Photoluminescence investigations on alumina ceramic bearing couples tested under different angles of inclination in a Hip joint simulator

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Aseptic loosening due to wear and dislocation of the implant represents the main complication after total hip arthroplasty. To gain more insight into the influence of the implant position on wear, commercial alumina couplings have been tested in a hip joint simulator under three different angles of cup inclination (23°, 45°, 63°) with respect to a horizontal plane. The planned length of the test was ten million cycles. However, the test was stopped at 5.5 million cycles due to the fracture of one of the femoral heads, tested with at angle of 63°. The residual stress state in the worn acetabular cups and in the fractured femoral head were evaluated by the frequency shift and broadening of the R_1 and R_2 fluorescence bands due to the Cr^{3+} ions naturally present in alumina ceramics as trace impurities. The gravimetric measurements did not show significant differences among the three different inclinations tested, in agreement with previous simulator studies, but in disagreement with *in vivo* findings. The fluorescence measurements allowed to affirm that an angle of 63° represents a worsened mechanical condition for the prosthetic component, with a consequently higher probability of fracture and/or damage potentially conducive to massive wear. In the light of the fluorescence results, it did not appear surprising that the femoral head that fractured was the one being tested at 63°.

1. Introduction

A total hip replacement (THR) is a surgical procedure where the damaged cartilage and bone of the hip joint is replaced with artificial materials. Although ultra-high-molecular-weight polyethylene (UHMWPE) has been the choice of material used as bearing surfaces in THR over the last 30 years, its wear remains the major cause of failure in long-term joint replacements. A possible reason for the high wear of polymeric materials, besides their soft behaviour, can be found in the property changes which occur in the biological environment [1–7]. Ceramic materials were introduced in orthopedics for coupling surfaces in hip prostheses to avoid the formation of polyethylene wear debris and to minimize wear [8, 9] because of their good properties, such as considerable hardness, good chemical resistance, high tensile strength, and good fracture toughness [10, 11]. Furthermore, ceramic materials present excellent biocompatibility, low coefficient of

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friction, high wear resistance [12], and good mechanical resistance at high temperatures [11, 13].

In vitro studies have demonstrated that ceramic components significantly reduce wear rate with respect to UHMWPE and metal couplings [14–17]. Wear particles released from ceramic-on-ceramic couplings are smaller than those released from conventional UHMWPE-on-metal bearings. Some tests done on the synovial tissue of revised ceramic-ceramic couplings or in the case of autopsy, have shown less inflammatory reaction and a reduction of the necrosis, compared with the ceramic-UHMWPE and metal-UHMWPE combinations [18–22].

Studies of alumina components retrieved up to 15 years after service have shown that the long-term performance of THR ceramic implants is not only material dependent, but it is mainly controlled by factors such as dimensional tolerances, global spherical conformity and, particularly, by optimal surgical alignment of the acetabular cup, in terms of inclination and anteversion [23–25]. All these factors favor load distribution, if properly regulated preventing excessive *in vivo* wear and are, therefore, acknowledged as the main technical determinants for the survival of ceramic THR implants [26].

Regarding the design of the femoral component, several aspects have been analyzed as potential risk factors for the dislocation of the prosthesis. Turner *et al.* [27] have reported that prostheses with a larger diameter of the femoral neck are more prone to dislocation. The dimension of the implant influences not only its position but also the joint contact area. The most important choice of component position with respect to joint contact area is acetabular abduction. As the acetabular abduction becomes more horizontal, the joint contact area increases. Malposition of the acetabular component in THR increases the occurrence of impingement, reduces the safe range of motion and increases the risk of dislocation [28]. The word *malposition* implies that an *optimal* or *neutral* orientation exists for the acetabular cup and the deviation with respect to this position is called malposition. However, to our knowledge, there exist no a standard to define the optimal/neutral orientation of the acetabular cup for a given patient. It is commonly accepted the existence of a "safe zone" that is defined approximately as having a cup position with an inclination of between 30° and 50° and an anteversion of between 5° and 25° [29, 30].

Actually, the effect of the acetabular cup position on wear is still a debated subject [31]. Several authors have reported that an increased angle of inclination of the ceramic acetabular cup is a possible cause of an accelerated wear phenomenon [32–39]. Others have reported a wear rate independent of the angle of inclination both in simulator studies and *in vivo* [40, 41].

