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Effect of Texture on Success Rates of Implants

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1. Introduction

The aim of the present study was to study the interface implant-bone by synchrotron radiation and neutron diffraction, the implant has two faces the first one coated with hydroxyapatite and the second uncoated. In orthopaedic surgery, Titanium alloy (Ti-Al-4V) implants are currently coated with hydroxyapatite (HAp), Ca_{10} $(PO_4)_6$ $(OH)_2$, in order to obtain a stable and functional direct connection between the bone and the implant. At the implant-bone interface, the new bone reconstituted after two months of implantation must have the same properties like the natural bone in order to have good mechanical properties at the interface with the implant. Therefore, we studied the texture of the reconstituted bone crystals at the interface applying two non-destructive diffraction methods, as well as the influence of the coating on crystallinity index. The required high spatial resolution was achieved utilizing high-energy synchrotron radiation on ID15 at ESRF in Grenoble, France, and the second method was done by neutron diffraction, the high-intensity with two-axis diffractometer was used, equipped with variable resolution: D20, at Institut Max von Laue-Paul Langevin (ILL) in Grenoble, France.

In orthopaedic surgery, it is necessary to use biocompatible implants in order to have good mechanical and fracture resistance. Bone is a composite material whose components are primarily collagen and HAp. The c-axes of the apatite crystallites and the collagen fibres are preferentially oriented, e.g., in the long bones in the directions of the stresses that the bones need to withstand. HAp crystallizes in the hexagonal system and its unit cell parameters are a=9,4 Å and c=6,8 Å its space group is P63/m. Bone occurs in two principal structural forms: cortical, or compact bone, which forms a dense matrix, and spongy bone. We use cortical bone in this work.

The long-term success of biomedical implants largely depends on the stable fixation of the implant to bones. Composite materials, in which metals have coated with ceramics, have been extensively reported as an alternative to plain materials in favour of a long-term fixation. The development of bioactive ceramic-alloy structures for implants has focused by the idea of combining the bioactivity of ceramics with the mechanical properties of selected metallic alloys.

Ti-6Al-4V presents good mechanical proprieties and is biocompatible. HAp has low mechanical strength, but has a very good osteointegration and biocompatibility. The idea is to combine these two materials in order to have mechanical strength and good osteointegration proprieties at the interface. Plasma spraying is the most popular method for coating implant parts with HAp. In order to improve these coatings, it is necessary to investigate the texture and crystallinity evolutions of the bone's crystal structure, as a function of the distance from the implant-interface.

2. Materials and method

The success of the biomaterial used in implantology necessarily depends on the interface between implant and bone. Among many biological parameters on the mechanical level, the lifetime of the implant depends on the distribution of the regenerated HAp crystals at the interface with implant. The reconstructed orientation of these crystals should respect the preferred orientation of crystals of the bone of origin. The advantage of this protocol is to test the effect of implant coating with a layer of HAp crystals on the properties of the reconstructed bone. We will use an implant Ti-6Al-4V parallelepiped with one face was coated with a deposit of HAp hoping to observe a different behavior of the distribution of HAp crystals in the bone between the coated surface and the face unpaved.

2.1 Plasma thermal spraying "PTS"

The plasma thermal spraying "PTS" has been applied to deposit the HAp onto a titanium substrate. It is a powerful tool for the high rate deposition of thin coatings with low cost precursors. It is necessary to choose the thickness of HAp between 60 and 120 μm in order to satisfy the clinic application. PTS process consists in introducing solid particles in a high-temperature and relatively high-velocity gas jet, where the particles are eventually melted and projected onto the substrate to form the deposed coating, layer by layer. The plasma jet accelerates and heats the particles in a highly complex manner, strongly depending not only on plasma thermal spraying setup, but also on individual particle trajectories, actually determining the thermal history of the latter.

2.2 Bones sample

In this study, we use the implant constituted by Ti-6Al-4V (20x5x2mm) with two faces: the first face coated with HAp (80μm) and the second face uncoated. The implant was been inserted in the tibia bone of a sheep respecting the clinical protocols. The sheep has been pre-medicated and anesthetized. Two separate longitudinal incisions, 5 cm long, have been made on tibia bone. Corresponding to each skin incision, the cortex of the head of the tibia has been exposed, and a 5 mm wide and 20 mm long strip of peri-osteal tissue has been

Fig. 1. Implant inserted in sheep tibia

removed. Using an oscillating saw, two longitudinal slots have been made at about 120° from each other on the circumference of the bone. The implant has been introduced in one of these slots, the other remains as a control. After 60 days of implantation, the sample constituted by implant and bone has been extracted and preserved in the ethanol in order to keep them under the best conditions before measurements. The specimen has been prepared in the Pius Branzeu Centre of Laparoscopic Surgery and Microsurgery (Romania).

