Acta Orthop Scand 56, 491–495, 1985

Comparison of ceramic and titanium implants in cats

Elin Barth
Helge Rønningen
Ludvig F. Solheim

Institute for Surgical Research, Rikshospitalet, University of Oslo, N-0027 Oslo 1, Norway

The mechanical strengths of the bone-implant interfaces of titanium coated with glass ceramic and pure titanium implants were compared. The glass ceramic had a chemical composition similar to 45S5F Bioglass®. Cylindrical implants were placed in feline femurs. Six weeks later the strength of the fixation was tested using pull-out tests. Energy dispersive X-ray microanalysis was applied to pursue any possible relationship between mechanical strength and changes in elemental composition at the bone-implant interface. The ultimate shearing force for the pure titanium implants was higher than for the implants coated with glass ceramic. Also, the glass ceramic coating was partly degraded by the tissues. Evidently, the mechanical properties of the glass ceramic to bone interface were governed by some sort of chemical reaction that implied transfer of elements from the implant to bone. It appears that this reaction eventually will destroy the coating which makes this kind of material unsuitable for prosthesis fixation.

Numerous investigators have concluded that titanium and ceramics are biocompatible materials that can be placed in living bone for years without causing foreign material reaction. The Brånemark group introduced the concept of *osseointegration*; i.e. when no tissue interface develops between implant and host (Brånemark et al. 1969). However, ceramics are brittle when subjected to tensile stress, and they are therefore usually applied as a coating on a solid metal core. Glass ceramics have attracted attention since they are claimed to react chemically with bone (Anusavice et al. 1977, Piotrowski et al. 1975, Greenspan & Hench 1976).

We have compared the strength of fixation in bone of pure titanium and titanium coated with a glass ceramic.

Material and methods

Implants. Sixteen cylindrical plugs of commercially pure titanium were used (CP-Titanium, IMI 115, Titanium Inc., UK). The plugs were made from a 5.0 mm diameter titanium rod which was cut into 10.0 mm lengths, and a threaded hole was made in the centre of each plug. Eight of the plugs were coated with a 0.05 mm thick layer of molybdenum, followed by a 0.20 mm thick, rough layer of glass ceramic (Hadelands Glassverk, Norway) using the technique described by Barth & Herø (1985). Both ends were left uncoated. The remaining eight control plugs were sandblasted with aluminum (corundum) granules (grain size 0.1–0.5 mm). The surfaces were perthographed (Perthometer C 50 D, Mahr, West-Germany) to measure the surface roughness over a length of 4 mm. The average surface roughness of the coating (130 ± 5 μm) and the sandblasted control plugs (128 ± 7 μm) did not differ. The threaded holes were plugged with silicone and the implants gas sterilized before implantation.

Implantation. Eight adult cats (four of each sex) weighing 2.3–4.4 kg, were used. During general anesthesia with xylacine chloride 20 mg i.m. (Rompun Vet.®, Bayer) and pentobarbital 30–60 mg i.v. (Mebumal Vet.®, Rikshospitalets Apotek, Norway) the animals were operated. A hole was drilled from the lateral side in both proximal femurs using a drill bit with an outer diameter of 4.9 mm on the right and 5.4 mm on the left side. The drill bit was directed perpendicularly to the long axis of the bone and centered at the level of the lesser trochanter. The holes were vigorously irrigated with saline to remove all bone debris. Each animal received a control plug in the right and a coated plug in the left trochanter. The ends of the implants protruded 2–3 mm outside the lateral cortex. The slightly undersized holes allowed primary fixation by press fit. This did not cause any of the coating to tear off in pilot studies on fresh cadaver femurs. The femurs were radiographed postoperatively to check the positions of the implants. Benzylpenicillin 0.25 g and dihydrostreptomycin 0.25 g (Proca-mycin Vet.®, Apotekernes Laboratorium, Norway) were given on the day of surgery

and the next 2 days. The animals were killed after 6 weeks, and the proximal third of both femurs was removed and radiographed.

