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# Mechanical properties and surface analysis of retrieved zirconia hip joint heads after an implantation time of two to three years

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The physical properties of ages zirconia ceramic hip joint heads were analysed, using four zirconia heads retrieved after an implantation time of two to three years. The mechanical properties of the zirconia ceramic and the surface characteristics of the heads, i.e. roughness, microstructure, etc., were compared to those of heads before implantation. No significant change in these properties was observed after two to three years in humans. Nor was calcium phosphate observed to form on the heads.

## 1. Introduction

Ceramic materials have been used in orthopaedy for about 20 years. They exhibit significantly higher corrosion and wear resistance than metals, thereby reducing the risk of inflamation reactions of surrounding tissues. Alumina ceramics were first used for this application [1], but now zirconia is also commonly being used.

Zirconia ceramics have, in fact, been used successfully as implant materials for 9 years [2] and have a strength and toughness approximately double that of aluminas, making them less sensitive to stress concentrations when attached to metal cones [3]. Yttria-stabilized zirconias (Y-TZP) are transformation toughening materials consisting of a fine-grained tetragonal microstructure which results in outstanding mechanical properties [4]. Y-TZP ceramics have consequently been used to manufacture various geometries of hip joint heads, having diameters down to 22.22 mm, which incorporate a high degree of reliability.

Many studies have been made of the mechanical properties and low temperature degradation (LTD) of engineering Y-TZP ceramics. Only a few papers have addressed their use as bioceramics and confusing results were reported in the last few years [5, 6]. However, two recent studies performed on commercially available implant materials have clearly established the stability of Y-TZP ceramic after *in vitro* or *in vivo* ageings [7, 8]. Furthermore, as explained in the review by Lilley [9] on low temperature degradation of zirconia and in the study by Swab [10] performed on several commercial yttria-stabilized zirconia, the resistance to LTD of Y-TZP depends upon the microstructure and composition. Some of the Swab commercial materials withstood the most severe conditions between 200 and 300 °C, which indicates that these materials would be stable at 37 °C for infinitely long times.

Most of the *in vivo* experiments have been performed by implantation in animals, in which the zirconia is unstressed. These ageing experiments may not be representative of ceramics in human beings under mechanical loading. It is time now to investigate the properties of Zirconia heads implanted in humans for significant periods, which has so far never been done. The aim of this paper is to demonstrate the stability of zirconia hip joint heads (PROZYR®) by analysing the physical properties of four retrieved heads. These heads were retrieved for clinical reasons not pertaining to the heads. For instance, head 12 183 was retrieved because of the catastrophic behaviour of the titanium metal-back used, and heads 33 693 and 12 163 were retrieved for femoral osteolysis reasons: the detail is given in Table I.

## 2. Mechanical properties of retrieved zirconia heads

The flexural strength was first investigated by machining test bars (4 × 2 × 24 mm) in a 28 mm diameter head, retrieved after an implantation time of 2 years. The part number (29602), engraved at the top of the cone was easily read and owing to the traceability system used for these heads, the batch of zirconia balls (batch 15920/B) from which this head was issued was clearly identified. Similar test bars were then machined in non-aged zirconia balls from the same batch, thus having the same microstructure and chemical composition.

The flexural strength before and after ageing was measured by a three-point bending method on ten test bars, using a span of 20 mm. The results are summarized in Table II. The standard deviation, which is lower than 10% in both cases, allows the conclusion that the fracture strength of the studied zirconia ceramic remains unchanged after 2 years in the human body, and is greater than 900 MPa.



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TABLE I Details on retrieved heads and their analysis

| Retrieved head number | Implantation time (months) | Reason for retrieving | Metrological and SEM analysis | Used to machine flexural bars | Used for burst test |
|---|---|---|---|---|---|
| 29 602 | 22 | Unknown | × | × | |
| 12 182 | 24 | Degradation of metal back | × | | × |
| 33 693 | 39 | Femoral osteolysis | × | | × |
| 12 163 | 27 | Fomoral osteolysis | × | | × |

TABLE II Fracture strength of the zirconia ceramic after two years of human implantation

| Fracture strength | Before ageing Head batch 15920/B | After ageing Head 29602 |
|---|---|---|
| Mean value | 1009 | 996 |
| Standard deviation | 85 | 57 |

This result is in good agreement with a previous analysis performed on the same zirconia ceramic in both *in vitro* and *in vivo* conditions [7, 8]. However, the present clinical results are more relevant insofar as the zirconia experienced the real cyclic stress and corrosion of the human body.

