Mechanical and histological assessment of uncoated and HA- or Ti-coated PE and POM plugs implanted in rabbits

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The surface material and surface structure of orthopaedic implants are considered to be key parameters for clinical success. The goal of this study was to assess mechanical and histological aspects of uncoated and coated polymer plugs implanted transcortically into the femurs of rabbits for 6, 9, and 12 weeks. Cylindrical plugs (diameter 3 × 12 mm) made of ultra-high molecular weight polyethylene (UHMW-PE) or polyoxymethylene (POM) uncoated or coated with hydroxyapatite (HA) or titanium (Ti) were analysed in a push-out test to determine the interface shear strength. Compared to uncoated PE plugs, coated PE implants were always significantly better in interface strength (up to a factor of 20). HA-coated PE plugs reached their final shear strength after a shorter period of implantation (at 6 weeks) than Ti-coated plugs, but both finally yielded the same strength (at 12 weeks). With a thin Ti-plasma coating, the increase of interface strength of POM plugs was much smaller than the increase found in PE implants coated with a different technique. Microscopic analysis suggested that interface failure initially occurred between coating and bone. Histology revealed a stable, bony integration of all plug types. The increase in interface shear strength could not be explained by histological findings and must be caused mainly by the different surface structures of the implants or coatings.

1. Introduction

The clinical success of an orthopaedic device is strongly related to the reaction at the implant/bone interface. Research projects [1, 2] aim at finding an optimal material composition and surface structure for improved attachment between bone and implant, and the achievement of the required long-term stability. An osteoconductive surface material is considered to be the most favourable with respect to a biocompatible interface. Additionally, a textured implant surface is a prerequisite for sufficient interface strength.

Polymer implants are used [3] to overcome the difference in mechanical properties, i.e. the stiffness between metal components and bone. However, a thin soft tissue layer may often be visible in the polymer–bone interface, which may deteriorate the stability and accelerate implant loosening. Surfaces of polymer implants can be coated with hydroxyapatite (HA) or titanium (Ti) particles, offering the potential for improvement with respect to their biocompatibility.

The goal here was to assess interfaces of various materials and coatings implanted in rabbits, in terms of mechanical and histological aspects. The interface shear strengths of HA- or Ti-coated versus uncoated polymer plugs were determined after three different periods of implantation. The failure sites at the implant/bone interface of the tested samples were analysed to assess the adhesion and strength of the coating on the substrate material. The tissue reaction at the interface of the different types of implant surfaces was investigated histologically.

2. Materials and methods

Uncoated and HA- or Ti-coated cylindrical plugs (diameter 3 mm, length 12 mm) made of ultra-high molecular weight polyethylene (PE) or copolymerized acetal resin (polyoxymethylene, POM) were implanted in rabbits for 6, 9, and 12 weeks (Fig. 1). Push-out tests were performed on the plugs according to Cook *et al.* [1] to determine the interface shear strengths.

To improve their stiffness and prevent buckling during push-out testing, the PE plugs were reinforced by a threaded Ti plug with a diameter of 1.4 mm. Some PE-plugs were then coated by hot-pressing HA granules (grain size 125–250 μm) or spongy, pure Ti powder (grain size 100–200 μm) onto the polymer surface [4]. Both types of coating resulted in a total coating thickness of 100–200 μm. Preliminary push-out tests with coated PE plugs embedded in epoxy resin were performed to determine the interface shear strength of the coating and substrate, and to have a basis for assessment of the failure mechanism after

