Texture analysis in rabbit tibia bone at the interface with implants

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We investigated the preferred orientation of hydroxyapatite $Ca_{10}(PO_4)_6(OH)_2$ (HAp) crystallites rabbit tibia bone at the interface with an implant after 40 and 60 days of implantation. The implant Ti-6Al-4V had two faces, one coated and one non-coated with plasma-sprayed HAp (80 µm). Using the 2-axis neutron diffractometer D20, we could probe samples with a spatial resolution of 0.5 mm starting from the interface to inspect the reorganisation of the HAp crystallite distribution and the crystallinity after implantation.

Medicine experiences an accelerated development of new technology, aiming for preventive, diagnostic and therapeutic progress. One has to make choices and establish strategies according to criteria of safety, effectiveness and utility. The European Commission estimates the world market of biomaterials at 25 G€, with an annual growth rate of 5 to 7%. The orthopaedic biomaterials represent 8 G€ of this market, with an annual growth rate of 7%. Hip and knee prosthetics alone represent 40% of this market segment, with 750 000 and 500 000 medical interventions per year respectively [1].

Bone is the primary structural material used to carry major loads in an enormous variety of vertebrate animals. It is a composite material in which two phases are associated, a mineral phase, formed by crystals of hexagonal HAp, and an organic phase, collagen. The *c*-axes of HAp and the collagen fibres are preferentially oriented in the direction of the stresses that the bones need to withstand [2]. Bone occurs in two principal structural forms: cortical, or compact bone, which forms a dense matrix, and spongy bone [3]. Compact bone is found in the axes of long bones and spongy bone at the ends of long bones, inside the vertebral bones of the spine and, compressed, between the layers of compact bone in the skeleton's plate structures [4]. We used cortical bone for this analysis.

HAp crystallises in the hexagonal crystal system and its unit cell parameters are a = 9.4 Å and c = 6.8 Å. Its space group is P63/m.
The texture is a non-random distribution of the crystallites in a polycrystalline material as a consequence of the preparation technique or the nature of the crystallites. It is defined by the fraction of volume of crystallites having the same orientation g.

$$(dV/V)/dg = f(g)$$

For representing the texture, we use pole figures defined by:

$$P(hkl)(\alpha,\beta) = \frac{1}{2\pi}\int_{\vec{h}//\vec{y}} f(g)\,d\Psi = \frac{1}{2\pi}\int_{(hkl)^{\perp}(\alpha,\beta)} f(\omega,\chi,\varphi)\,d\Psi$$

Here, y is the sample direction and h is crystal direction, ψ measures a rotation of the crystal about the common h and y direction; ω, φ and χ are the Euler angles.

Implants with plasma sprayed HAp coatings on titanium alloy Ti-6Al-4V are frequently used in orthopaedic surgery. Ti-6Al-4V presents good mechanical properties and is biocompatible. HAp, as an implant material, has low mechanical strength, but very good osteointegration and biocompatibility. The combination of these two materials gives mechanical strength and good

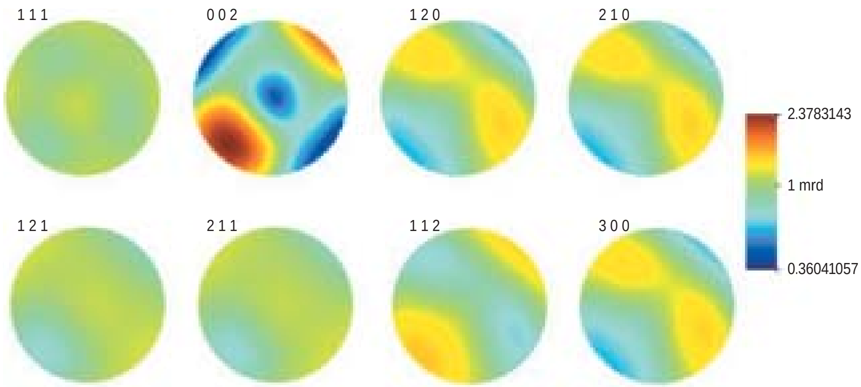

Figure 1: Pole figures of rabbit tibia bone (1 cm x 5 mm x 5 mm) showing the preferred orientation of HAp crystallites, in particular when comparing the reflection (002) and the (111) reflection, nearly not affected by texture.