In a second experiment, the burst strength of zirconia heads was likewise measure before and after implantation to obtain a direct comparison for the device. A uniaxial standard burst test was performed using titanium (TA6V) trunnions. Three 26 mm diameter retrieved heads were tested (Table I); two of long neck design and one of standard neck design. For comparison, burst test data were taken from our manufacturing statistical process control (SPC), in which three heads were burst for each head batch. The average burst load of the SPC data and burst load for the retrieved heads are presented in Fig. 1 for the two different designs.

It should be noted that in both designs the retrieved heads have burst strength slightly higher than the mean values of the SPC measurements. For the head 12 182 (long neck design), it corresponds to the high statistical limit (111 kN). For these heads, we can conclude that there is no degradation in strength after two to three years of implantation.

## 3. Surface characteristics of retrieved zirconia heads

The wear of polyethylene cups is known to control the long-term durability of artificial joints. The wear rate

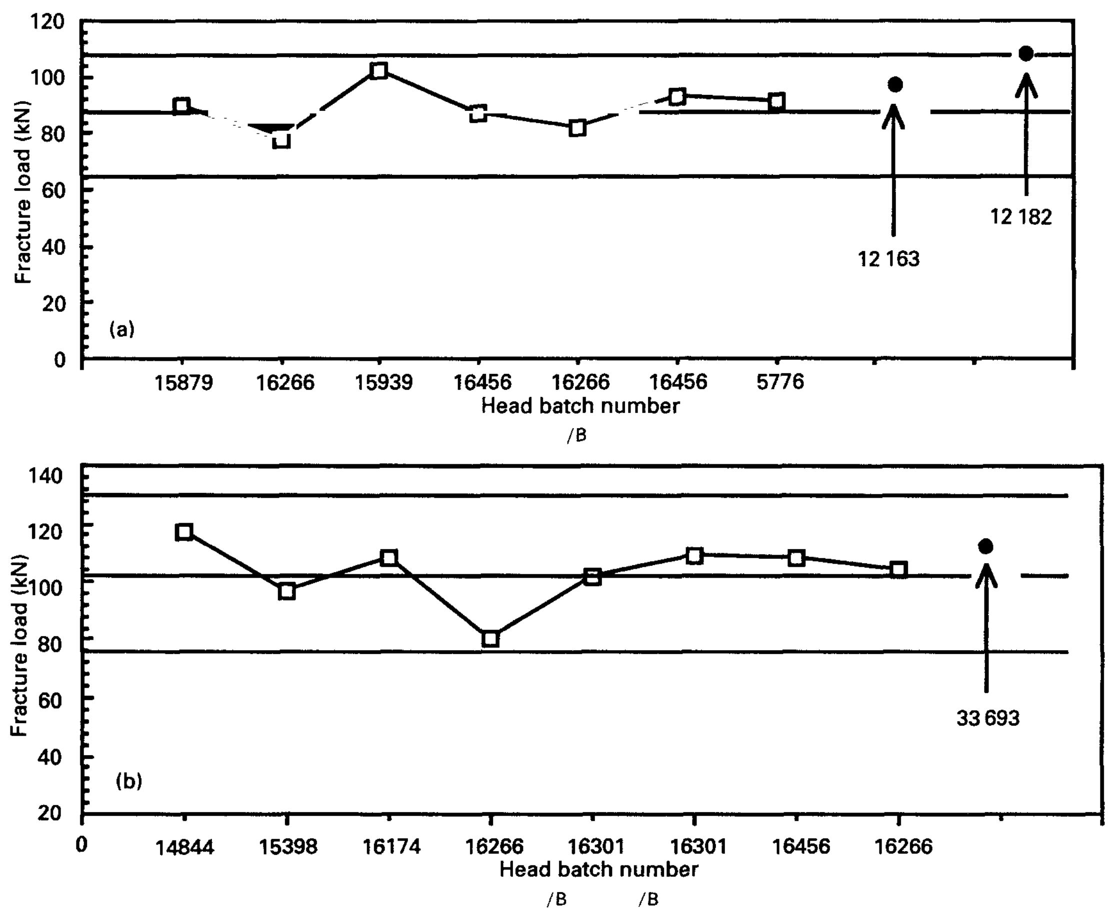


*Figure 1* Fracture strength of the retrieved heads, compared to SPC fracture strength of heads of the same design (a) design 190093-long neck; (b) design 190092-standard neck.

of polyethylene components has been found to be directly dependent on the surface characteristics of the heads [14]. Consequently, an analysis of surface properties of retrieved heads is of prime importance because it can give insight into the time dependence of wear behaviour in the joints.