The sample will be characterize by synchrotron diffraction at ESRF and after by neutron diffraction at ILL in order to compare the results of this two techniques by studying the texture and crystallinity of the new bone reconstituted at the interface with implant.

2.3 Texture

The preferred orientation or texture is defined by a non-random distribution of crystals in the polycrystalline material. The study of preferred orientation is an important technique for understanding the structures of polycrystalline materials. One of the main problems in materials science is to link the physical properties of anisotropic polycrystalline material to the preferential alignment of the components in the preferred directions. For many years the development of textured metal or textured polymer is area research subject in improving the behaviour and life of components. Several areas of research are relevant today as electronics: the development of thin films, the texture not only controls the electrical properties but also the mechanical stability of films. In our study, the texture is present in biomaterials, yet the relationship between the mechanical properties of bone and alignment of hydroxyapatite crystals in bone has never been studied in spatial resolution, who laid the groundwork for the analysis of texture in biomaterials. In many cases, the characterization of texture has an important influence in the resolution and refinement of powder diffraction patterns. In an effort to characterize texture at all scales, from micrometer to the centimeter, researchers have used an increasing range of techniques to characterize quantitatively the texture. In this context, the use of large facilities such as synchrotrons and neutron sources, is particularly important and finds a new application in biomaterials.

2.4 Neutron diffraction study

With the neutrons diffraction, it's very necessary to remove the organic part from the bone in order to reduce the intense incoherent scattering of neutrons by hydrogen and that is done by heat treatment. The heat treatment does not affect the preferred orientation of the mineral bone crystallites and crystallinity. With synchrotron we can do the measurement without remove the organic part.

By neutron diffraction, we used the high-intensity two-axis diffractometer with variable resolution: D20, at Institut Max von Laue-Paul Langevin (ILL) in Grenoble, France. D20 provides a high flux of up to 108 $cm^{-2} s^{-1}$ at the sample position and medium to high resolution. It is equipped with a large linear curved position sensitive detector (PSD) permitting numerous short-time measurements. The PSD has an aperture of 153.6° with 10 cells per degree (2θ). It operates in a wide range of wavelengths. It is used either for fast data-acquisition, e.g. time-resolved powder diffraction or texture measurements, or for diffraction experiments that require accurate intensity measurements, e.g. investigations of disordered systems or physisorbed layers. D20 is particularly suited for real-time measurements and offers different sample environments, among others a vacuum vessel and an Eulerian cradle for texture measurements.

The sample has been mounted in the Euler cradle. The scan was made with step sizes of 10° for $\Delta\varphi$ and $\Delta\chi$, with φ from 0 to 360° and χ from 0° to 90°, and ω constant at 90°. The data acquisition was done during the motor motion of φ. Every 1˚ of rotation, the motor stopped for a fixed amount of time, and after rotation of 10˚, the accumulated acquisition during these ten steps of motor motion is attributed to the average angle of φ. This method enabled us to gain one hour of dead time normally spent on motor motion, without losing information on the orientations of crystallites. The size of beam was 9 mm × 0.5 mm and λ = 2.4 Å. It takes 6 hours for each slice of 0.5 mm of the tibia.

2.5 Synchrotron diffraction study

For the study by synchrotron diffraction, we used the high spatial resolution utilizing high-energy synchrotron radiation with a monochromatic beam of 88.9 keV on ID15 at the European Synchrotron Radiation Facilities ESRF in Grenoble. A Mar345 image plate detector was set at 1m from the sample to allow a complete collection of the Debye–Scherrer rings. In order to obtain a complete pole figure, it is enough to rotate the sample in the axial direction. The synchrotron radiation scan was carried out from 0° to 180° in 10° steps at a step time of 120s, beam size : 300µm and λ=0,14Å.

2.6 Material Analyzed Using Diffraction techniques "MAUD" program

In the texture analysis field, we need the sophisticated tools in order to get the maximum information from the analyzed material using diffraction techniques. Currently, very powerful tools can gather in a short time a large quantity of data. It is difficult to process all this data in a traditional way of consuming even with very powerful workstations. The processing time by computer from the latest generation was now significantly reduced. However, the amount of data collected also increases with measurement time granted to the user, time is always the same and limited. Several informations of data collected by the instruments were lost or never used. The solution is to develop new analytical tools that we can process more data and optimize the measurement time allowed for each user who wants to use large instruments. Material Analyzed Using Diffraction Techniques "MAUD" program meets this need.
L. Luttorotti has developed particularly powerful software based on the Rietveld method for quantitative analysis with two modes of refinement. MAUD program is written in Java and allows to adopt all the current machines at ILL and ESRF.

