

The study of passive film formed on 316L stainless steel surface for orthopedic implant applications

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The characterization of passive films formed on 316L stainless steel surface implanted in a femur for 1 year is discussed according to Mössbauer Spectroscopy and scanning electron microscopy (SEM). The Mössbauer spectroscopy shown that surface layer consist mainly of oxidic species and suggested a possible apparition of a ferrite phase on surface. In according to Mössbauer Spectroscopy, SEM shown that during implantation a change in the superficial microstructure took place and a corrosion process was presently.

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1. Introduction

The degradation of metals and alloys in the human body is a combination of effects due to corrosion and mechanical activities. The orthopedic implants must sustain the human body weight, to stand to cyclic solicitations of the 3.106 cycles a year, to weariness corosions, cleaving corrosion under mechanical tension, friction, pitting, crevasse¹⁻⁶. In saline media like the human body the corrosion propagates also under the form of small pit, which give off to a significant quantity of metal ions, being very dangerous to the body and a threat to the safety of using the prosthesis. The metal ions resulted from the corrosive processes have allergic, carcinogenic and cytotoxic effects.

Metals and alloys are widely used as biomedical materials and indispensable in the medical field. The most important property of biomaterials is safety. Therefore, corrosion resistant materials such as stainless steel are employed. Metals ions released into human bodies do not always damage the bodies. The partner for combination with metals ion is very important. Every molecule has chance to combine with the ion. Reactions between the surface of metallic materials and living tissues are the initial events when the materials are implanted in human body. Tissue compatibility is governed by the reactions in the initial stage. In this regard, the surface properties of materials are important⁷⁻⁹.

In this study the behavior of surface of 316L stainless steel implanted in a femur for 1 year was studied by Mössbauer spectroscopy and scanning electron microscopy (SEM).

2. Experimental

2.1 Mössbauer Spectroscopy

Measurements were performed at room temperature in the transmission (TMS) and conversion electron spectroscopy (CEMS)¹⁰ using a conventional constant-acceleration spectrometer with a ⁵⁷Co-Rh source. The CEMS measurements were conducted with a high degree of accuracy, ensuring the same geometry of the detection space and same gas flow rate for all the samples. The information obtained by scattering method (electron conversion) is restricted to the layer to which the secondary radiation employed in the measurement can penetrate from the surface of the sample. In the ⁵⁷Fe Mössbauer spectroscopy the penetration depth maximum of conversion electron is of the order of 250 nm. The parameters of the Mössbauer spectra were calculated using a computer-fitting program, which assumed a Lorentzian line shape. The isomer shifts were referred to α -Fe.

2.2 Scanning Electron Microscopy

The morphology of 316 stainless steel surface after implant in a femur for 1 year was examined by scanning electron microscope-VEGA TESCAN.

3. Results and discussions

3.1 Mössbauer Spectroscopy

Transmission Mössbauer spectroscopy [TMS] was performed on reference test sample and conversion

electron Mössbauer spectroscopy [CEMS] measurements were performed on uncorroded test sample and of 316L stainless steel implanted in a femur for 1 year sample. All spectra (TMS and CEMS) show a central broad line, typical for austenitic stainless steels. We will present and discuss the CEMS spectra. We used for them 3 variants for data fit: single line, doublet and two lines. The best fitting of the spectra for reference sample was obtained for single line. The parameters of this line for reference of 316L stainless steel sample (Fig. 1) were following: position – 0,11 mm/s; width 0,41 mm/s; effect 11,70%. The Mössbauer parameters of this line are typical of stainless steel, which is paramagnetic at room temperature^{11,12}.

In the case of the sample implanted from the 3 variants for data input the goodness of the fit impose two line variant (Fig. 2). The relative areas of the two lines are: 27% and 73%. It is clear that during implantation a change in the superficial microstructure took place and a corrosion process was presently.

Another difference between the reference sample and implanted sample consists in a decrease of the intensity line for implanted sample to 10,60%. This proved the presence of a thin superficial layer on implanted sample. We believe that surface layer consist mainly of oxidic species^{13,14}. A slight improvement of the goodness of the fit, within the experimental errors, was obtained for implanted sample by using a sextet line near 2 lines. This fact suggests a possible apparition, in a small proportion, of a martensitic phase on surface.¹⁵

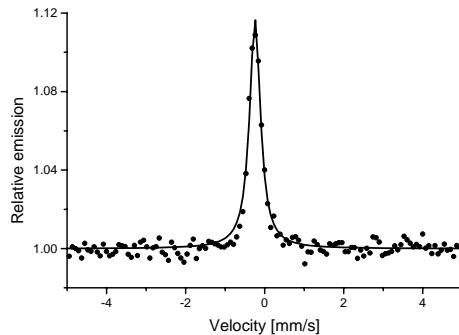


Fig. 1. Conversion electron Mössbauer spectrum for reference sample of 316L stainless steel (• data; — fit)

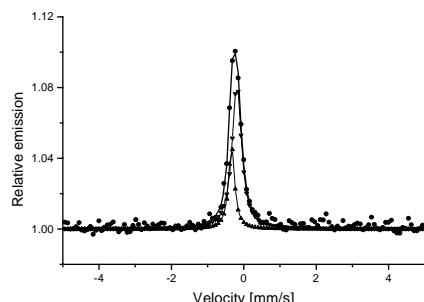


Fig. 2. Conversion electron Mössbauer spectrum of the surface after 1 year implant in the femur (• data; □ fit; —▲— line 1; —▼— line 2)

3.3 Scanning Electron Microscopy

SEM examination of stainless steel surface uncorroded and corroded in human body was carried out (Figure 3 and 4). Uncorroded sample did not show any particular feature in SEM imaging (Figure 3). The results of scanning electron microscopy for implanted sample shown evidence of corrosion spots, and the formation of an uncontinuous film on the steel surface was observed. (Figure 4). The results of scanning electron microscopy (SEM) are due to around the corrosion spots in the early stage of formation (initiation and propagation).

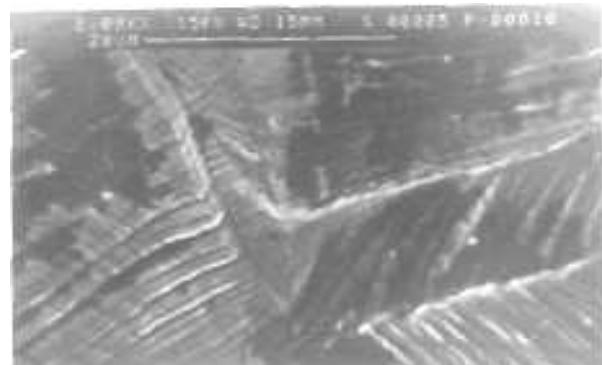


Fig. 3. Scanning electron micrograph for the reference sample of 316L stainless steel



Fig. 4. Scanning electron micrograph for the implanted sample of 316L stainless steel

4. Conclusions

Behavior of surface of 316L stainless steel implanted in a femur for 1 year was studied. Mössbauer spectroscopy proved the presence of a thin superficial layer on implanted sample, which consist mainly of oxidic species. A slight improvement of the goodness suggests a possible apparition of a ferrite phase on surface. Scanning electron microscopy shows that during implantation a change in the superficial microstructure took place and a corrosion process was presently.

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