Previous studies on wear behaviour of hip joint heads against UHMWPE cups with serum lubrification indicated a possible deposition of calcium phosphate on the heads during testing [11]. The calcium phosphate deposition was reduced or eliminated by addition of EDTA in the serum. Although such calcium phosphate deposition was never reported for wear experiments conducted in saline or Ringers solution, it was important to verify the presence or absence of calcium phosphate deposited on the heads after human implantation. Such a deposition, if present, would strongly affect the wear rate of polyethylene.

An investigation was performed on the four heads retrieved after 2 or 3 years in humans. The retrieved heads were analysed using scanning electronic microscopy (SEM) and associated X-ray analysis (EDAX). The sensitivity of X-ray analysis allows the detection of very thin films of calcium phosphate, of a few nanometres, if they exist.

Several areas 220 × 160 μm were analysed by the EDAX technique, searching for the presence of phosphorous and calcium. The results are identical for the four retrieved heads, and typical EDAX spectra are shown in Fig. 2. It should be noted that the spectral line for phosphorus exactly coincides with the line for zirconium, which is very intense and, for this reason, phosphorus cannot be detected. Fortunately, the spectral lines of calcium do not coincide with other lines and so a calcium phosphate layer could be detected by analysis of the calcium line. Our analysis revealed no calcium at the surface of the four retrieved heads, and consequently no detectable calcium phosphate deposition.

A tentative X-ray diffraction analysis was made on the retrieved heads. The presence of a small amount (of the order of a few per cent) of monoclinic phase was identified. However, due to the spherical shape of the samples, the results were not reproducible and the exact amount of monoclinic content could not be accurately determined by this technique. In addition,