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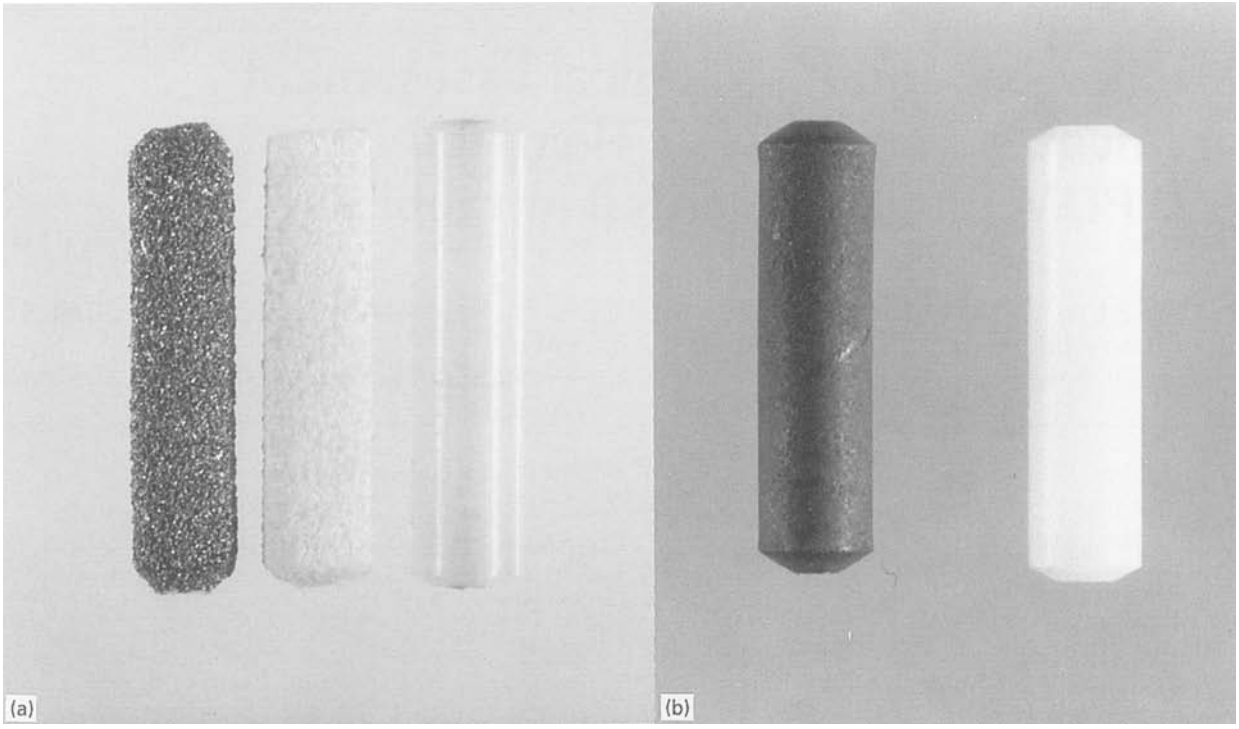
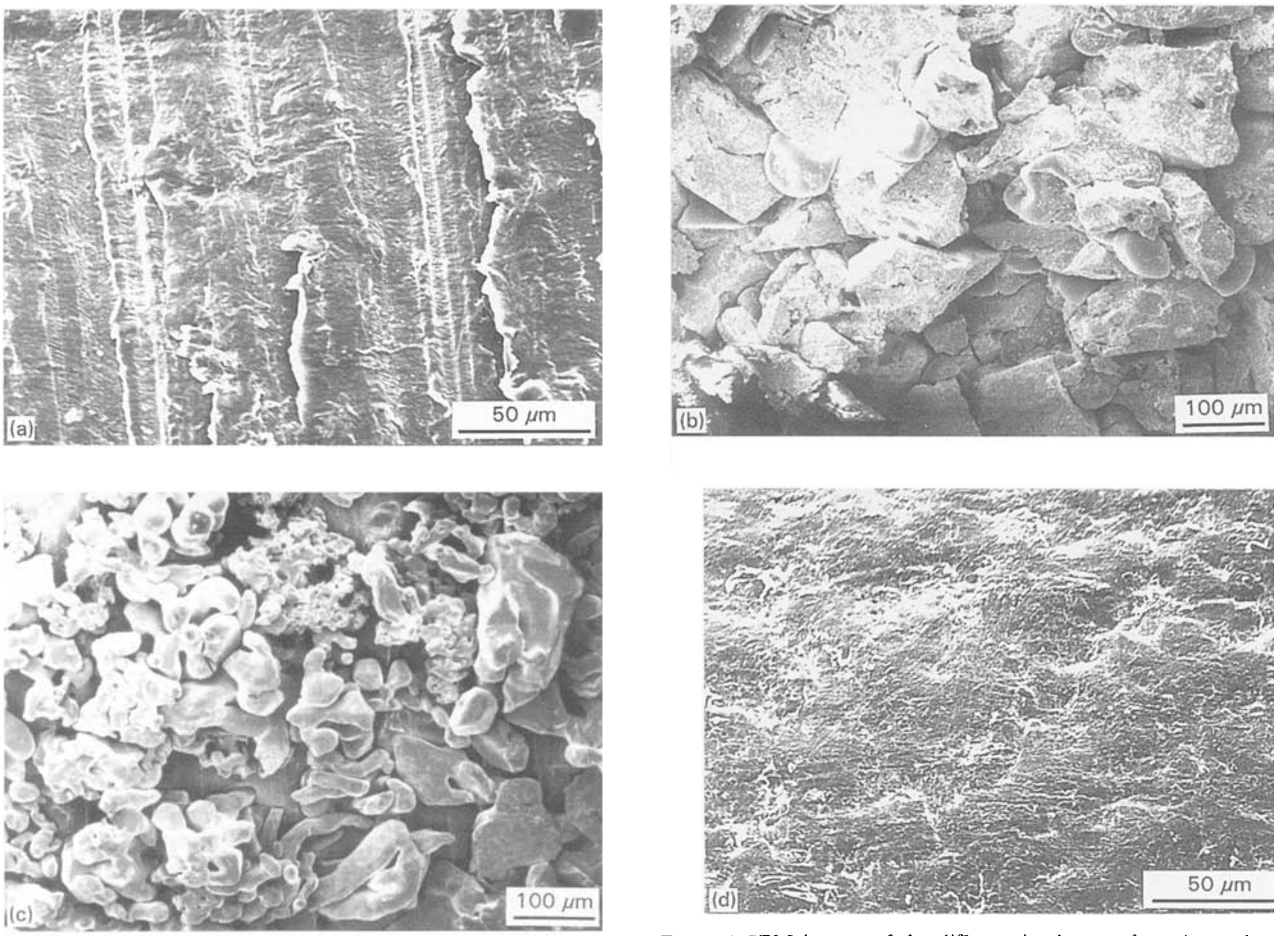


