Characterisation of alumina hip-joint wear by FIB Microscopy

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Abstract. The wear of hip-joints is a significant clinical problem, which causes adverse tissue reactions leading to bone absorption and consequent loosening of the fixation. Artificial hip joints retrieved after use and tested on simulators typically exhibit a ‘stripe’ wear area on the surface of the alumina bearing components. Focused Ion Beam (FIB) microscopy has been used to investigate the sub-surface damage mechanisms in worn alumina hip-joints for the first time. The alumina acetabular cup, both inside and outside the ‘stripe’ wear trace, has been cross-sectioned by FIB milling. The sub-surface microstructures revealed by the FIB machining, outside, inside and at the edge of the ‘stripe’ have been imaged by SEM and FIB and are compared with the microstructure of unworn bulk material. The advantage of this technique is that it enables site specific selected areas of the worn surface to be analysed.

1. Introduction:
Total hip replacement (THR) is one of the most successful applications of biomaterials, with joints survivorship commonly more than 10 to 15 years [1]. However, with the increase of younger and high demand patients, as well as an increase in life expectancies, long-term (>15 years) high performance THRs are required. The major reason for failure of THRs is due to wear, which causes adverse tissue reactions leading to bone absorption and consequent loosening of the fixation [2]. As a wear resistant material, there is renewed interest in alumina for next-generation THRs. Although over 2.5 million alumina femoral heads and nearly 100,000 alumina acetabular cups have been implanted worldwide since 1970s [3], the wear mechanism of alumina hip-joints is still unclear. It is crucial to investigate the alumina hip-joints performance and clarify the wear mechanism of alumina under body-type wear conditions.

Recently, localized high wear, known as ‘stripe wear’, has been observed on the surface of both alumina acetabular cups and femoral heads in retrieved alumina hip implants [4]. This wear has been simulated in the laboratory where the articulation includes microseparation [4]. In the present study, the surface morphology around the stripe wear zone of an alumina acetabular cup was analyzed. Focused ion beam (FIB) microscopy was applied to examine the sub-surface damage of the alumina acetabular cup around the stripe wear zone, and cross-sections of bulk unworn alumina were also obtained by FIB microscopy as a microstructural comparison.

2. Experimental procedure:
A hot isostatic pressed (HIPed) Biolox® forte alumina acetabular cup (manufacturer: CeramTec AG, Plochingen, Germany) was studied, which had been tested in a hip joint simulator against a Biolox® forte femoral head in Leeds University [5]. The nominal diameter of the cup was 28mm. This modern generation of alumina has an increased density of 3.98 g/m³, and a fine grain size of 1.8µm.
Samples for analysis were cut using an Accutom-5 (Struers, Rødovre, Denmark), and gold coating was adopted as an electrically conductive layer. The worn surface of the acetabular cup around the stripe wear zone was investigated using a JEOL 6500F FEGSEM and a Camscan SEM.

The sub-surface microstructures of the worn surface were exposed by FIB milling using a JEOL 6500F FEGSEM with Orsay Physics Ion Column FIB. A layer of gold and another layer of carbon were sputtered onto the sample surface to prevent charging during FIB processing and protect the worn surface. Tungsten deposition was then applied on the region of interest to prevent Ga⁺ implantation and sputter erosion of the top portion of the surface. The sample was tilted 55º from horizontal to make the ion beam perpendicular to the worn surface, giving a 35º angle between the imaging electron beam and the worn surface. A 700 pA Ga⁺ ion beam was used for coarse milling, and a 50 pA Ga⁺ ion beam was used to polish the sub-surface cross-sections. SEM images of the sub-surface microstructures were recorded with a 55º angle between the electron beam and the sub-surface cross-sectional planes. Ion-induced secondary electron (ISE) images of the sub-surface microstructures were recorded by tilting with a 35º angle between the ion beam and the sub-surface cross-sectional planes.

3. Results and discussions

3.1. Surface morphology

Typical surface morphology of the worn surface around the stripe wear zone on the alumina acetabular cup is shown in Fig.1. Plan view of the stripe (Fig.1a) shows the sharp boundary between the stripe wear zone (severe wear) and mild wear zone. Fig.1b is an SEM image of the worn surface about 10µm away from the stripe boundary. The original polishing marks from manufacture were still visible, alongside occasional pits (arrowed) and wear debris (white contrast). Fig.1c is an SEM image of the worn surface at the edge of the stripe wear zone, showing a transition to a rough surface with micro-fractures. There were pits along the edge of the stripe wear zone, whose dimensions are about 2µm of the order of the alumina grain size. Fig.1d shows an SEM image of the worn surface 10µm inside the stripe wear zone. In addition to the pits, many smoothly worn grains exhibiting numerous fine scratches can be observed. Abundant debris is trapped in the pits, and was also present at the edge of the stripe wear region, Fig.1c.