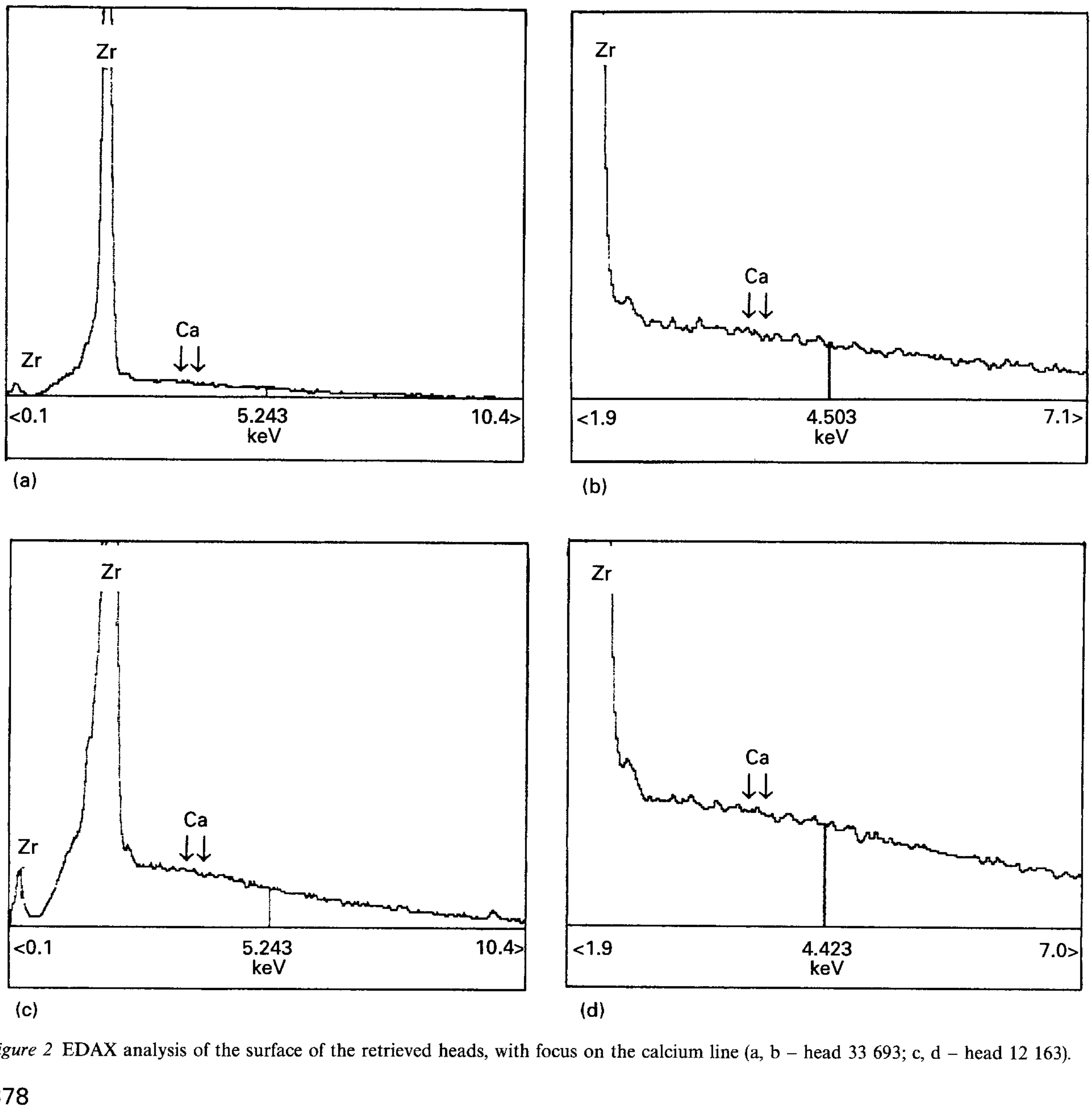


*Figure 2* EDAX analysis of the surface of the retrieved heads, with focus on the calcium line (a, b – head 33 693; c, d – head 12 163).

TABLE III Surface characteristics of retrieved heads

| Retrieved head number | Metrological parameters | Tolerances of measurements | Before Implantation | After Implantation |
|---|---|---|---|---|
| 29 602 | Diameter (mm) | ± 0.01 | 27.96 | 27.97 |
| | Cone angle | ± 1'30" | 6° 01' 20" | 6° 00' 34" |
| | Roughness (μm) | ± 0.002 | 0.012–0.016 | 0.014–0.021 |
| | Roundness (μm) | | – | – |
| 12 182 | Diameter (mm) | ± 0.01 | 25 95 | 25.97 |
| | Cone angle | | – | – |
| | Roughness (μm) | | – | – |
| | Roundness (9m) | ± 0.05 | 0.9 | 0 6–0.8 |
| 33 693 | Diameter (mm) | ± 0.01 | 25.959 | 25.966 |
| | Cone angle | ± 1' 30" | 6°01' 12" | 6°02' 00" |
| | Roughness (μm) | ± 0.002 | 0.010 | 0.015 |
| | Roundness (μm) | ± 0.05 | 0 8 | 0.7 |
| 12 163 | Diameter (mm) | ± 0.01 | 25.938 | 25938 |
| | Cone angle | | – | – |
| | Roughness (μm) | ± 0.002 | 0.012 | 0.015 |
| | Roundness (μm) | ± 0.05 | 0.5 | 0.3 |