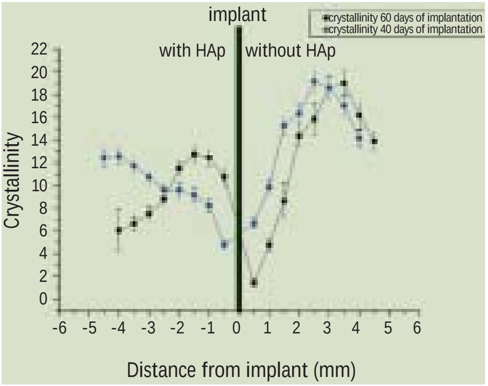

Figure 2: Relative crystallinity of rabbit tibia bone using the intensity of peak (111), not affected by texture.

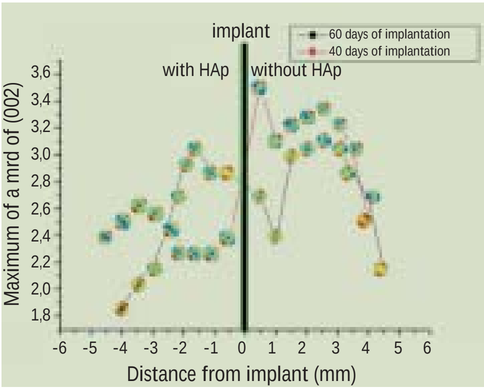

Figure 3: Maximum of multiples of a random distribution (mrd) of (002), indicating the degree of preferred orientation, as function of the distance from the implant interfaces.

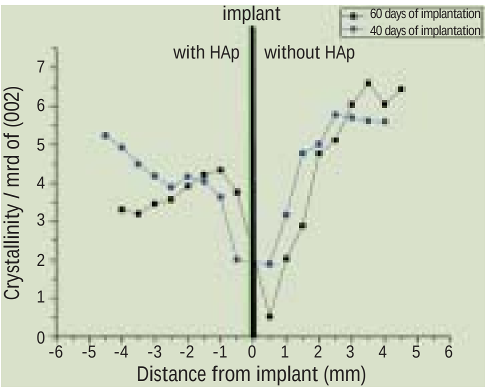

Figure 4: The ratio of crystallinity and the maximum of multiples of a random distribution (mrd) of (002).

osteointegration properties at the interface [5]. In order to improve these coatings, it is necessary to investigate the transformation of the bone's properties, such as crystallinity and texture, as a function of the distance from the implant-interface and the time since implantation. It is necessary to remove substantially the collagen in order to reduce the incoherent scattering of hydrogen.

We used two 20 to 22 week-old rabbits, the implant volume was 6 $\times$ 4 $\times$ 2 mm^3 and the thickness of its HAp coating was 80 µm. 40, respectively 60 days after the implantation, the bone-implant samples were extracted. It was necessary to remove the collagen from the samples, in order to reduce the intense incoherent scattering of neutrons by hydrogen. This is preferably done by heat treatment in a furnace [6]. A piece of rabbit tibia (10 $\times$ 5 $\times$ 5 mm) was mounted in an Eulerian cradle. We made an embraced scan with step sizes of 10° for $\Delta\varphi$ and $\Delta\chi$ around φ and χ, with j from 0 to 360° and χ from 0° to 90°, and ω constant at 90°. The size of beam was 9 mm $\times$ 0.5 mm and λ = 2.4 Å. It takes 6 hours for each slice of 0.5 mm of the tibia. The pole figures and the orientation distribution function were obtained from simultaneous Rietveld refinement of all patterns for each slice (Rietveld texture analysis, RTA) with the program MAUD [7], applying spherical harmonics to model the texture [8].

For quantifying the crystallinity, we have selected the intensity of the peak (111), not affected by texture, normalised to the constant incoherent scattering of hydrogen, which is proportional to the mass of the illuminated bone. The evolution of texture and crystallinity is traced depending on the distance from the implant.

The results show that bone presents at the interface with an implant two particular different behaviours:

- A non-coated implant favours the orientation of crystallites in the bone but not the crystallinity. We can consider that this fixing is still unstable at the date of the extraction.
- A coated implant favours the crystallinity of HAp in the bone but not the orientation of crystallites. Mechanical fixing appears more stable at the date of the extraction [9].

We can conclude that, at the interface with an implant, a bone tends to change its properties, affecting the acceptance of the implant. Surface coating can reduce significantly the rejection rate of implants.

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