Figure 1 Coated and uncoated plugs (diameter 3 mm, length 12 mm) implanted in rabbits for three different periods of time (left to right): PE–Ti, PE–HA, PE, POM–Ti, POM.

plug implantation. The minimum strength of the coating or coating/substrate interface of PE plugs investigated was 21 N/mm^2, higher than the results of similar transcortical models with maximum push-out strengths of implanted samples being smaller than 10 N/mm^2 [1, 5] or 18 N/mm^2 [6]. Based on these results, the coating adhesion was assumed to be strong enough for the planned push-out tests. The POM implants were coated by plasma-spraying with pure Ti powder (grain size < 125 μm). The plasma-spraying process works at high temperatures and therefore melts the powder particles resulting in a coating thickness of 10–40 μm. The standard mechanical treatment renders the surface of the two uncoated plug types quite smooth in comparison to that of the coated plugs. Although distinctly rougher than uncoated plugs, the POM plug coated with Ti-plasma showed less surface roughness than the two types of coated PE plugs (Fig. 2). The final diameter of the uncoated and coated plugs was between 2.90 and 3.12 mm.

Surgery was performed on a total of 33 white New Zealand rabbits. At least six plugs of the two uncoated and three coated implant types (PE, PE–HA, PE–Ti, POM, POM–Ti) were implanted for push-out tests and two or three for histological investigation after 6, 9 or 12 weeks of implantation. Surgery on rabbits was performed under strictly aseptic conditions and general anaesthesia. Two plugs were implanted transcortically in each rabbit femur, in the latero-medial direction, 14 and 30 mm distal of the trochanter tertius. Drilling and reaming of the holes to the final diameter was done stepwise by hand to prevent heat trauma and oversize. At insertion, the fit of the implants was classified as "sliding", "snug" or "press-fit" for possible correlation with the interface strength values. No additional treatment was performed intraoperatively on the front faces of the plugs. Immediately after sacrifice of the rabbits, tubular bone samples were cut out of the femurs (Fig. 3) and embedded with dental plaster into a metal tube. Embedding included partial filling of the cavity of the bone sample without contacting the implant. This technique was used to minimize possible bending effects impairing the push-out tests. Additionally, in contrast to other studies [1], this prevented the need to saw the bone samples into two pieces for push-out testing and thereby potentially loosening the interface between implant and bone because of vibrations. Push-out tests were performed on a material-testing machine (type Zwick 1474, Zwick Ulm, Germany; Fig. 4) at a rate of 2 mm/min with immediate feed stop after passing the peak force representing a failure of the interface. After embedding the tested sample in epoxy resin, sawing and grinding it to the longitudinal mid-plane of the implant, implant/bone contact lengths of each implant were measured on the proximal and distal side at the contact zones of the cis- and trans-cortices using a microscope with an integrated scale. The mean value of the four contact lengths measured in the ground section of each implant became the basis for calculation of the effective contact area between implant and bone. The implant/bone contact area, in the shape of a cylinder, could then be determined by multiplying the circumference of the implants and the mean implant/bone contact length. The ratio of the peak force divided by the implant/bone contact area was calculated to determine a standardized interface shear strength. The one-tailed Wilcoxon test (w-test) was used for the statistical analysis. The level of significance for comparing the different coating types was always $\alpha = 0.025$.



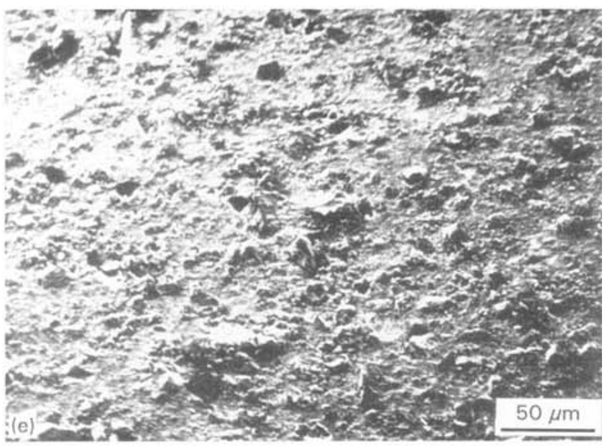


Figure 2 SEM images of the different implant surfaces (note the different magnification scales): (a) PE (500 ×); (b) PE–HA (150 ×); (c) PE–Ti (150 ×); (d) *POM* (500 ×); (e) POM–Ti (350 ×).

For the histological investigations as a control study specimens not used for push-out tests were fixed in 4% formaldehyde for at least 48 h and dehydrated by the following procedure: 1 h in 70% ethanol, twice for 3 days in 80% ethanol, twice for 3 days in 96% ethanol, 30 min in a 1:1 mixture of acetone and ethanol, and three times for 3 days in 100% ethanol. Afterwards, the specimens were embedded in methacrylate, sawn and then ground to a thickness of 20–30 μm. The sections cut perpendicularly to the implant axis at the level of bone contact, were stained at the surface according to "Levat-Laczko". The sections were etched by 0.25% formic acid for 1 min, rinsed with water and dried. Then they were stained by a solution of 0.25% azure II (Merck No. 9211), 0.25% methyleneblue (Merck No. 1283) and 0.5% Na_2CO_3 (Merck No. 6392) for 15 min, rinsed with water, dried, and stained again with 0.5% fuchsine for 2 min. The histology was assessed regarding the contact between bone and implant surface, the remaining gap size, implant particles loosened from the surface and cellular reaction to particles and implant.

3. Results

From a total of 132 implanted specimens, 116 were used for assessment. Ten plugs had to be excluded from the push-out tests because of a bony apical encapsulation of the implant. Another six specimens

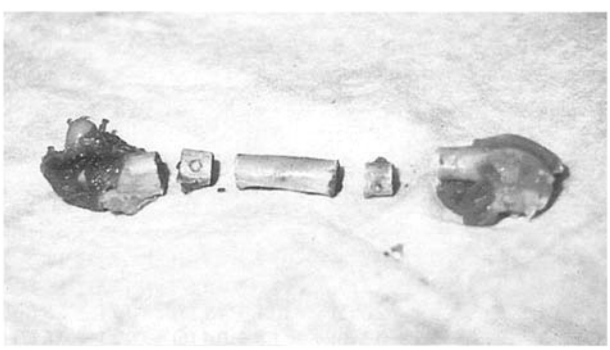


Figure 3 Tubular bone samples with implants cut out of a rabbit femur immediately after sacrifice and prior to mechanical testing.