![SEM images of the worn surface of the alumina acetabular cup.](image)

Fig. 1 SEM images of the worn surface of the alumina acetabular cup. (a) plan view of the stripe wear zone and mild wear zone at either side; (b) worn surface outside the stripe wear zone; (c) worn surface at the edge of the stripe wear zone; (d) worn surface inside the stripe wear zone.
The sharply defined stripe on the worn surface of the alumina acetabular cup (Fig.1a) marks a transition from a mild wear region outside the stripe (Fig.1b) to a more severe surface damage within (Fig.1d). A few small pits observed outside the stripe wear zone may be due to fracture and pull out of the grain fragments. The pits inside the stripe wear zone were approximately the same size as the grain size (2 \( \mu m \)) and clear intergranular facets were also observed, consistent with previous work on explanted hip joints [6]. However, in the current work, the wear debris that obscured the fracture surface was not removed to allow subsequent FIB characterization. In any event, the loss of large fragments of alumina would have led to subsequent 3-body abrasive wear. Attrition of this wear debris would have subsequently occurred, with the resultant agglomerated debris smeared onto the surface, or accumulated in the surface pits. The edge of the stripe wear zone shows the wear transition from mild to fracture dominated severe wear. Such transitions are well known for the wear of alumina [7]. However, the full details of such a transition are not known for hip prosthetics and, since the transition defines the life of the component, further investigation by FIB microscopy was undertaken to examine the subsurface changes.

3.2. Sub-surface analyses.
Cross-sections of bulk alumina and worn surfaces around the stripe wear zone of an alumina acetabular cup were examined using FIB microscopy and SEM. Fig.2 shows SEM images of cross-sections of bulk alumina/ worn surfaces of the alumina acetabular cup, while, Fig.3 shows corresponding ISE images of the same cross-sections. The top layer is the protective tungsten deposition. Because of the non-perpendicular imaging angle, the scale bars are different between x and y directions in both SEM and ISE images.

Fig.2a and Fig.3a show respectively SEM and ISE images of the microstructure of bulk alumina. Occasional pores can be observed in the cross-section with ISE image giving a stronger pore contrast than the SEM image. Fig.2b and Fig.3b show SEM and ISE images of the sub-surface microstructure of the worn surface outside the stripe (10\( \mu m \) away from the stripe edge sectioning an occasional surface pit) ;(c) sub-surface microstructure at the edge of the stripe wear zone; (d) sub-surface microstructure inside the stripe wear zone (10\( \mu m \) away from the stripe edge). Arrowed features are discussed in the text.

Fig.2a and Fig.3a show respectively SEM and ISE images of the microstructure of bulk alumina. Occasional pores can be observed in the cross-section with ISE image giving a stronger pore contrast than the SEM image. Fig.2b and Fig.3b show SEM and ISE images of the sub-surface microstructure of the worn surface outside the stripe (10\( \mu m \) away from the stripe edge), which crossed an occasional pit (black arrow in Fig.2b and Fig.3b) on the surface. A sharp micro-crack (white arrows in Fig.2b and Fig.3b) can be seen originating at the pit. More detailed TEM work is needed to clarify the origin of fracture and pit formation mechanism in the mild wear surface.

Fig.2c and Fig.3c show SEM and ISE images of the sub-surface microstructure of the edge of the stripe wear zone. The stripe boundary occurs near the pit: left is the stripe wear zone and right is mild wear zone. There is no obvious feature in the sub-surface microstructure of the stripe edge to distinguish the stripe wear zone and mild wear zone. Again, micro-cracks (white arrows) were observed near the pits and occasional pores.
Fig.2d and Fig.3d show SEM and ISE images of the sub-surface microstructure of the worn surface inside the stripe wear zone (10 µm away the stripe edge). A higher density of pits and micro-cracks (white arrows) are observed in the chosen cross-section. All the micro-cracks observed are ≤ 2 µm beneath the surface. The profile of the cracking suggests intergranular fracture. A pit (black arrow) has been seen in both SEM and ISE images, suggesting recent fracture had occurred in the severe wear area. Further TEM investigations are necessary to reveal more details of the exact fracture mechanism.

4. Summary:
The SEM analysis of the worn surface around a stripe wear zone on an acetabular cup shows a clear boundary between the mild wear area and the severe wear area. Understanding the mechanism of the mild wear to severe wear transition is crucial to increase the life-time of the alumina acetabular cups. SEM and FIB microscopy analyses reveal the sub-surface damage around the stripe wear zone. Compared with cross-sections of bulk alumina (same composition), the sub-surface microstructure in the mild wear zone shows a population of micro-cracks associated with occasional surface pits, which were probably caused by highly localised stress build up and partial grain removal. Sub-surface microstructure inside the stripe wear zone reveals a higher micro-cracks density about 2 µm beneath the worn surface, which may be intergranular cracking. However, advanced FIB and TEM studies are needed to reveal more details and clarify the exact wear mechanism.

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