VMC preparation using saccharose particles as a space holder material dissolved in water or evaporated at an elevated temperature. In both cases the Ti scaffold remains, which may have medical or catalyst applications. Tantalum is gaining increasing attention as a new biomaterial. Tantalum has been shown to be corrosion resistant [13] and bioactive [14]. Bermudez et al. have shown excellent corrosion–erosion resistance of tantalum in a highly acidic environment, in comparison to titanium and stainless steel implants [13].

The oxide formed on the surface of tantalum implants in vivo by self-passivation has been found to be quite stable over a variety of pH and potential ranges [14]. Through the formation of a bone-like apatite layer in a simulated body fluid, tantalum has been proven to biologically bond to the bone. Porous Ta has provided a scaffold for bone ingrowth and mechanical attachment [15–18]. Johansson et al. have shown excellent biocompatibility of tantalum and titanium with only subtle differences in interfacial tissue reactions between the two metals [19]. Dating back to the mid-1900s, multiple medical devices have been fabricated that utilize tantalum, including: pacemaker electrodes, foil and mesh for nerve repair, radiopaque markers, and cranioplasty plates [20].

Zimmer Inc. (Warsaw, IN, USA) prepared porous tantalum structures by a pyrolysis of thermosetting polymer foam, creating a low-density vitreous carbon skeleton. Commercially pure Ta is deposited onto this interconnected scaffold using chemical vapor deposition/infiltration [21]. Balla et al. show the fabrication of porous Ta structures with total porosities between 27% and 55% using LENS™ [22].

The work shows the procedure of new Ta-based scaffold formation with different levels of porosity. This method could be an alternative to other methods of Ta foam preparation.

2. Materials and methods

The procedure of Tantalum foam preparation has been shown in Fig. 1. A mixture of Ta particles (325 mesh, Alfa Aesar) and sucrose (table sugar crystals from Pfeifer & Langen) (chemical formula: $C_{12}H_{22}O_{11}$) has been used for the preparation of the Ta metallic foam. Quasi-spherical sugar particles have been used, which provide spherical pores (voids). In this case the polyhedral table sugar crystals were mechanically modified to get rounded. It was done by mixing the crystals in a SPEX8000 mixer mill without balls for 36 ks. Ta and sucrose particles were manually mixed together for 60 s in a 100 ml beaker (in different volume ratios to achieve different porosities of 50, 60 and 70%).

The volume ratios were calculated based on the theoretical densities of the materials (16.65 g/cm^3 for Ta and 1.58 g/cm^3 for sucrose). During

* Corresponding author.
E-mail address: grzegorz.adamek@put.poznan.pl (G. Adamek).

http://dx.doi.org/10.1016/j.ijrmhm.2015.04.012
0263-4368/

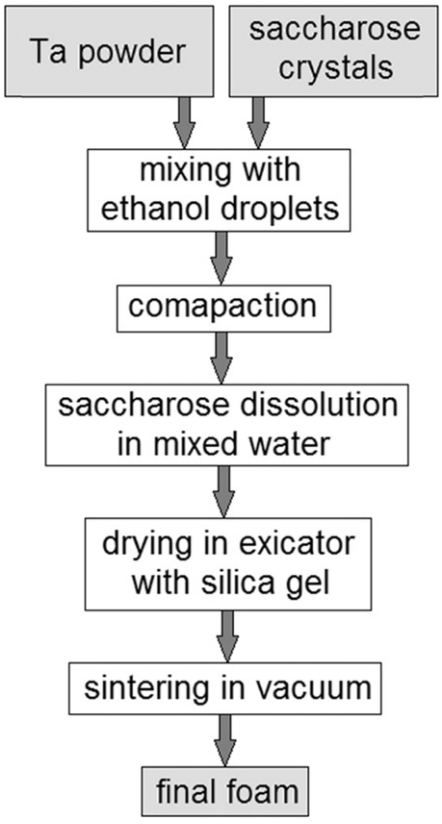

Fig. 1. The procedure of Ta foam preparation.

mixing, 5 drops of ethanol (binding agent) were added for each 5 g of mixed material. The mixture was uniaxially pressed (1000 MPa) to form green compacts (8 mm in diameter and approx. 5 mm in height). In the space holder dissolution stage, the green compacts were placed into a beaker filled with 1 l of distilled water of the temperature of 20 °C. Sugar solubility in water of the temperature of 20 °C is relatively high (211.5 g/100 ml). The water was mixed through magnetic stirring with the speed of 250 rpm. The dissolution process took 5.4 ks. The prepared porous green compacts (Ta scaffolds) were dried for 172.8 ks in room temperature (RT) in a desiccator filled with granules strongly absorbing moisture (silica gel). After drying, the green compacts were heated for 600 s to the temperature of 2000 °C and kept at this temperature for 1.8 ks for particle sintering using high frequency induction heating equipment. After that, the sinters were slowly cooled down to RT together with the furnace. The sintering was done in 10^{-2} Pa vacuum to prevent excessive oxidation.