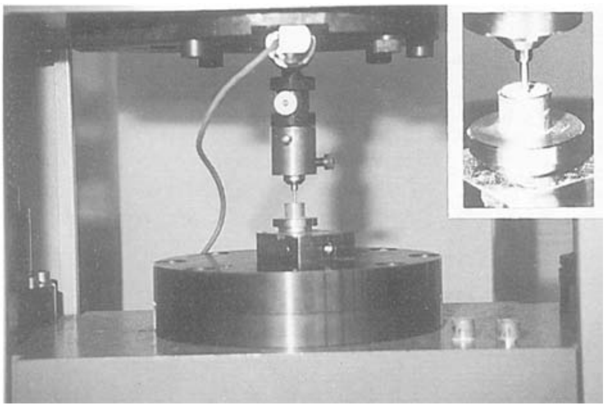


Figure 4 Experimental set-up for push-out testing with the bone sample embedded in a metal tube.

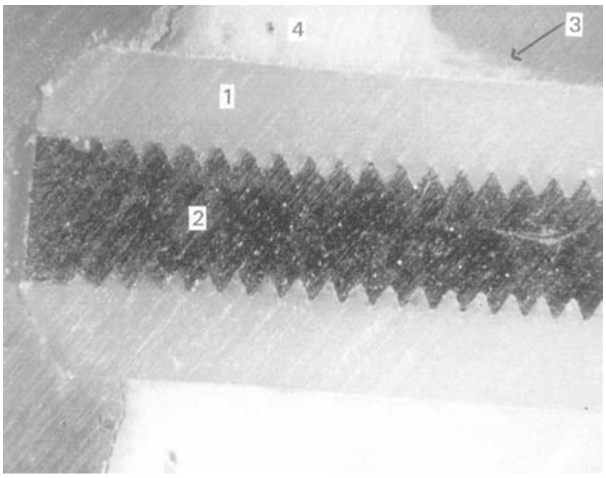


Figure 5 Longitudinal section through an uncoated PE plug (1) with reinforcement (2) showing the bone/implant contact given by the cortical femoral wall (4) and the bone apposition (3).

could not be analysed because of malposition of the plug or fragmentation of the implant during preparation of the histological section.

Five to seven specimens were used for each combination of plug type and implantation period to determine the interface shear strength values. Push-out forces were highest for HA- and Ti-coated PE plugs, with maximum values above 800 N and mean values of 733 N and 725 N after 6 and 12 weeks, respectively, for the HA coating, and 633 N after 12 weeks for the Ti coating. Microscopical analysis of sections through longitudinal planes of the femoral bone and implant showed enhanced bone formation in the interface zones (Fig. 5). Bone/implant contact areas determined

TABLE I Mean interface shear strength values and standard deviations of the five plug types implanted

Material type	Mean interface shear strength (N/mm^2) ± SD after		
	6 weeks (n)[a]	9 weeks (n)	12 weeks (n)
PE	0.5 ± 0.2 (6)	0.7 ± 0.2 (6)	0.7 ± 0.2 (7)
PE–HA	15.9 ± 3.5 (6)	14.6 ± 3.6 (7)	15.2 ± 3.0 (5)
PE–Ti	8.9 ± 2.0 (5)	10.4 ± 4.1 (5)	15.7 ± 5.8 (5)
POM	1.5 ± 0.3 (6)	1.8 ± 0.4 (6)	2.1 ± 0.8 (7)
POM–Ti	2.2 ± 0.6 (5)	2.7 ± 0.5 (5)	3.7 ± 0.6 (5)