To gain more insight into the influence of the implant position on wear, in this work, commercial alumina couplings were tested in a hip joint simulator under three different angles of cup inclination (23°, 45°, 63°) with respect to a horizontal plane. The planned length of the test was ten million cycles. However, the test was stopped at 5.5 million cycles due to the fracture of one of the femoral head.

To evaluate the residual stress state in the worn acetabular cups and in the fractured femoral head, we used a piezospectroscopic technique proposed in the late 1970s [42] and widely applied to the study of alumina and alumina-zirconia composites [43–51]. The piezospectroscopic effect may be defined as the shift in the frequency of a spectroscopic transition in a solid in response to an applied strain or stress. Although the spectral signal can be due to a variety of different phenomena (Raman scattering, absorption or luminescence), in this work we concentrate on the frequency shift and broadening of the R_1 and R_2 fluorescence bands as a function of stress. The reason for this is that the fluorescence signal is several orders of magnitude greater in intensity than the Raman bands (see Fig. 1) and hence more precise measurements

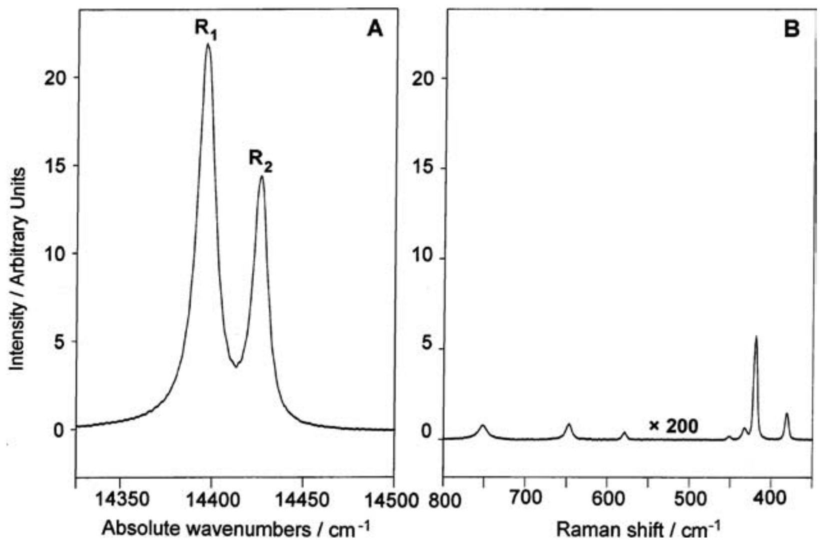

Figure 1 The R_1 and R_2 fluorescence bands (A) of Cr^{3+}-doped alumina are several orders of magnitude greater in intensity than the Raman spectrum of alumina (B).

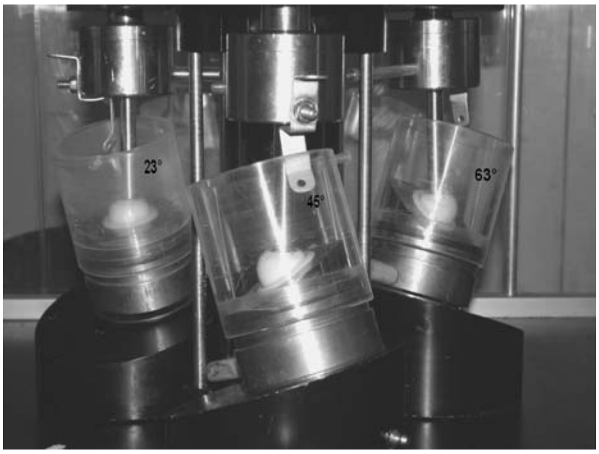

Figure 2 Set-up of the wear test; three couplings with the three different angles of cup inclination are shown.

specimens due to absorption. This procedure is recommended by ISO 14242 – Part 2 (2001).

The alumina acetabular cups and femoral heads were commercial components for surgical implants (ISO 6474), manufactured by CeramTec (Plochingen, Germany) and supplied by Wright Cremascoli Ortho (France).

can be made. The fluorescence, which derived from the red photoluminescence of ruby, is due to the radiative electronic transitions of the Cr^{3+} ions, naturally present in alumina ceramics as trace impurities which substitute the Al^{3+} ions in the Al_2O_3 lattice. The origin of the piezospectroscopic effect is that when the lattice of ions surrounding the Cr^{3+} is distorted, for instance by an applied stress, the crystal field potential at the site of the Cr^{3+} ion is altered, which, in turn, alters the energies of the electronic transitions. Thus, the analysis of the fluorescence spectrum of Cr^{3+}-doped alumina can give information on the residual stress state of the sample.

