*ELEMENTAL COMPOSITION AND MICROSTRUCTURE OF ULTRASOUND-ASSISTED MICRO-ARC CALCIUM PHOSPHATE COATINGS

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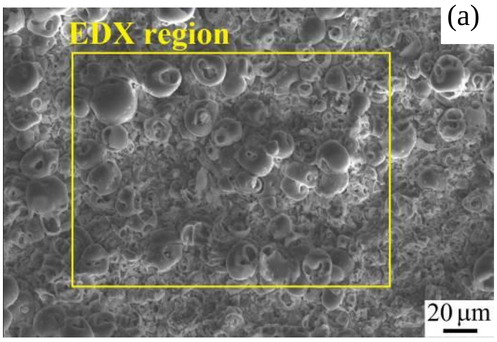
The aim of this work was to study the elemental composition and microstructure of the calcium phosphate (CaP) coatings deposited by the ultrasound-assisted micro-arc oxidation (MAO) method. The synthesis of the CaP coatings on titanium samples was carried out by the MAO method under the conditions described previously [1,2]. There were three types of the coatings depending on the conditions of external ultrasound (US): 1) MAO coating (control regime without US); 2) MAO/US coating (US-assisted MAO regime, P_{US} = 100 W, υ_{US} = 35 kHz); 3) MAO/PUS coating (pulsed US-assisted MAO regime, P_{US} = 35 W, υ_{US} = 35 kHz).

The SEM data show that the surface morphology of all types of coatings is represented by the spheroidal structural elements (sphere) with inner pores and pores between the spheres (Fig. 1a). The EDX microanalysis reveals the following elements in the all types of the CaP coatings: oxygen, phosphorus, calcium, and titanium (Fig. 1b). It was found that the application of the US field during the MAO process intensified the diffusion in the electrolyte and activated the crystallization of molten compounds. As a result, the Ca, P, and Ti contents are larger in the US-assisted MAO coatings than in MAO coatings (Fig.1b).

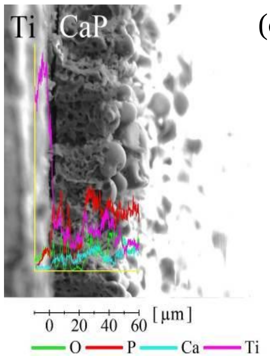
The cross-sectional SEM-images show that the all types of the coatings are characterized by the hierarchic porous structure with the numerous branched porous channels (Fig. 1c). As can be seen from the EDX scan line, the Ti amount decreases, the P and Ca amounts increase, and the O amount almost does not change throughout the coating thickness of the all types of the coatings.



(a)

(b)

Element, at.%	MAO	MAO/ US	MAO/ PUS
O Kα	69.7	52.6	51.7
P Kα	14.9	22.8	23.2
Ca Kα	4.4	7.7	7.8
Ti Kα	11.0	16.9	17.3
Ca/P	0.3	0.3	0.3



(c)

Fig. 1. SEM image of surface (a), quantitative elemental composition (b) and the EDX scan line of the elements through the thickness of the CaP coatings

The TEM studies showed the amorphous-nanocrystalline microstructure in the all types of the CaP coatings. The SAD patterns include the both point reflections from different crystalline phases and diffuse halos from amorphous phase. Indication of SAD patterns showed the presence of the following phases in the MAO coatings: $CaHPO_4$, β-$Ca_2P_2O_7$, and TiO_2 (anatase). In the US-assisted MAO coatings, the additional to previous phases the α-$Ca_2P_2O_7$ phase is detected. This high-temperature phase is formed at the temperature above 1170 °C [2]. Such significant increase of the electrolyte temperature occurs in the local electrical breakdown areas under the action of US vibrations. TEM data indicating the amorphous-nanocrystalline state in the MAO and MAO-assisted coatings are in agreement with the XRD results described in ref. [2].

REFERENCES

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