2.7 Results and discussion

The data were analyzed in order to define the structure of the HAp in tibia bone and texture. The Fig.2 shows all diffractions patterns of HAp in the bone at 6 mm from the interface with implant.
In the first, we refine all parameters of HAp structures, and after we refine the texture of HAp using the MAUD program. For presenting the orientations of tibia bone, we trace the pole figures of HAp in bone at 6mm from the implant. This distance is very far from the interface in order to show how the HAp crystallites are oriented in unperturbated parts of the bone, the pole figures tracing in this distance indicating the reference of orientations for this work. The reconstructed pole figures from Rietveld texture analysis with MAUD program of a tibia bone showing the little preferred orientation of HAp crystallites when comparing the reflection (002) and the (111) reflections. We choose the reflection (002) for the study the texture and (111) a measure of the crystallinity of the HAp at the interface with implant.

2.8 Result of synchrotron radiation

ID15B has a 2D detector, the image consist by the Debye–Scherrer rings at the interface with the implant (Fig. 2).

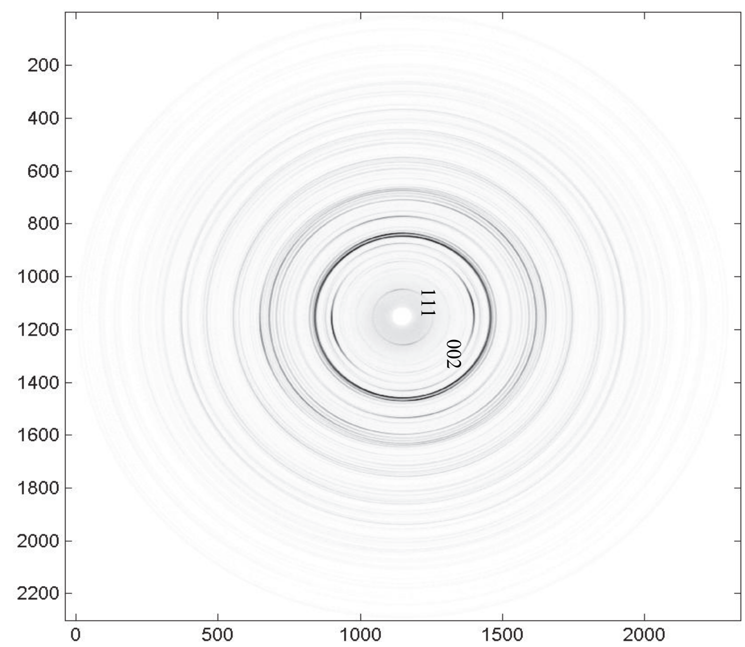

Fig. 2. Diffraction image of tibia bone at the interface with the implant.

In order to get the data for Rietveld analysis, the one-dimensional diffraction patterns were obtained by integration of the diffraction rings along each ring in 10° steps, we got 684 diffraction patterns (Fig. 8). It is necessary to refine the crystal's structure and texture in order to trace the pole figures at the interface with implant.