2. Materials and methods

The wear behavior of nine commercial 28 mm alumina-on-alumina couplings was investigated using a hip joint simulator. Another three acetabular cups were stored (non-loaded) in bovine calf serum as control specimens, in order to estimate the total change in mass of the tested

2.1. Hip simulator wear test details

The acetabular cups were mounted onto the simulator with three different angles of inclination with respect to a horizontal plane: 23°, 45°, and 63°, as shown in Fig. 2. Normally, in an in-vivo situation, the surgeons fix the acetabular cups with an abduction of about 45° [29, 30, 40]. So that, with an acetabular cup fixed inclined at an angle of 45°, the force of reaction of the hip is inclined at 67° as shown in Fig. 3a [52–56]. This scenario is reproduced *in-vitro* considering an inclination of 23° with respect to a horizontal plane (Fig. 3b). At the same manner this situation is valid for the other angles of inclination. The orientation of the load axis change continually during the relative motion between the acetabular cup and the femoral head, but in our hip simulator we considered the force was a constant in a range between –23° to +23°.

The wear tests were carried out using a twelve-station hip joint wear simulator (Shore Western, U.S.A.) in bovine calf serum as lubricant. The simulator and the test procedure have been described in detail elsewhere [57]. The load profile was sinusoidal with a peak magnitude of 2030 N and a frequency of 1 Hz. The wear of the acetabular cups was determined gravimetrically in terms of weight loss using a microbalance (SARTORIUS AG, Germany) with a sensitivity of 0.01 mg and an uncertainty of ±0.10 mg. The weight loss of each acetabular cup, was measured and corrected by acetabular soak controls as previously described [57]. Each weight measurement was

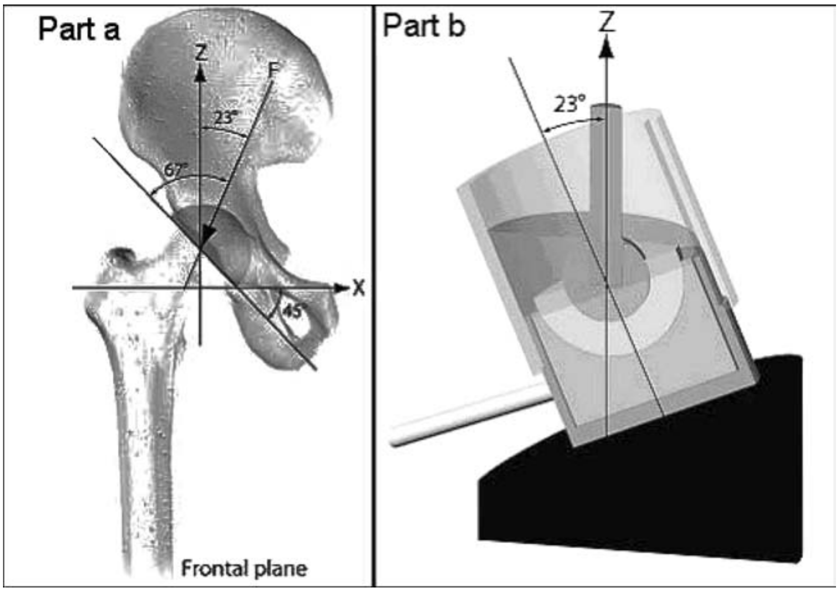

Figure 3 Schematic representation of the angles of inclination in an *in-vivo* and in an *in-vitro* scenario.

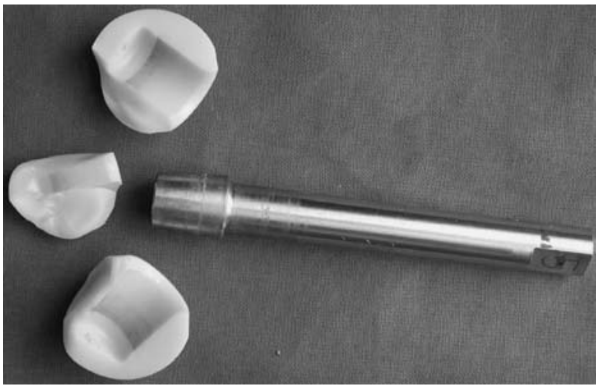

Figure 4 Photograph of the retrieved materials: femoral head fragments and stainless steel jig.

repeated three times and the average weight was used for calculations.

