A SCANNING ELECTRON MICROSCOPY (SEM) STUDY OF THE HYDROXYAPATITE SOL-GEL COATING OVER MA956 ALLOY

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Metallic biomaterials, such as titanium and its alloys, have enjoyed clinical successes because of their superior strength, durability, corrosion resistance in physiological environment, biocompatibility and bioinductibility. The high mechanical strength and toughness of these biometals are the most important advantages over bioactive hydroxyapatite (HAp) ceramics. Therefore, a system that combines both materials has the mechanical advantages of the underlying (metallic) substrate and biological affinity of the HAp surface to natural tissue. Thin HAp film deposits onto MA956 super alloy substrates were prepared using sol–gel technique (recently attracted much attention) [1]. The sol-gel preparation procedure has been detailed by D. M. Liu [4]. Briefly, triethyl phosphite was first hydrolyzed for 24 h with a fixed amount of distilled water under vigorous stirring. A stoichiometric amount of 4 M aqueous calcium nitrate solution was added dropwise into the hydrolyzed phosphite soln. Then, the resulting mixture was agitated for additional 3 min and aged at ambient for 24 h. Metallic substrates, MA956 super alloy (squares of 10 mm side and 2 mm height covered with an alumina layer generated by thermal treatment) were surface roughness with 150 grit SiC for enhanced coating adhesion. All samples were ultrasonically cleaned in alcohol for 10 min, in acetone for 15 min, and then in deionized water for 5 min. All cleaned samples were treated in 15 M NaOH solution at 60°C for 24 h. These substrates were dip coated with the sol-gel solution, with a withdraw speed of 5 cm/min. The coatings were dried at 80°C, heating in air at 350ºC, 450ºC, and 550ºC for 18 h. Phase formation, surface morphology, interfacial microstructure of the coatings surfaces and polished cross sections were investigated by scanning electron microscopy with associated energy dispersive spectroscopy analysis (SEM-EDS). They were typically calcined at 300 to 400°C, described elsewhere [2] in order to obtain a stoichiometric, apatitic structure, however, crystallization leads to non-chemical homogeneity in the final product (Fig. 1a). As it can be observed in figure 1a and b, first for 350°C and later for 450°C, 550°C respectively, the degree of crystallization of coatings increase according to the calcinations temperature. Low calcinations temperature carries to a dendrite type structures. A high crystalline and dense Hap coating was obtained only after heat treatment at 450 and 550°C for 18 h (regard the coating morphology in Fig.1b). The coated calcined at 450; 550°C (Fig. 2a and b) show the HAp coating morphology at small and large magnifications, respectively. The coating is porous and no cracks are found. The pores developed in the coating are believed to be a result of gas evolution during thermal decomposition. These pores seem to be connected to form a continuous network separated by from 0.2 to 0.3 μm thick walls (Fig 3). The sol-gel coated onto a MA956 substrate, calcinated at 450°C and 550°C, found to be porous with pore size ranging from 0.3 to 1 μm. This morphology may be an advantage to permit the circulation of physiological fluid, when the composite is used for biomedical applications [3]. The satisfactory adhesion between the coating and substrate suggests its suitability for load-bearing applications (Fig. 4). This result showed that no coating came out during polish work, suggesting a substantial adhesion between the coating and the substrate. This study demonstrates the synthesis of HA ceramic via a novel low-temperature sol-gel process.

Key words: Hydroxyapatite, Coating, Interface, scanning electron microscopy

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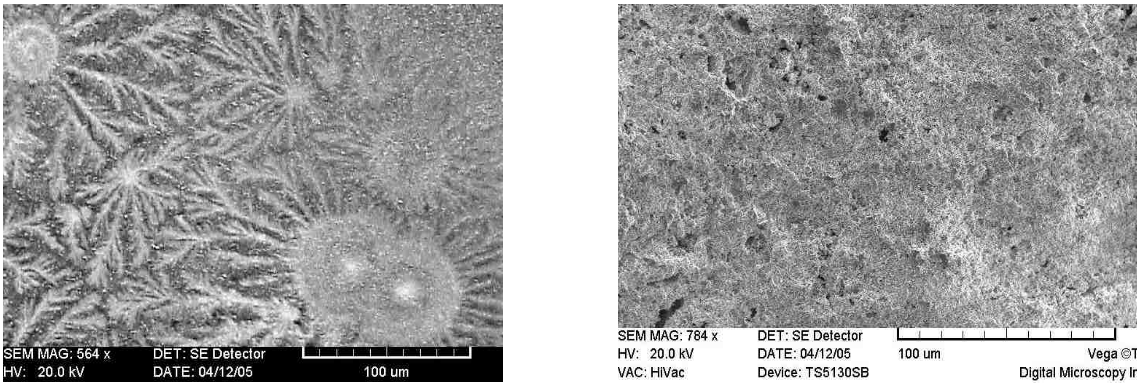

Fig. 1 SEM micrographs of HAp coating, a) after heat treatment at 350ºC, b) after heat treatment at 450 and 550ºC.