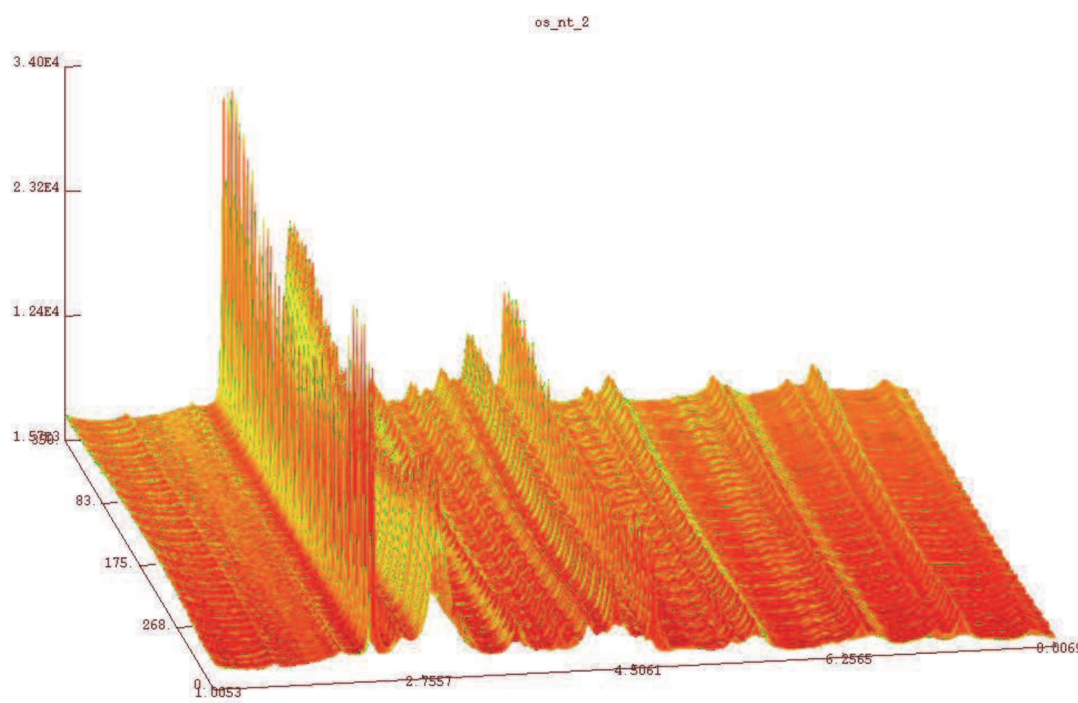

Fig. 3. 684 diffractions patterns of HAp

In the first, we refine the sum of all data in order to calculate the parameters structures of HAp Fig. 4.

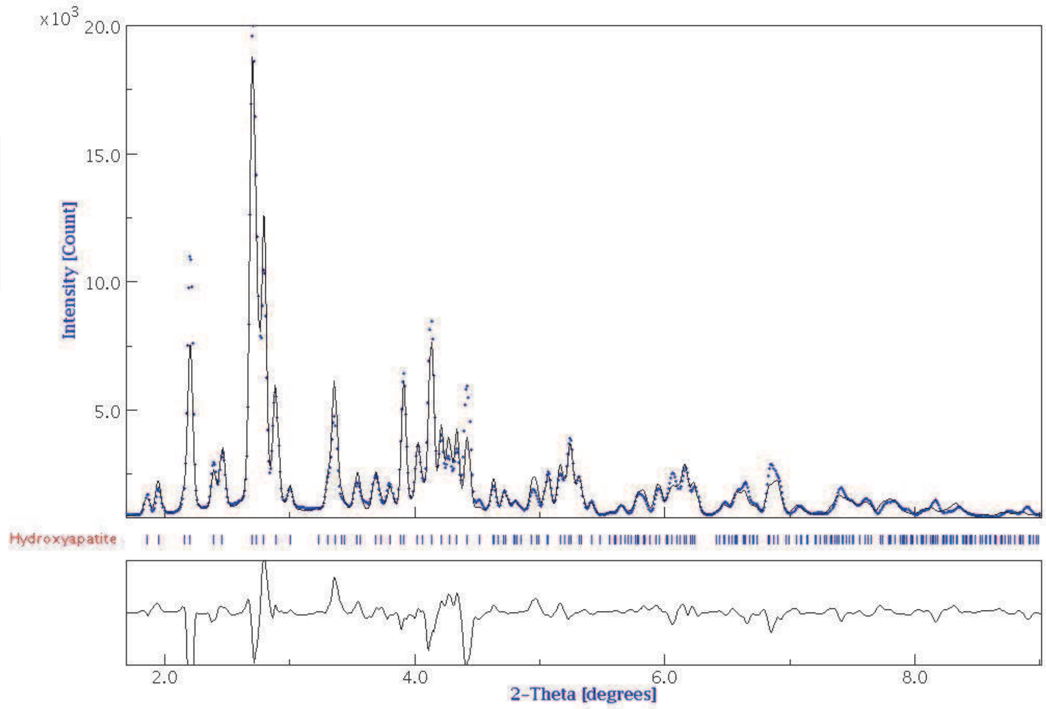

Fig. 4. Reitveld refinement of sum of all data

The second step is the refinement the texture and to trace the pole figures. The all structure parameters have been and the texture of 684 diffraction patterns has been refined simultaneously by introducing the texture parameters on MAUD program.