Mechanical tests. The bone specimens were anchored in cold-setting epoxy resin blocks (Epofix Resin, Struers Ltd., Denmark) before being mounted in the test equipment. They were freed of soft tissue except periosteum; the periosteum was an effective barrier against penetration of the resin in pilot studies on fresh cadaver femurs. The long axis of each implant was directed perpendicularly to the bottom of the epoxy resin container, leaving the lateral cortex with the implant protruding outside the resin. After hardening at 37°C for 3 h, the silicone plug was removed and each specimen mounted for pull-out testing (AB Lorentzen & Wettres Maskinaffär, Sweden). The pull-out test was performed at a constant rate of 1.0 mm/s. The peak of the load-displacement curve represented the ultimate shearing force (F). The ultimate shear stress (τ) of the implant-bone interface was computed by dividing the ultimate shearing force by surface area in contact with bone. These areas were measured using a sliding caliper (accuracy of ± 0.01 mm).

Interface analysis. The bones were split longitudinally in half through the central axis of their implant cavities. The bone and implant specimens were dried in an incubator at 60°C for 24 h. Their surfaces were then covered with carbon film, and energy dispersive X-ray microanalysis (EDXA) was performed by means of a scanning electron microscope (15 kV JSM Jeol 50, Japan) connected to an energy dispersive X-ray analyzer (EDAX 707 A, Philips, Netherlands). This method samples a depth of 0.2–2 μm within a specimen. The Kα peak-heights of calcium (Ca), phosphorus (P), silicon (Si) and sulfur (S) were measured as described by Bacon & Lifshin (1976). Each reading was based on the analysis of four standardized points on each specimen, comprising an area of 16 mm^2. Corresponding points on the implants and their surrounding bones were sampled. The Ca/P, Si/P and S/P peak height ratios were computed. Four samples of trabecular bone from cats and four unused coated implants were also prepared and analyzed as described.

Statistics. Analysis of variance was used to test differences between multiple means (Brown & Hollander 1977). The relationship between ultimate shear stress and the various Kα peak-height ratios was examined by linear regression (Draper & Smith 1981).

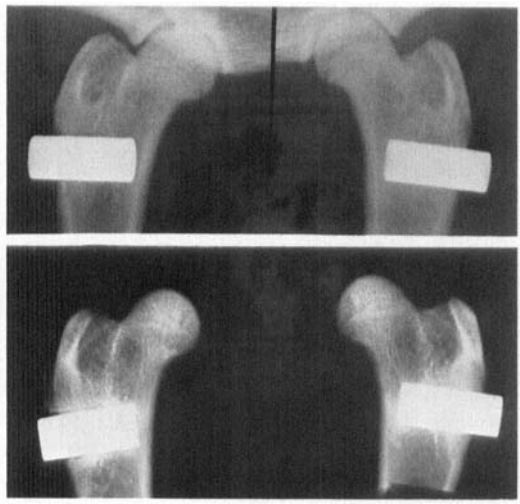

Figure 1. Implants coated with glass ceramic (left) and non-coated (right) postoperatively (top) and at killing 6 weeks later (bottom). After 6 weeks' implantation there was a radiolucent zone around the coated implant (bottom, left).

Results

There was a radiolucency around the coated implants (Figure 1). Figure 2 shows the load-displacement curves for a control and a coated implant. The ultimate shearing force was higher for control than for coated implants (Table 1). There was a notable change in appearance of the surfaces of the coated implants. The light grey color of the coating before implantation had changed to chalk-white in some areas and to black in others (Figure 3). EDXA de-

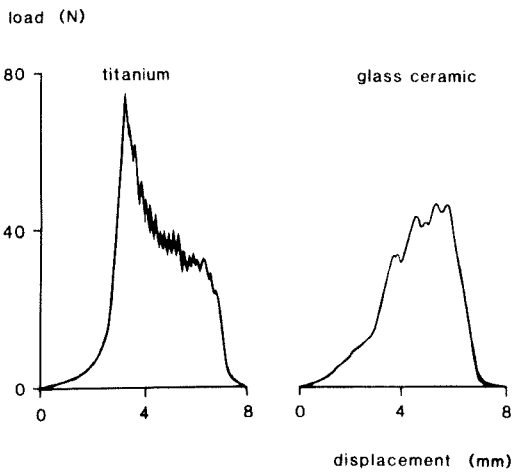

Figure 2. Typical load-displacement curves from pull-out tests of a control and a coated implant.