While tested at 63°, one of the alumina couplings had to be stopped at 5.5 million cycles, due to the failure of the ceramic femoral head. The retrieved specimen consisted of three fragments, as shown in Fig. 4.

To remove dust and possible particle debris, the ceramic fragments and the jig were cleaned by immersion in an ultrasonic bath filled with a suitable detergent (Clean 70—Elma GmbH, Germany), at 20°C for 10 min. The fragments and the jig were rinsed and put again in the ultrasonic bath with deionized water for additional 15 min. Therefore, they were put in an oven for 20 min where they were dried with nitrogen. The ceramic fragments were examined by non-destructive (fluorescence measurements) and destructive (scanning electron microscopy, SEM) tests.

2.2. Fluorescence spectra and SEM analysis

The fluorescence spectra were obtained using an argon ion laser (Innova Coherent 70) operating at 488 nm to excite the fluorescence and a Jasco NRS-2000 C micro-Raman spectrometer equipped with a 160 K frozen digital CCD detector (Spec-10: 100 B, Roper Scientific Inc.) to collect the excited fluorescence. To ensure that no laser heating occurred and contributed to the observed frequency shifts, all measurements were performed at a low laser power (i.e. 1 mW). Instrumental fluctuations represent another source of possible variation in the measured frequency. In order to correct for this, a characteristic neon line at 14431 cm^{-1} was used as a frequency calibration standard.

The spectra were recorded in back-scattering conditions with 1 cm^{-1} spectral resolution using an objective lens of 10× magnification; the laser spot size was larger than the grain size of the ceramics, assuring that the fluorescence was being averaged over a large number of grains. Moreover, to obtain a good representation of the stress distribution, ten spectra were collected in ten different points of each sample.

The spectra were recorded in a non-destructive way on three worn acetabular cups (one for each angle of inclination) in the inner surface near the center (in a spatial range of about 1.5 mm from the center). A soaked unworn cup was analyzed as control. As regards the fractured head, the spectra were recorded in the cross section of the fragments. An unworn femoral head was analyzed as control.

The bands monitored were at 14396 (R_1) and 14424 cm^{-1} (R_2). Their width (expressed as full width at half maximum, FWHM) and frequency were determined by fitting the experimental spectra with mixtures of Lorentzian and Gaussian functions. The fitting was done using a commercial software (OPUS 5.0, Bruker Optik GmbH, Germany). The obtained data were statistically analyzed by an ANOVA test for repeated measurements.

SEM analysis of the fractured femoral head was carried out with a Jeol JSM 5400 microscope (Tokyo, Japan).

3. Results

As reported above, the specimens did not complete the planned ten million cycles. Due to a mechanical problem, one of the femoral heads tested at an angle of inclination of 63°, fractured after 5.5 million cycles and the wear test was stopped. Except for this femoral head, no macroscopic damage was observed neither on the cups or on the heads after 5.5 million cycles.

3.1. Analysis of the acetabular cups

The total weight loss of the alumina acetabular cups tested at 23°, 45° and 63° of inclination was 0.47 ± 0.03 mg, 0.43 ± 0.05 mg, and 0.40 ± 0.03 mg, respectively. No significant difference was observed among the weight losses of the three sets of acetabular cups at a level of significance of $\alpha = 0.05$ (*t* test).

As regards the photoluminescence measurements, all the samples contained an adequate Cr^{3+} impurity level for the R_1 and R_2 bands to be recorded with a high signal-to-noise ratio (see Fig. 1A) so that precise measurements of band frequency and FWHM were assured.

The data obtained from the fitting of the experimental spectra are reported in Table I. It can easily be seen that after the test, irrespectively of the angle of inclination, the R_1 and R_2 bands did not show any significant frequency shift with respect to the control specimen. Moreover, the FWHM of the R_1 band did not significantly change, while the FWHM of the R_2 band significantly increased ($P < 0.05$), indicating a wider range of residual stress values [47]. Significant differences were observed among the FWHM values found for the R_2 band at the three different angles of inclination ($P < 0.05$): the acetabular cup tested at 63° showed the most significant change with respect to the control specimen.