it should be noted that the small amount of monoclinic phase results from a stress-induced transformation [12] and not from low temperature degradation (LTD), as suggested in previous papers [5, 6]. Indeed, it has been clearly shown in two recent reviews on LTD that low temperature degradation is not a general characteristic of all Y-TZP zirconia ceramics [10, 13] and must be evaluated for each Y-TZP material, since it directly depends on microstructure and $Y_2O_3$ content, i.e. on manufacturing process. A critical analysis of previous papers is included in [13]. In the case of the zirconia ceramic studied, it has been clearly established, by X-ray diffraction analysis on polished pellets aged up to 1 year in Ringer's solution, that there is no T–M transformation by low temperature degradation, and the relative change in monoclinic content is close to zero after ageing for 1 year [13].

A metrological study of the four retrieved heads was also performed to investigate any change in the surface during service in humans. The unitary traceability of these heads allowed the surface finish characteristics before implantation to be compared (in Table III) to those of the retrieved heads. The tolerances of the metrological measurements are also indicated in Table III. The roughness $R_a$ was measured using a cut-off of 0.8 mm and the roundness was deduced from two circularity measurements in two planes at 90°. No significant change of the geometrical factors (diameter, cone angle, roundness) and surface characteristics (roughness) for the four retrieved heads was found. Indeed, the variation of the metrological parameters before and after implantation are either smaller than the measurement tolerances or within the limits observed for the different head designs by statistical process control (SPC).

To complement the profilometry measurements, an SEM micrograph of the surface of retrieved head 29 602 is shown in Fig. 3. For comparison, the micrograph (at the same magnification) of the surface of heads before implantation is shown in Fig. 4. The details of the surface finish can be seen only at high magnification ( × 5000) for both implanted and non-implanted heads (Figs 3 and 4). The surfaces are very smooth and exhibit a combination of short and long and randomly distributed scratches. The features in Figs 3 and 4 confirm that there is no notable degradation of the surface finish after an implantation time of 2 years.

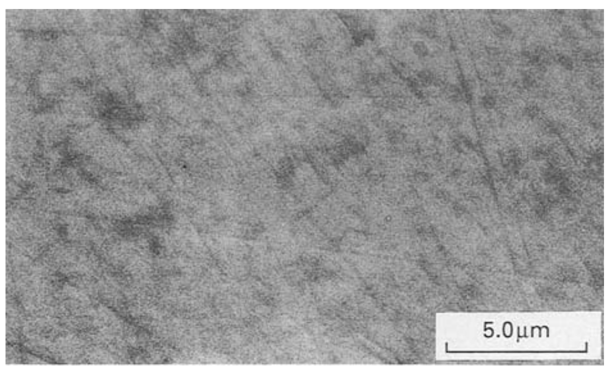


*Figure 3* Microstructures of retrieved heads at high magnification. Detail of the surface finish.

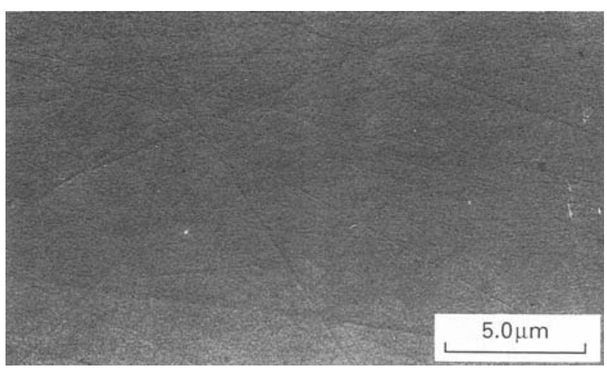


*Figure 4* Typical microstructure of a zirconia heads before implantation at high magnification, with the detail of surface finish.

## 4. Conclusions

The mechanical properties and the surface characteristics of four zirconia ceramic retrieved hip joint heads were analysed. After two to three years in humans, the fracture strength of the zirconia ceramic and the hip joint heads remain unchanged when compared to the material and heads before implantation. The same conclusion is obtained for the surface finish of the heads, i.e. there is no change in the surface roughness. Finally, no calcium phosphate deposition was observed at the surface of the retrieved heads.

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