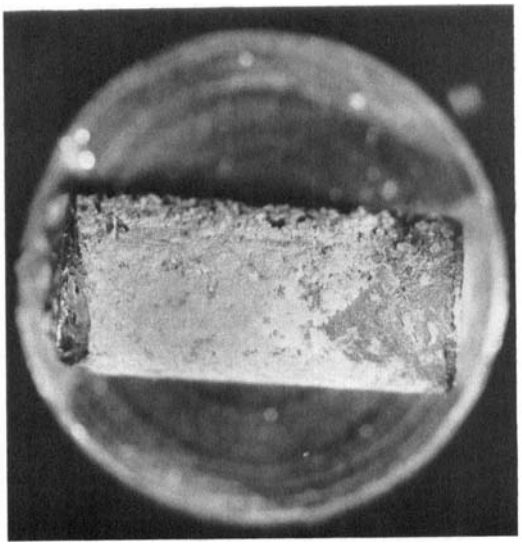

Figure 3. Patchy pattern of the surface of an extracted coated implant. The dark grey areas represent molybdenum and the light grey to white areas represent glass ceramic.

tected calcium, phosphorus and silicon representing glass ceramic in the white areas and molybdenum in the black areas. Simple linear regression analysis showed that the ultimate shearing force increased with increasing white areas on the implants ($p < 0.01$, coefficient of determination = 0.96). Thus it was considered that the mechanical properties were determined by the white areas, i.e. by areas of glass ceramic. Accordingly the ultimate shear stresses for the coated implants in the text below refer to the ultimate shearing forces divided by these areas (Table 1).

Figure 4 depicts typical results from the EDXA. The radiation of phosphorus in unused, coated implants was too low to be discernible from background radiation. However, the white areas on the used, coated implants contained a high Kα peak of phosphorus in addition to calcium, whereas the Kα peak of silicon was very small. Silicon was also found in the bone around the coated implants. This bone also contained sulfur, an element never found in the bone around the control implants. The *average* Ca/P and Si/P ratios of the white areas on the coated implants did not differ from those of the surrounding bone (Table 2). In comparison, the Ca/P ratio of the bone around control implants was higher, although below the value of the reference trabecular bone.

The ultimate shear stress (Table 3) increased with an *increasing* Si/P ratio of the surround-

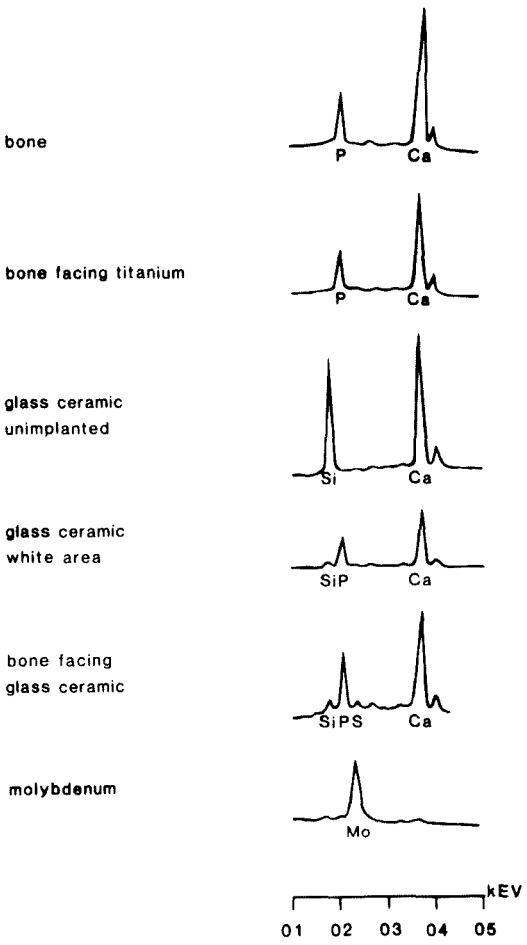

Figure 4. Typical curves of different specimens drawn from energy dispersive X-ray microanalysis (EDXA). EDXA cannot detect elements with atomic number below 12. The Kα peak-heights for phosphorus (P), silicon (Si) and sulfur (S) are given. For calcium (Ca) there is a high Kα peak and a low Kβ peak. Molybdenum (Mo) has a slightly asymmetric L-curve. The abscissa gives the energy level of radiation in kilo Electron Volts (kEV). The peak-heights of the curves reflect the intensity of radiation.