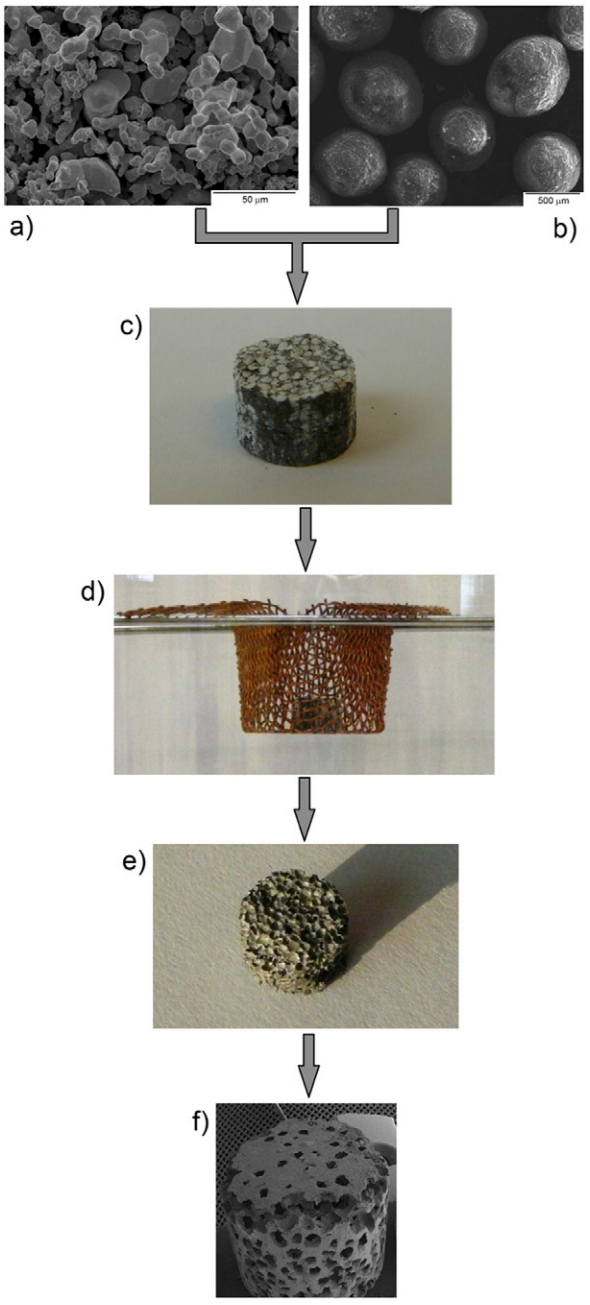

Fig. 3. Materials for scaffold preparation: Ta powder (a), sugar crystals (b); and the scaffold in different process stages: green compact (c), dissolution stage (d), scaffold after space holder dissolution (e), final foam (f).

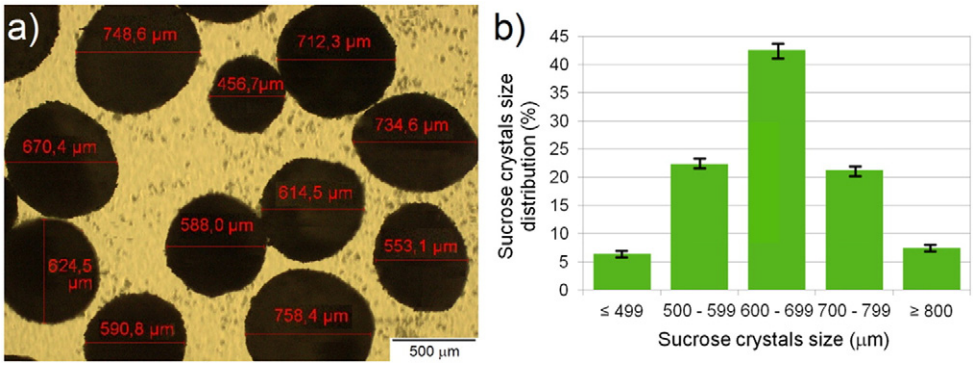

Fig. 2. Quasi-spherical sucrose crystals (a) and their size distribution (b).