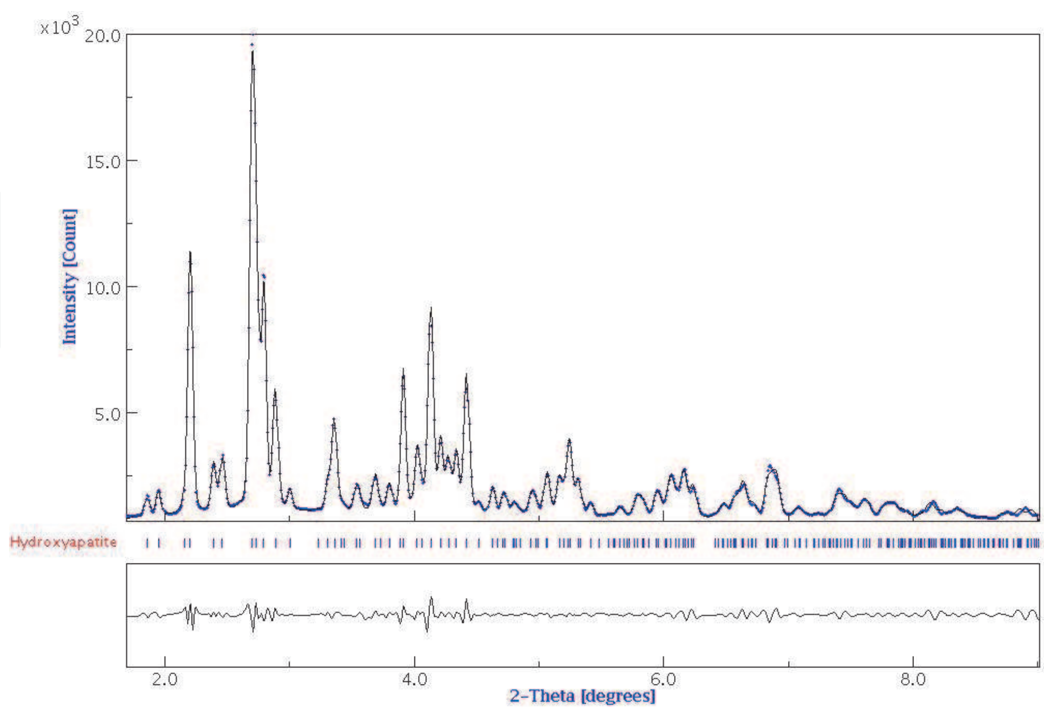

Fig. 5. Reitveld refinement of sum of all data with texture parameters

The figure 6 represents the pole figures obtained from a layer (0,3 × 0,3 mm²) of bone, very far from the implant in order to have the reference and to show how the HAp crystallites are oriented in the unperturbed part of bone.

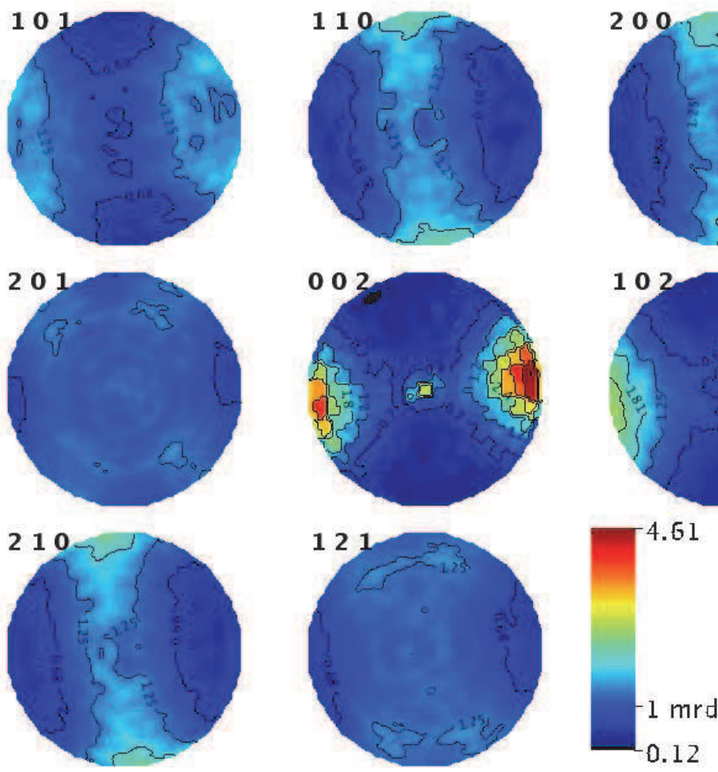

Fig. 6. Pole figures of tibia bone at 6mm from the interface bone-implant

The reflection (002) was choose for the study the texture and (111) for characterizing the HAp crystallinity evolution at the interface with implant.
The pole figures at the interface with face coated and uncoated were traced in figures 7 and 8.

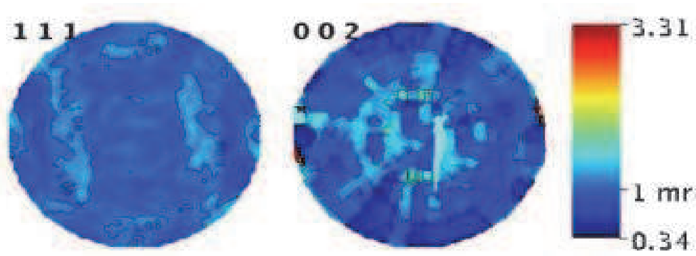

Fig. 7. Pole figures of tibia bone's at the interface with the face coated with HAp

