

Electrochemical Characterization of Porous Titanium Foams Intended for Orthopaedic Applications

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Since the mid-1980s, many porous biomaterials have been developed for hard and soft tissue ingrowth while porous implants are becoming increasingly important in orthopaedics and dentistry. The interest mainly comes from the fact that interconnected porosity allows tissue ingrowth and integration. Porous titanium has been used extensively for that purpose in orthopaedics. A new process has been developed recently to produce porous materials having structure and properties similar to those of cancellous bones.

Ti foams were produced (A to E in Table 1) by dry mixing the metal, a solid polymeric binder and a foaming agent (all in a powder form). The mixture was molded and underwent a three-step thermal treatment to foam and consolidate the material. The foams produced have open porosity and are permeable (Fig. 1). Modifying the initial powder mixture (MIX. in Table 1) and the thermal treatment conditions produce materials having different microstructures. One specimen (E) was coated with TiO₂ by sol-gel. Density was calculated by measuring the weight and size of the specimens, the average pore size (P. size, Table 1) was determined by image analysis while the surface area was evaluated both by BET (S_1) using Kr as the adsorbate and by electrochemical impedance spectroscopy (EIS) (S_2). Electrochemistry was used for three roles: i) Corrosion evaluation in a simulated body fluid (SBF) at 37°C through corrosion current density (j_o) and potential evaluation (E_{corr}), ii) Protective film stability evaluation by performing cyclic voltammetry (CV) studies, iii) Material characterization by EIS.

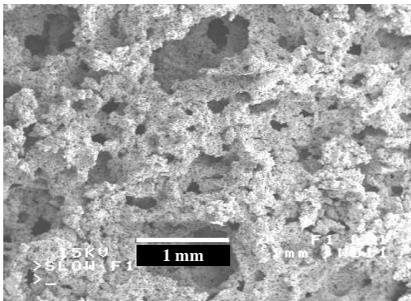


Fig. 1.
Titanium foam
B, micrograph

For each material, evaluation of the double layer capacitance (C_{dl}) and of the rugosity factor (R) by EIS [1] (see Table 1) were essential in order to normalize the j_o values [2] displayed in Fig. 2. In this figure, it is clearly shown that corrosion resistance of the foams is not affected by the structure of the material ($1 < j_o < 1.7$ nA.cm⁻²) even after a 144-hour immersion period (see foams C and D). Moreover, there is a slight improvement compared to solid titanium (S.Ti: polished) (4 nA.cm⁻²). It must also be pointed out that foam E, coated with TiO₂, exhibited the highest corrosion resistance ($E_{corr} = +155$ mV/SCE and $j_o = 0.8$ nA.cm⁻²). Corrosion resistance for the different titanium materials is linked to the thickness of the TiO₂ layer formed during the fabrication process. In fact, a CV study showed that Ti passivation increases with

TiO₂ layer thickness (Solid Ti < foams A to D < foam E). As far as surface areas were concerned, some differences between S_1 and S_2 were observed for foams C and D but still remained in the same order of magnitude. Additional studies are in progress to explain these variations.

Table 1. Ti materials processing and physical data

	S. Ti	A	B	C	D	E
T.trea. °C	--	1200	1200	1300	1300	1200
Atm.	--	Ar	Ar	Ar	vac.	Ar
MIX.	--	M1	M2	M1	M1	M1 + TiO ₂
Density g.cm ⁻³	4.5	1.19	1.30	--	1.35	see A
P. size µm	--	650	430	--	560	see A
C_{dl} mF/cm ²	0.01	31	32	32	42	1230
R	1	2560	2670	2637	3491	101447
$^a S_1$ m ² .g ⁻¹	--	--	0.055	0.033	0.036	--
$^a S_2$ m ² .g ⁻¹	--	0.074	0.066	0.061	0.098	2.587

^a S_1 and S_2 measured by BET and EIS respectively.

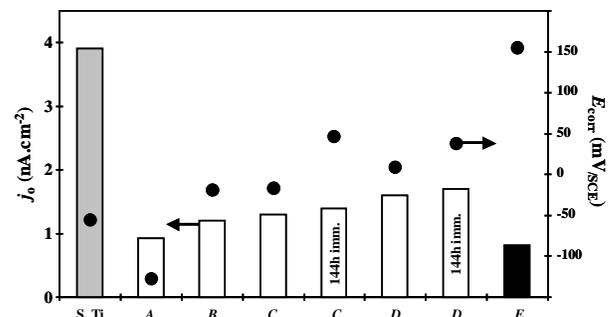


Fig. 2. Corrosion current densities and potentials for Ti materials after 18-hour immersion in SBF at 37°C

The EIS data for foams A to D were modeled according to the equivalent circuit (Fig. 3) proposed by Azziz-Kerrzo *et al.* [3], where the resistance R_{pr} and the CPE Q_{pr} correspond to an outer TiO₂ porous layer.

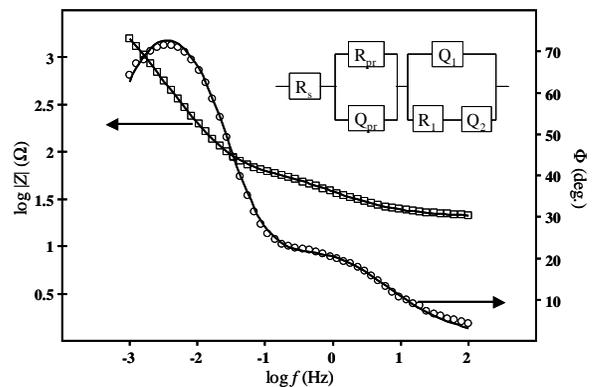


Fig. 3. EIS data fitting for the Ti foam B

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- [3] M.Azziz-Kerrzo *et al.*, *Biomaterials*, **22**(2001)1531