Table 1. Ultimate shearing force (F) and shear stress (τ) for the coated and the control implants. (Mean ± SD)

	F (N)	τ (MN/m²)
Glass teramic	34.5±18.2	0.6±0.3
Titanium	81.3±16.4*	0.5±0.2

* Significantly different, $p < 0.0005$.

Table 2. The Ca/P, Si/P and S/P Kα peak-height ratios for each category of specimen. The Ca/P-ratios were compared to that of bone. (Mean ± SD)

Category	Ca/P	Si/P	S/P
White area of glass ceramic coat	1.5±0.2	0.4±0.3	–
Bone that had faced glass ceramic	1.5±0.1	0.7±0.3	0.7±0.1
Bone that had faced titanium	2.2±0.3 *	–	–
Trabecular bone	2.8±0.1	–	

* Significantly different, $p < 0.0005$.

Table 3. Linear regression analysis of ultimate shear stress using the following predictors: I. The Si/P Kα peak height ratio of bone that had faced glass ceramic. II. The Si/P Kα peak height ratio of the white areas of the glass ceramic coating.

	β0	β1	R^2	F	v1,v2	p
I	0.0 (–0.1–0.2)	0.9 (0.6–1.2)	0.95	108	1,6	<0.0005
II	1.0 (0.8–1.2)	–1.0 (–1.3–(–0.6))	0.93	74	1,6	<0.0005

The numbers in parentheses represent the 95% confidence intervals, β0 is the intercept and β1 is the slope of the regression line. R^2 is the coefficient of determination, F is the F ratio and v1,v2 are the degrees of freedom. Percentile points (p) of F (v1,v2) are given.

ing bone (slope = 0.9, coefficient of determination = 0.95) and with a *decreasing* Si/P ratio of the white areas of the coat (slope = −1.0, coefficient of determination = 0.93).

Discussion

The control implants were more firmly anchored to bone than the coated implants. For the latter there was a precise relationship between ultimate shear stress and Si/P ratios of the interface.

A 6-week observation time was chosen, assuming that the host-implant reaction is a bone repair process which can be expected to be well established at this time (Rønningen et al. 1984).

The extraction curves looked different for pure titanium and glass ceramic. The titanium curves resembled the curves obtained using implants with a rough surface, and the glass ceramic curves resembled the curves obtained with externally threaded implants (Lundskog 1972). It appears that the patchy loss of the glass ceramic coating gave the same effect as a threaded surface. The mechanical tests excluded sectioning of undecalcified bone and biomaterial *in situ*. Therefore no histological slides of the material-to-bone-interfaces were prepared.

The low Ca/P ratio of the bone around the titanium plugs compared to mature, trabecular bone might suggest the presence of immature, newly formed bone, as observed by Lundskog (1972).

The coated implants failed where glass ceramic interfaced with biological tissue. This is inferred from the presence of sulfur in the surrounding bone and the absence of sulfur on the extracted implants. The finding that ultimate shear stress *increased* with an increasing Si/P ratio in the surrounding bone and a *decreasing* Si/P ratio of the implant, i.e. Si/P ratios varied in opposite directions, also suggests a failure at the interface (Table 3). The low Ca/P ratios, the presence of sulfur in the bone cavities and the radiolucency indicate a difference in the reparative process around the glass ceramic compared to that around titanium. The sulfur may indicate a process of enchondral ossification (Simmons 1980, Triffitt 1980). This disagrees with Hench et al. (1971, 1977) who claimed that bioactive glass ceramics are in fact osseointegrated. On the other hand, our ultimate shear stresses were of the same magnitude as the values reported by Hench et al. (1977) and Ducheyne et al. (1979). The presence of sulfur can also be due to glycosaminoglycans at the surface of glass ceramics (Embery et al. 1979).

A glass ceramic coat with a rough surface was chosen in this study because a rough surface increases bioreactivity compared to a smooth surface. According to Hench & Clark (1978), a rough surface does not reduce the corrosion resistance of the coating. The patchy loss of the glass ceramic on the extracted implants did not result from tearing off during insertion of these implants. Nor is it likely that the coating had been torn off during extraction since the ultimate shear stress of the coating-