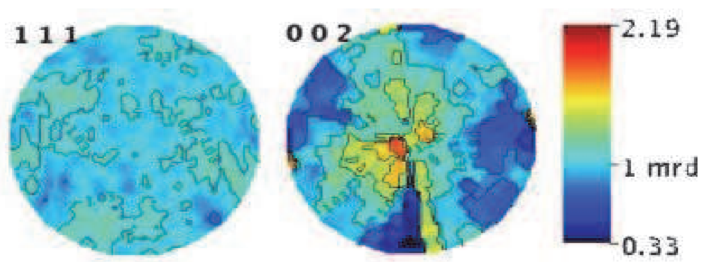

Fig. 8. Pole figures of tibia bone's at the interface with the face uncoated

At the interface with the face uncoated (Fig. 8), the pole figure (002) shows several orientations of the HAp crystallite's.
We get the preferred orientation like the unperturbated parts of the bone (Fig. 9), after 2,4 mm from the uncoated interface.

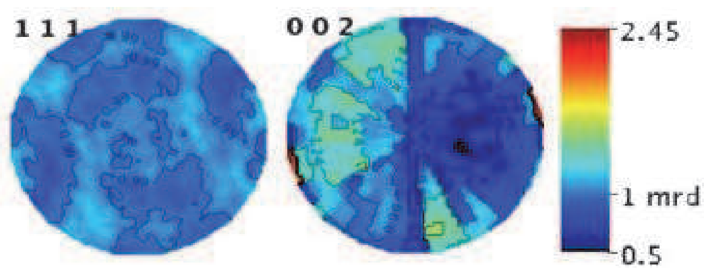

Fig. 9. Pole figures at 2,4 mm from the interface with the face uncoated

2.8.1 Results of neutron diffraction

With neutron diffraction, we got 360 diffraction patterns; like the synchrotron diffraction data, the all structure parameters have been refined and after the 360 diffraction patterns have been refined simultaneously by introducing the texture parameters on MAUD program. The figure 10 represents the pole figures obtained from a layer (0.5 × 9 mm^2) of unperturbed bones very far from the interface.