3.2. Non-destructive and destructive analysis of the fractured femoral head

The three ceramic fragments were reassembled to check that no other fragments were missing (Fig. 5). No

TABLE I Frequency and full width at half maximum (FWHM) of the R_1 and R_2 bands as obtained by fitting the experimental fluorescence spectra of the samples analyzed. The data reported are mean values referring each to ten spectra

		R_1 band		R_2 band	
Sample		Frequency (± standard deviation)	FWHM (± standard deviation)	Frequency (± standard deviation)	FWHM (± standard deviation)
acetabular cups	Control	14396.1 ± 0.1	11.70 ± 0.03	14424.1 ± 0.1	9.58 ± 0.03
	Worn, 23°	14396.2 ± 0.1	11.72 ± 0.03	14424.1 ± 0.1	9.65 ± 0.03
	Worn, 45°	14396.3 ± 0.1	11.73 ± 0.03	14424.2 ± 0.1	9.63 ± 0.03
	Worn, 63°	14396.3 ± 0.1	11.74 ± 0.03	14424.3 ± 0.1	9.69 ± 0.03
femoral heads	Control	14396.1 ± 0.1	11.70 ± 0.03	14424.0 ± 0.1	9.46 ± 0.03
	Fractured	14395.9 ± 0.2	11.5 ± 0.1	14423.9 ± 0.2	9.5 ± 0.1

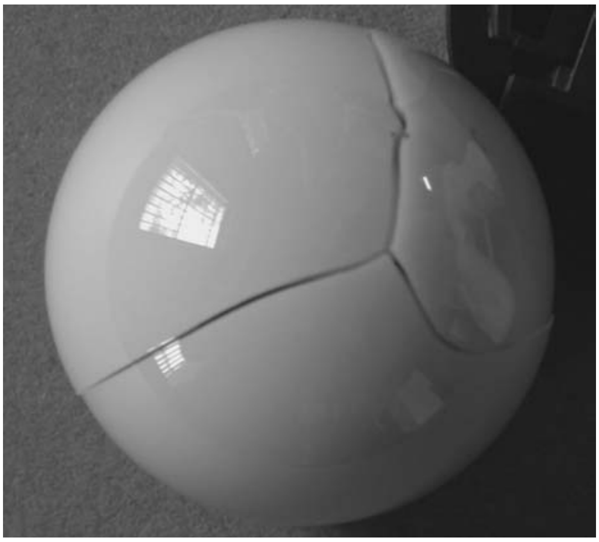

Figure 5 The three larger ceramic fragments reassembled to confirm that no other fragments were missing.

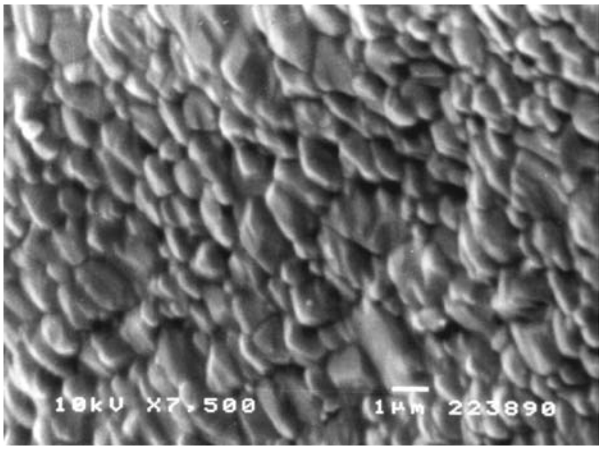

Figure 6 SEM micrograph of the grain size distribution on a ceramic head fragment.

scratches or damage on the ceramic surface fragments were observed by visual examination. As regards the jig, it was observed that the original machining marks on its tapered conical surface were intact, indicating that the metal jig did not penetrate into the bore and no mismatch in jig roundness contributed.

SEM analysis showed no evidence of anomalies in the ceramic structure, such as pores or foreign inclusions and grain size ranged between 1 and 2 μm. No fractured grains were observed (Fig. 6).