core interface exceeds that of glass ceramic-bone (Barth & Herø 1985). The regression analysis suggests that a process of corrosion is more likely. The presence of silicon in the cavities can only be explained by leaching from the glass ceramic. This does not agree with the findings of Hench & Clark (1978) who claimed that bioactive glass ceramics have a dual protective layer consisting of a calcium-phosphate-rich layer on top of a silica-rich layer. This reportedly protects against corrosion, and still allows desired chemical reactions. But clearly, local factors *in vivo* can destroy the surface film and expose the glass ceramic to progressive corrosion. Attempts to increase its corrosion resistance by modifying its chemical composition may also reduce the bioreactivity of the glass ceramic (Hench et al. 1971). The highly significant relationship between the ultimate shear stress and the Si/P ratios of either side of the interface indicates that migration of silicon may be part of this bioreactivity. Thus it appears that bioreactivity can only be achieved at the expense of corrosion resistance and *vice versa*.

We conclude that in our experimental model the implants coated with glass ceramic were not as firmly anchored to bone as the titanium implants. Therefore titanium appears to be a preferable material for prosthesis fixation.

References

Anusavice, K. J., Ringle, R. D. & Fairhurst, C. W. (1977) Bonding mechanism evidence in a ceramic-nonprecious alloy system. *J. Biomed. Mater. Res.* **11**, 701–709.

Bacon, F. & Lifshin, E. (1976) The analysis of surfaces and microstructural detail. *Nat. Bur. Stand. Spec. Publ.* Symp. No. 7 IMR, 541–573.

Barth, E. & Herø, H. (1986) Bond strength of bioreactive glass ceramic on titanium substrate. The effect of molybdenum as bond coat. *Biomaterials*, **7**, In Press.

Brånemark, P. I., Breine, U., Adell, R., Hansson, B.-O., Lindstrøm, J. & Ohlsson, Å. (1969) Intraosseous anchorage of dental prosthesis. I. Experimental studies. *Scand. J. Plast. Reconstr. Surg.* **3**, 81–100.

Brown, W. M. R. & Hollander, M. (1977) *Statistics. A biomedical introduction*, pp. 234–249. John Wiley & Sons, New York.

Draper, N. R. & Smith, H. (1981) *Applied regression analysis*, 2nd. ed., pp. 459–517. John Wiley & Sons, New York.

Ducheyne, P., Hench, L. L., Kagan, A. II, Martens, M. & Mulier, J. C. (1979) Short-term bonding behaviour of bioglass-coatings on metal substrate. *Arch. Orthop. Traumat. Surg.* **94**, 155–160.

Embery, G., Rølla, G. & Stanbury, J. B. (1979) Interaction of acid glycosaminoglycans (acid mucopolysaccharides) with hydroxyapatite. *Scand. J. Dent. Res.* **87**, 318–324.

Greenspan, D. C. & Hench, L. L. (1976) Chemical and mechanical behaviour of bioglass-coated alumina. *J. Biomed. Mater. Res.* **7**, 503–509.

Hench, L. L. & Clarks, D. E. (1978) Physical chemistry of glass surface. *J. Non-Crystalline Solids* **28**, 83–105.

Hench, L. L., Pantano, C. G., Buscemi, P. J. & Greenspan, D. C. (1977) Analysis of bioglass fixation of hip phrosthesis. *J. Biomed. Mater. Res.* **11**, 267–282.

Hench, L. L., Splinter, R. J. & Allen, W. C. (1971) Bonding mechanisms at the interface of ceramic prosthetic materials. *J. Biomed. Mater. Res.* **2**, 117–141.

Lundskog, J. (1972) Heat and bone tissue. An experimental investigation of the thermal properties of bone tissue and threshold levels for thermal injury. *Scand. J. Plast. Reconstr. Surg.* Suppl. 9.

Piotrowski, G., Hench, L. L. & Allen, W. C. (1975) Mechanical studies of the bone-bioglass interfacial bond. *J. Biomed. Mater. Res.* **6**, 47–61.

Rønningen, H., Solheim, L. F. & Langeland, N. (1984) Invasion of bone into porous fiber metal implants in cats. *Acta Orthop. Scand.* **55**, 352–358.

Simmons, D. J. (1980) Fracture healing. In: *Fundamental and clinical bone physiology* (ed. Urist, M. R.), pp. 283–330. Lippincott, Philadelphia.

Triffitt, J. T. (1980) The organic matrix of bone tissue. In: *Fundamental and clinical bone physiology* (ed. Urist, M. R.), pp. 45–82. Lippincott, Philadelphia.