[a] n – number of samples

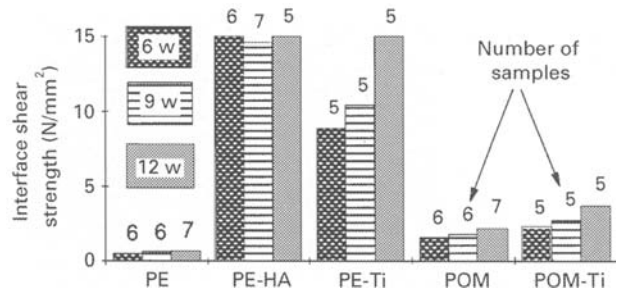


Figure 6 Graphic representation of the mean interface shear-strengths showing the coating-specific increase of the investigated samples.

on the basis of the measured implant/bone contact lengths in the longitudinal mid-planes were between 32.0 and 83.9 mm^2, with mean values for the different types and implantation periods between 41 and 58 mm^2. Compared to uncoated implant specimens, coated plugs did not show a larger contact area, and longer implantation periods did not reveal an increase in the bone/implant contact area.

Coated PE implants always showed significantly higher ($\alpha = 0.025$) interface strength (Table I and Fig. 6) than uncoated PE plugs. An increase in the interface shear strength of up to factor 20 could be determined for the coated PE plugs. Six weeks after implantation, the PE–HA plugs showed significantly higher interface strength values than PE–Ti implants. After 12 weeks of implantation, the mean interface shear strength of the PE–Ti plugs surpassed the value of the PE–HA implants without revealing a significant difference ($\alpha = 0.025$). The interface shear strengths of POM plugs coated with Ti by plasma-spraying and uncoated POM implants showed no significant difference after 6 weeks, whereas the differences were found to be significant 9 and 12 weeks after implantation. The increase in the interface shear strength of the coated versus the uncoated POM implants was much smaller than the increase due to the coating of PE plugs. In POM plugs, this increase showed a maximum factor of 1.7 after 12 weeks of implantation and was about 12 times smaller than in PE implants. The classification of the implant fit, as assessed intraoperatively, could not be correlated with the interface strength values. This confirms that the measured push-out forces are representative for the implant type and are only minimally affected by operative procedures.

Microscopic analysis of sample sections suggested that interface failure began between coating and bone; increased displacement of the plug during push-out testing also often caused failure within the coating or between coating and implant (Fig. 7). This observation is additionally supported by the fact that the measured maximum interface shear strength is of similar magnitude to the coating/substrate strength (21 N/mm^2) measured in preliminary tests.

In terms of histology, all plug types, regardless of their different surface structures, yielded a stable, bony integration into the femur without adverse tissue reactions. The Ti-coated PE plugs had less bone contact than the HA-coated PE implants. Sections with