The frequency and FWHM of the R_1 and R_2 bands, as obtained from the curve fitting, are reported in the bottom of Table I. It can easily be seen that for the fractured head the R_1 and R_2 bands were not significantly shifted with respect to the control specimen. The mean FWHM of the R_1 band was significantly lower for the fractured head than for the control sample ($P < 0.05$), while the mean FWHM of the R_2 band remained practically unaltered. However, it must be observed that for the fractured head the standard deviation associated to both these mean values was higher than for the control sample (0.1 versus 0.03), indicating a higher dispersion of the FWHM values of the R_1 and R_2 bands.

4. Discussion

Published alumina wear rates measured *in vivo* appear highly variable. The discrepancies are related to material and design considerations. The first data were published more than 20 years ago [58, 59] and since that time numerous improvements have been achieved in terms of alumina quality [60]. Recent clinical studies have shown that the long-term performance of THR ceramic implants is not only material dependent; it is generally perceived that the position of the ceramic acetabular cup can have a dramatic influence on the clinical outcome, although this subject appears controversial. Clinical experiences have shown that once the prosthetic components have been properly positioned by the surgeon, the critical factor that influences the *in vivo* performance and longevity of THR is the stability of the initial cup alignment [26]. The significance of this factor is proved by the observation of the most severe wear characteristics of loosened and tilted cups [23–25, 35, 37]. Any departure from an optimal cup inclination of 45° has been reported to be associated with increased microscopic wear [38]. On a macroscopic scale, a too vertical socket has appeared to increase wear rates; in those particular situations, the contact area between the femoral head and the socket is decreased and the maximum load is transferred to the head by the edge of the socket [25, 36]. The abnormally high stress then exerted on the ceramic surfaces has been reported as responsible for the occurrence of gross wear, grain excavation, and third-body wear [23, 36, 61, 62]. On the other hand, a finite element study investigates the effects of a wide range of inclination angles from 45° to 84° and anteversion angles from 0° to 25° on the predicted contact mechanics at the articulating surfaces [31]. These authors have found no edge contact at the rim of the acetabular cup, and the effect of the acetabular cup position on the predicted maximum contact pressure has been found to be very small (less than 10%). The results of this study suggest that the edge contact and associated stripe wear observed clinically in ceramic-on-ceramic hip implants should be related to other mechanisms (such as micro-separation), rather than to the position of the acetabular cup directly. Actually micro-separation kinematics have been suggested as necessary in the simulator to duplicate the clinically relevant wear rates and patterns [63, 64].

There is also a certain controversy as to the choice of the best angle of inclination. Some authors stress that to have a good range of motion and a correct positioning of the artificial hip joint, the best angles should be in the range of between 25° and 65° [32, 65–67]. Others report that, in the case of ceramic cups, the inclination angle should be approximately 45° [32, 67, 68]. Finally, if the cup is too horizontal, flexion and abduction are greatly limited [20]; but on the other hand, with an inclination of 60°, the range of motion in flexion increases. Some authors report a wear rate independent on the angle of inclination both in simulator studies and *in vivo* [40, 41].

In order to evaluate the influence of the implant position on wear, the wear performance of the alumina-on-alumina bearing couple has been characterized in a hip joint simulator at different cup inclination angles as aforementioned in the previous sections.

Gravimetric measurements showed no significant differences among the three different inclinations tested, in agreement with the gravimetric results reported by Nevelos *et al.* [40, 41]. These authors have shown that increasing the acetabular cup angle to 60° in a hip joint simulator does not significantly affect the volumetric wear rate of 'Biolox forte' alumina. This was in contrast with the clinical results reported for 'Biolox' couples. Refior *et al.* [34] have also observed that: 'Contrary to a simulator test under normal conditions, wear may increase more than a hundredfold in case of a singular harsh repositioning even without simulation of subluxation. On the other hand, a high angle positioning of the cup from 45° to 55° causes a wear rate increase of 25 to 65%'. The clinically relevant wear rates, patterns and mechanisms of ceramic-on-ceramic bearing couples have not been reproduced in the *in vitro* simulator studies of Nevelos *et al.* [41, 69]. They have explained this discrepancy by considering that hip simulators provide ideal conditions for lubrication, as there is continuous motion. The lubricant (25% bovine serum, as in the present study) contains many proteins and lipids able to provide boundary lubrication which may protect the bearing surfaces from the more severe wear patterns clinically observed such as grain boundary fracture and hence wear. The recent results of Walter *et al.* [39] have important implications for the testing of hip prostheses; according to these authors, studies using standard hip simulators to reproduce the forces of normal walking and conclude that one million cycles equals a year of *in vivo* service are scarcely realistic. Hip simulator studies should include also edge loading if they are aimed at giving an indication of the *in vivo* performance of new bearings.