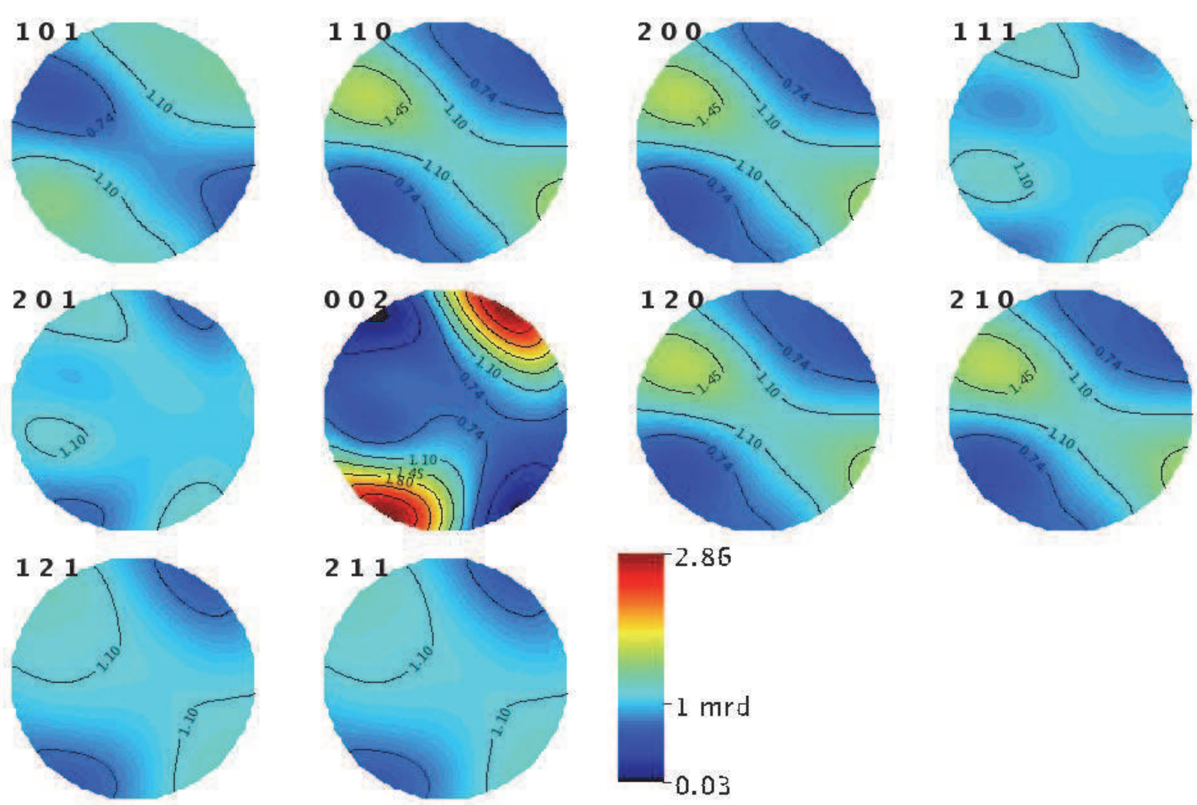

Fig. 10. Pole figures of tibia sheep bone by Neutron diffraction

The figure 10 reveals that the reconstructed pole figures from Rietveld texture analysis with MAUD of a tibia bone showing the little preferred orientation of HAp crystallites when comparing the reflection (002) and the (111) reflections, like the pole figures measured by synchrotron radiation. The pole figures at the interface with face coated and uncoated were traced in figures 11 and 12.

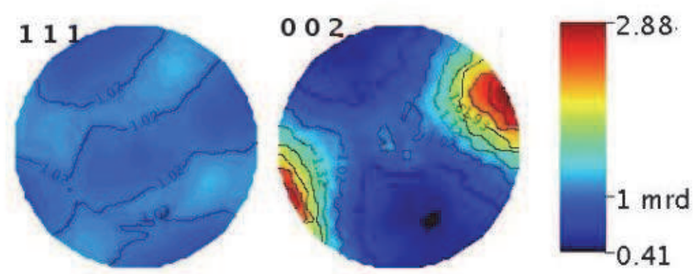

Fig. 11. Pole figures at the interface with face coated with Hap

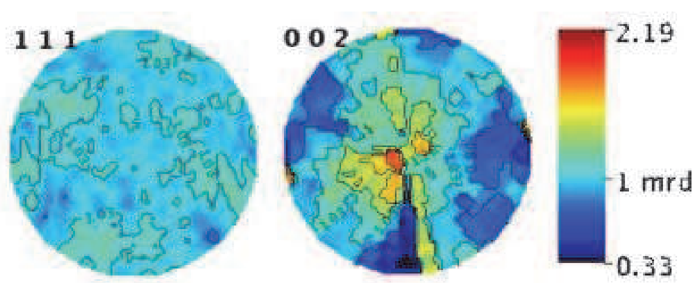

Fig. 12. Pole figures at the interface with face uncoated

AT the interface with the implant, the reconstituted bone at the interface with the face coated with HAp show the preferred orientation like the tibia bone. However, at the interface with the face uncoated (Fig. 6), the pole figure (002) show several orientations of the HAp crystallite's. At the interface with the face coated with HAp the new bone reconstituted conserves the preferred orientation of natural bone.

2.9 Crystallinity study by neutron diffraction and synchrotron radiation

The crystallinity index evolution has been measured from the interface with the implant. Therefore, we measured the intensity of the peak (1 1 1), because it is not affected by texture. The sheep tibia was measured with beam size of 0,5 mm by neutron diffraction and 0,3 mm by synchrotron radiation. The refinement was be done by MAUD.

The evolution of the crystallinity index has been traced from the interface with the implant coated with HAp and uncoated.

The crystallinity index of (111) peaks revealed that at the interface, the measurement by synchrotron diffraction is more intense than the measurement by neutron diffraction (Fig. 13) at the interface with the face coated with HAp and uncoated. At the interface with the fac coated with HAp the crystallinity is very high, the coating favoured the crystallinity by 20% compared with the face uncoated.

The results of the new bone crystals at the interface obtained by the synchrotron radiation and neutron diffraction study are particularly interesting and reveal a great advantage of the HAp coated implant interface on the crystallinity anf texture. At the interface with implant coated with HAp, the crystallinity is more intense and the new bone has the same orientation of the tibia's bone.