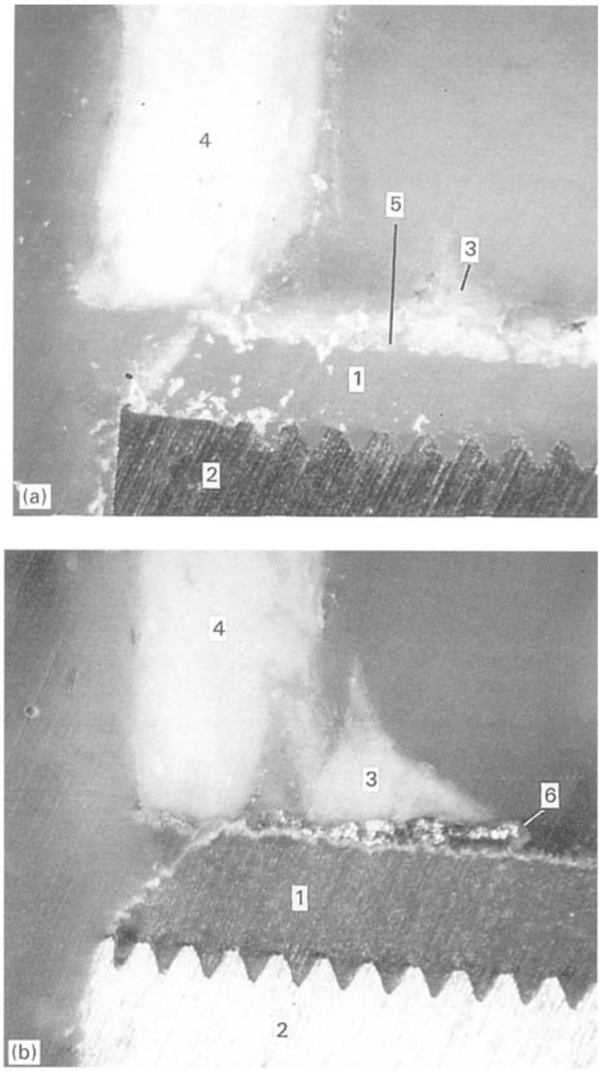


Figure 7 Location of interface failure of a HA- (a) and Ti-coated (b) PE plug. The fracture line is in the crossover zone of the bone from the cortical femoral wall to the newly formed bone wedge which adheres to the coated implant (1 = PE plug, 2 = reinforcement, 3 = bone wedge, 4 = cortical femoral wall, 5 = HA-coating, 6 = Ti-coating).

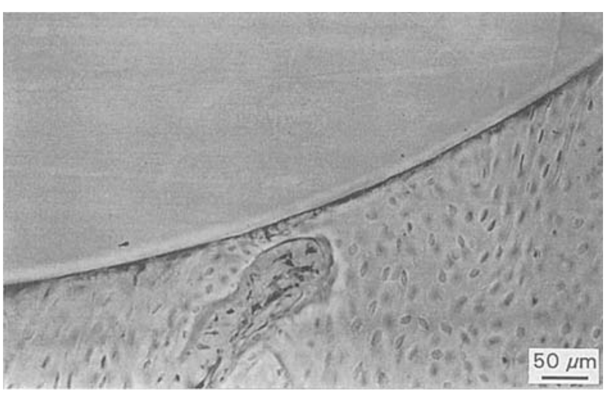


Figure 8 Bone/implant contact of an uncoated PE plug 12 weeks after implantation: no interface gap is visible in this histological section.

uncoated PE plugs seem to reveal the smallest gaps and thinnest fibrous tissue layer (Fig. 8). Detached particles were found in both types of coated PE implants. Detachment of some HA particles may have occurred during or after implantation, leading to their encapsulation in fibrous tissue. The cellular reaction

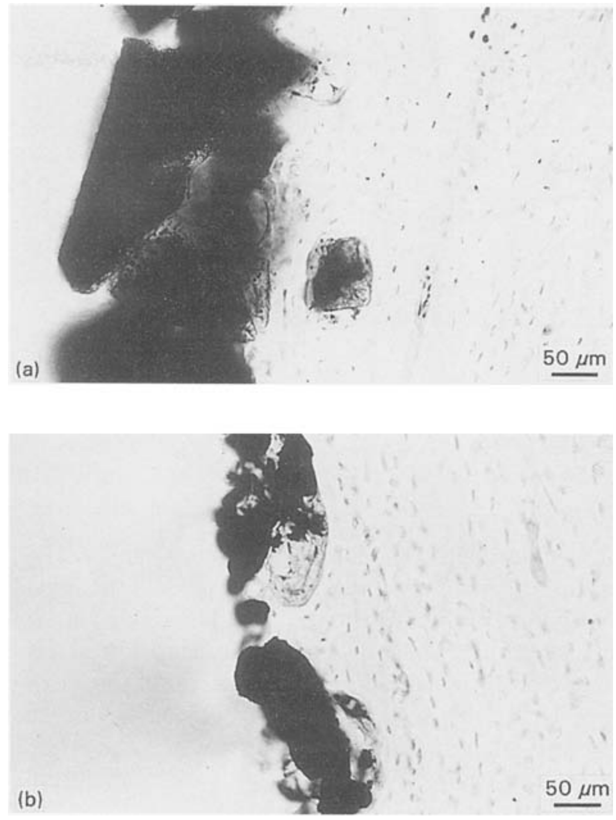


Figure 9 Bone/implant contact of HA- and Ti-coated PE plugs 12 weeks after implantation: (a) direct contact between bone and HA-coating of PE plugs showing a small cellular reaction. Detached particles are encapsulated in fibrous tissue. (b) Partial contact between bone and Ti-coating of PE plugs with cellular reactions to Ti particles.

was stronger in PE–Ti than in PE–HA (Fig. 9) implants. When compared to PE implants, the uncoated and coated POM plugs showed reduced direct bone contact and wider interface gaps. In POM implants, no improvement due to Ti-coating was visible with respect to interface conditions (Fig. 10).