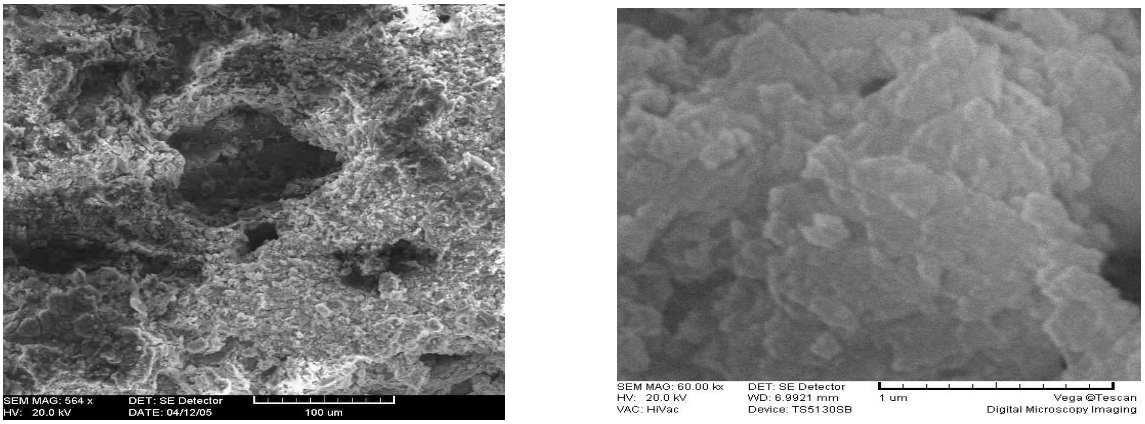

Fig. 2 Surface morphology of the sol-gel coating onto the MA956 substrates, a) small magnifications, b) large magnifications.

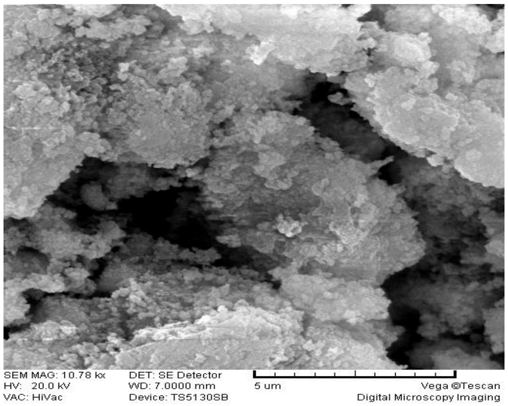

Fig. 3 Porous morphology of the coating.

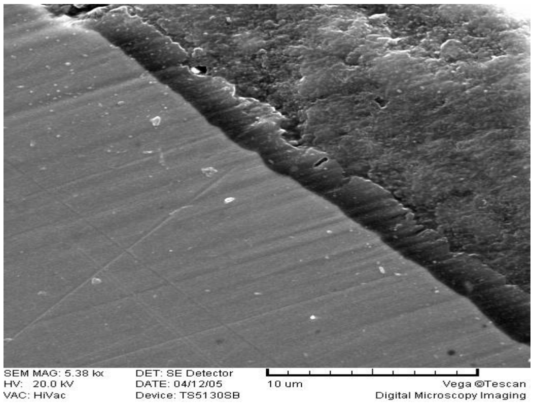

Fig. 4 Cross section of HAp coating onto MA956.

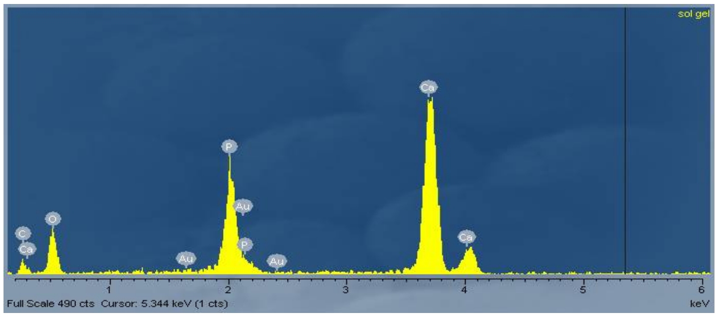

Fig. 5 Energy dispersive spectroscopy analysis of sol-gel HAp coating onto MA956 super alloy