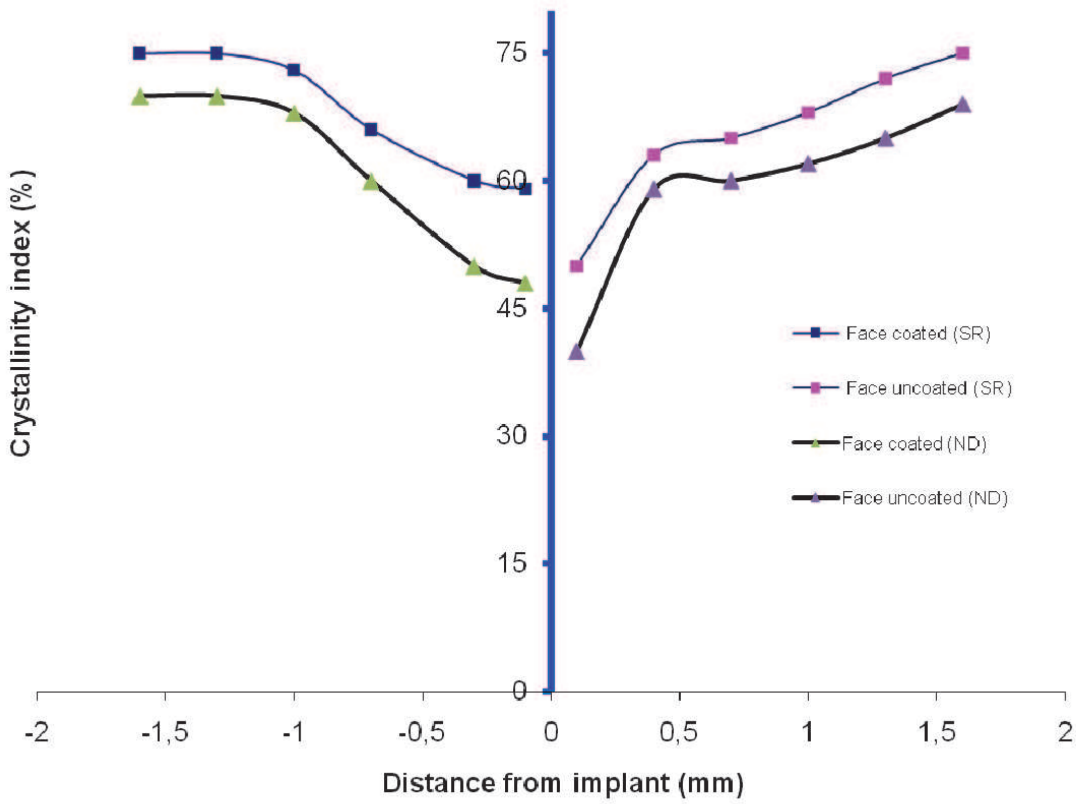

Fig. 13. Crystallinity index of the HAp at the interface with implant, ND: Neutron diffraction and SR: Synchrotron Radiation

3. Conclusion

The method of characterizing the texture and crystallinity, which uses the technique of neutron diffraction and synchrotron radiation at high spatial resolution, finds an application in implantology. In mechanical terms, the success of bone regeneration around implants depends directly on the orientation distribution and crystallinity index of HAp crystals in reconstituted bone after implantation. The non-destructive methods study allows also to compare the results from the faces coated with HAp and uncoated. The texture of HAp crystallites in new bone around the implant and crystallinity are the preferable conditions for successful implantation. If the implant was coated with HAp, the new HAp crystallites preserve the preferred orientation of HAp inside the bone. If the implant is uncoated, the orientation of HAp crystallites were changed, in this case we have several orientations, this situation generates the perturbations and the mechanical properties at the interface will be very low, therefore the crakes will appear and the implant can be rejected. At the coated interface, the bone has a high crystallinity index and has the same orientation like the natural bone; therefore, the bond constituted presents good mechanical properties. This study reveals that it is necessary to cover implants with HAp in order to have good mechanical properties at the interface with implant and increase the lifetime of the implant. by the two methods at the interface, we have the same orientation of the HAp crystallites like the originally orientation of the tibia bone. The characterization with synchrotron radiation reveal that the texture and the crystallinity of the HAp crystallites at the interface are more intense than the measurement by neutron diffraction, and show after 60 days of regeneration that the crystallinity index of HAp at 1 mm from the interface with the implant is similar to crystallinity index of the original tibia bone (at 2 mm from the interface). The bond reconstructed at the interface with the face caoted with HAp is very strength and it is one of the most important factors indicative of the reliability of fixing the implant to the bone tissue. Ti-6Al-4V coated with HAp by PTS, offers the potential of combining the superior mechanical performance of the metal component with the excellent biological responses possible of HAp. The two techniques confirm that it is necessary to use the implant coated with HAp in order to improve the lifetime of implants.

The success of the biomaterial used in implantology necessarily depends on the interface between implant and bone, so bone restored after several months of implantation.

Therefore, in this study the high resolution of the synchrotron radiation and neutron diffraction allow to characterize the interface and show the texture effects of the new bone crystallites at the interface bone-implant on the success rate of the implant.

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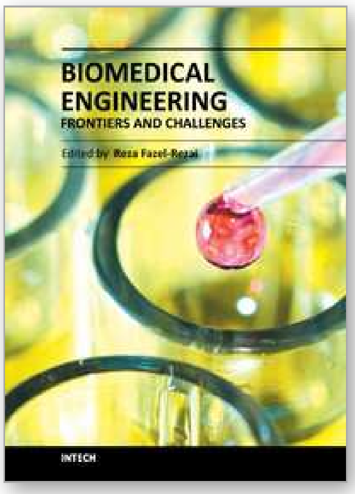

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