The discrepancy between the gravimetric results reported in the present study and the *in vivo* results can suggest analogous conclusions. Actually, on the basis of the trend of the FWHM found for the R_2 band, it can be stated that an angle of 63° represents a worsened mechanical condition for the prosthetic component, with a consequently higher probability of fracture and/or damage potentially conducive to the increased wear observed *in vivo* [32–39].

From this point of view, it did not appear surprising that the femoral head that fractured was being tested just at 63°. Fracture of a ceramic femoral head component has been reported as a rare but potentially serious event [70] and the *in vivo* fracture rate for 'Biolox' femoral heads manufactured after 1994 has been estimated as 0.004% [71].

The large fracture traversing from the top surface of the inner bore to its bottom (Fig. 7) may represent the starting point of the crack that caused the failure of the femoral head. The border of the fracture is irregular, with fragments, and it is not homogeneous (Fig. 8). Fig. 9 shows an elliptical trend of the fracture lines that continue up to the top surface of the border.

The fluorescence measurements are in agreement with these findings. As can be seen from Table I, the mean FWHM of the R_1 band significantly decreased in the fractured femoral head. Moreover, for both the R_1 and

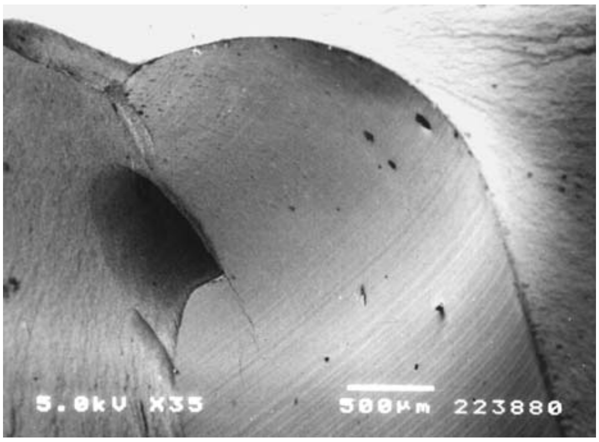

Figure 7 SEM micrograph of the fracture traversing from the top surface of the inner bore to the bottom of the bore.

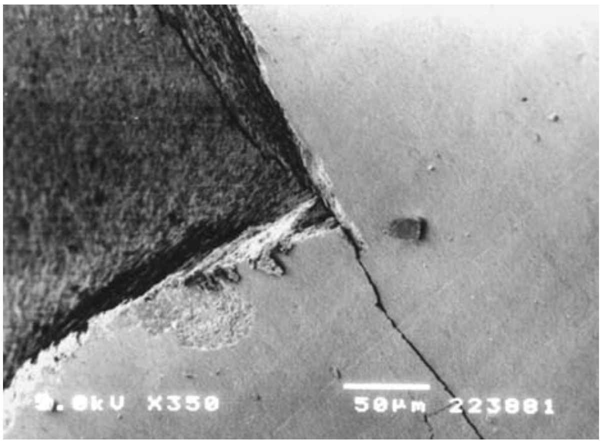

Figure 8 SEM micrograph of the fracture. The border of the fracture is not distinct, irregular and not worn.

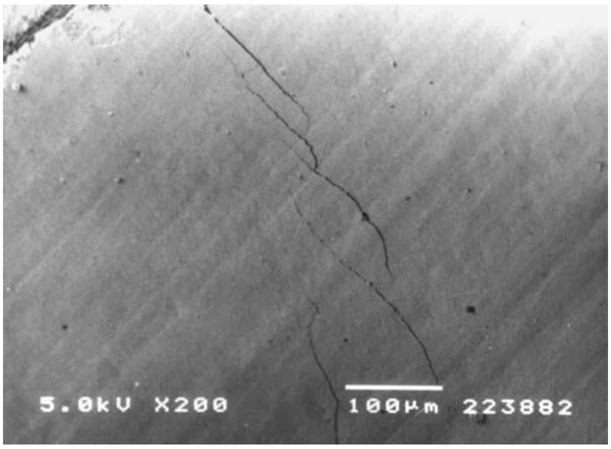

Figure 9 SEM micrograph of fracture lines with an elliptical trend.