4. Conclusions

The interface shear strength of HA- or Ti-coated versus uncoated PE implants was significantly higher. PE–HA plugs reached their final shear strength after a shorter period of implantation than PE–Ti plugs. Twelve weeks after transcortical implantation in rabbits, both PE coatings reached the same interface shear strength. Compared to the effect of the coating of PE implants, the increase of interface strength of POM plugs with Ti-plasma coating was negligible.

The failure at the interface revealed a considerable and sufficient attachment strength of the coating on the implant itself. Failure of PE implants initially occurred at the coating/bone interface. After large interface deformations, coating breakage could occur due to shear. Ti-plasma coated POM plugs showed a small interface strength and a relatively smooth surface compared to PE implants coated by hot-pressing: here, the failure was always located at the interface between coating and bone.

The mean interface strength values determined in this study are assumed to be lower than the effective

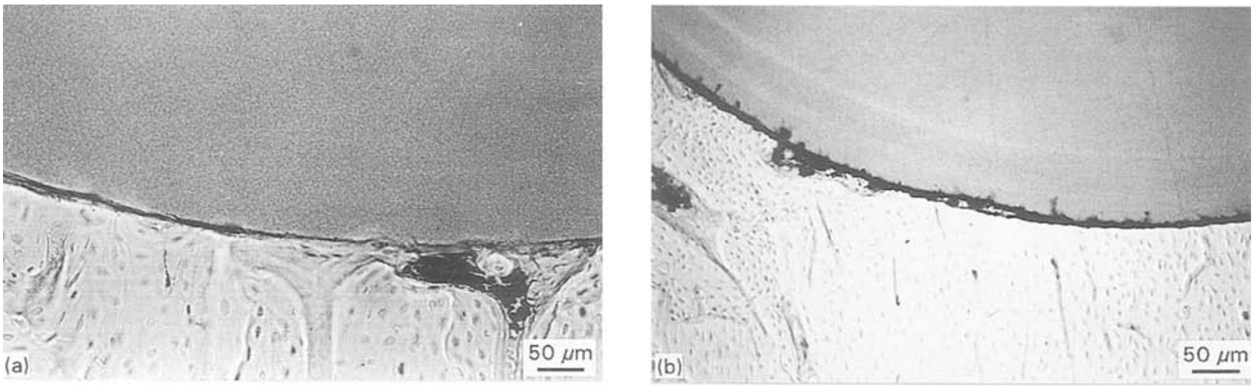


Figure 10 Bone/implant contact of uncoated and coated POM plugs 12 weeks after implantation: (a) Mainly direct contact between POM and bone showing a moderate cellular reaction. (b) Good bony contact between bone and Ti-plasma coated POM surface showing some cellular reactions due to detached particles located close to the coating. Ti particles far from the interface are due to difficulties in grinding.

interface strength. The measuring method and analysis applied here neglect the inhomogeneity of the stress distribution along the interface [7]. Thus, mean stress values are determined while failure most probably occurs locally close to the supported end of the specimen. To be able to determine absolute values of the interface strength, torsional tests are proposed [8]. Pull-out tests are still useful to compare different implant types, as done in this study.

All implant types were well integrated into bone with some cellular reactions, depending on the surface structure or material. The interface shear strength values could not be explained by the relatively small differences found in histology, and must therefore be caused mainly by the different surface roughness of the implants or coatings. The measured data are comparable to those of other studies [1, 6] and confirm the beneficial effect of rough HA- or Ti-coatings for anchoring orthopaedic implants. The importance of the surface roughness with respect to the interface strength is also known from the investigation of surface variations on metal substrates [9].

Experimental models using higher loads and less stable or even unstable [2] implants will lead to larger interface micromotion, and may hence reveal different and specific cellular findings and a variation of differences in the interface shear strengths.

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