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Porous yttria-stabilized zirconia for bone implants

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Zirconia is a biocompatible and hypoallergenic ceramic material with established use in implants, especially in dental medicine. In order to produce bioactive bone implants, it can be modified by application of bioactive calcium phosphate (CaP) coatings. Resulting composite ceramic implant would derive its bioactivity from the CaP coating, and mechanical and chemical stability from the zirconia scaffold. Porous zirconia stabilised with 10 wt% yttria was prepared by sol-gel process from zirconium butoxide and yttrium acetate. The resulting dry gel was sieved to particle size < 80 μm, calcined at 300 °C / 2 h and then milled at 500 rpm / 2 h. Obtained powder was uniaxially pressed (180 MPa) into tablets and sintered at 900-1400 °C / 8 h. For comparison, unpressed calcined powder was sintered in same conditions. XRD analysis has shown that zirconia crystallizes in tetragonal form, but since tetragonal and cubic zirconia structures have similar lattice parameters, Raman spectroscopy was used to distinguish the two phases and confirm the tetragonal structure of stabilised zirconia. Structure and porosity of obtained ceramics was examined by SEM (Fig. 1). Since higher sintering temperatures lead to denser ceramics, samples for mechanical characterization were sintered at 1400 °C and polished. Porosity of obtained ceramic was investigated by BET and calculated from theoretical and true density as determined by Archimedes principle. Density was determined to be 4.34 g cm⁻³, which is 76.4 % of theoretical density. Pore volume as determined by BET was 8.80 cm³ g⁻¹, which upon calculation gives apparent porosity of only 0.7 %. This is due to closed porosity of obtained ceramics, since only surface pores are available for adsorption/desorption during BET analysis. Hardness of the samples was measured by Vickers indentation test under two different loads, 5 and 20 kg. Vickers hardness was determined to be 3.8 ± 0.2 GPa, and true hardness 4.1 ± 0.3 GPa, for both loads. Under 5 kg load the samples showed no cracking, and a limited cracking (~450 μm total crack length for ~380 μm diagonal) at 20 kg load. Lack of cracking is an indication of material’s toughness. The strength was measured using ball-on-3-balls test method on 20 samples, and the results were analysed by Weibull statistical analysis. The strength was determined to be 257.7 MPa (with 90 % confidence interval 240.00 – 277.06 MPa). Fractographic analysis of samples was performed on a stereoscopic microscope and SEM. It showed possible surface crack origins, but the analysis was difficult on most samples. Both strength and hardness are lower than expected values for zirconia due to samples’ porosity, but sufficient for bone implants (bone strength ranges 4 – 24 MPa [1]).

References: 1. J. Wang et al., Bone 72 (2015) 71-80. This research was supported by Croatian-Austrian bilateral project entitled „Design, physical-chemical and mechanical characterization of innovative ceramic composites for bone implants”, and Croatian
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Fig. 1. SEM micrographs of zirconia tablets sintered at: a) 900 °C, b) 1200 °C and c) 1400 °C; d) polished surface of tablet sintered at 1400 °C.