R_2 bands, the standard deviation associated to the mean FWHM values was significantly higher than for the control sample, indicating a higher variability of these parameters within the section of the fractured femoral head. It is interesting to note that the highest decrease of the FWHM value for the R_1 band was observed in the spectra measured near the center of the bore. As an example, Fig. 10 shows the fluorescence spectra fitted into the two R_1 and R_2 components recorded on the control head (black) and in the section of one fragment of the fractured head, near the center of the bore (gray). As can be seen from the figure, the FWHM of both the R_1 and R_2 bands—even if to a different extent—are lower for the fractured head than for the control specimen. This effect can be attributed to microcracking. Actually, microcracks are known to reduce the width of the Gaussian residual stress distribution [72].

No wear signs were observed. Besides, the presence of the most significant spectroscopic changes near the center of the bore of ceramic specimen could indicate that the overstress was responsible for fracture was caused by an excessive impact between the prosthetic cone and the ball head during the initial fitting phase. The position of the ceramic femoral head and the metallic stem plays an important role in THR safety. Stress distribution and intensity in the femoral heads depend on the cone angle, on the extent of the contact and on the friction coefficient between the two mating surfaces [73]. Flaws in the finish of the tapered surfaces or a mismatch in female-to-male taper are also among the causes of ceramic femoral head failures [74]. In fact, impacting of the ceramic head onto a metal trunnion creates permanent hoop stresses in the ceramic [75] and these excessive hoop stresses in the absence of apparent damage may cause delayed spontaneous disintegration of the ceramic head even after some years [76, 77]. On the other hand, the occurrence of the fracture just in a femoral head tested at 63° would indicate that also the angle of inclination could have played a certain role: the extreme angle of inclination could have created cracks into the head that grew until final fracture.

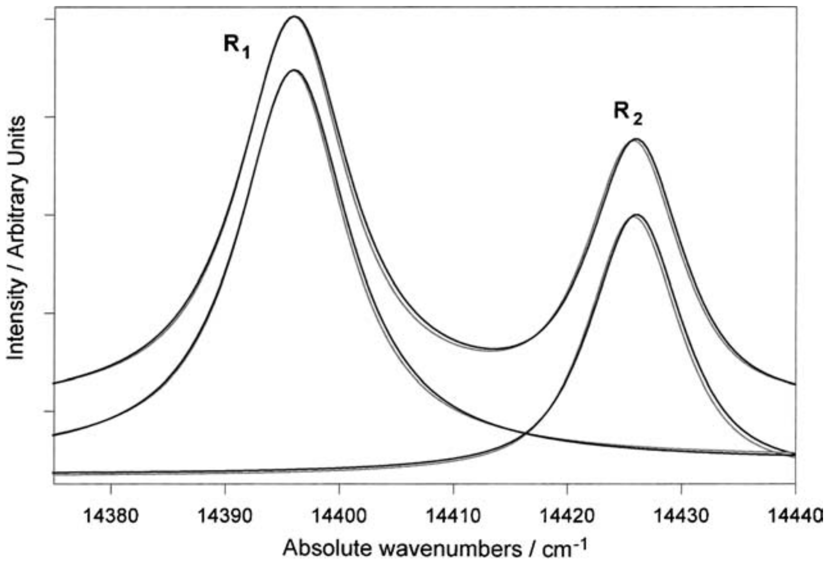

Figure 10 Fitted fluorescence spectra resolved into the two R1 and R2 components recorded on the control head (black) and in the section of one fragment of the fractured head, near the center of the bore (gray).

5. Conclusions

The gravimetric measurements did not show significant differences among the three different inclinations tested. This results are in agreement with previous simulator studies but in disagreement with *in vivo* findings. On the other hand, the fluorescence measurements allowed to affirm that an angle of 63° represents a worsened mechanical condition for the prosthetic component, with a consequently higher probability of fracture and/or damage potentially conducive to massive wear.

In the light of the fluorescence results, it did not appear surprising that the femoral head that fractured was being tested just at 63°. The piezospectroscopic technique used in the present study confirmed its validity for the evaluation of the stress state of Cr^{3+}-doped alumina samples.

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