FATIGUE PERFORMANCE OF COMMERCIAL BONE CEMENTS

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ABSTRACT

High resolution imaging techniques including micro-Computed Tomography and Scanning Electron Microscopy have been combined with four-point bend fatigue testing to conduct a systematic investigation of the effect of radiopacifier particles on crack initiation and fatigue life of commercially available acrylic bone cements. Microstructural characterisation of two commercial cements of similar formulation, one radiopaque and one radiolucent,has been undertaken.

Ten specimens of radiopaque cement (CMW-1) containing barium sulphate ($BaSO_4$) radiopacifierwere tested to failure at a peak stress of either 25 MPa or 30 MPa (R-ratio = 0.1, f = 3Hz). Eleven specimens of radiolucent cement (CMW Original)were tested to failure at 35 MPa (R-ratio = 0.1, f = 3Hz). $BaSO_4$ particles in CMW-1 showed a tendency to form large agglomerates (up to 0.5 mm equivalent spherical diameter) at spatial densities of ~6 mm^{-3}. Failure in CMW-1 was dominated by crack initiation from the largest agglomerates, and fatigue life was found to scale consistently with defect size. CMW-1 demonstrated inferior fatigue performance in comparison to the equivalent radiolucent formulation. The tendency of $BaSO_4$particles to agglomerate is therefore considered to be detrimental to the fatigue performance of CMW-1 cement.

INTRODUCTION

Polymethylmethacrylate (PMMA) bone cement is used extensively in total joint arthroplasty for fixation of prostheses [1]. Fatigue failure of cement has been reported to lead to debonding at the cement-prosthesis interface and aseptic loosening of the components, ultimately requiring revision [2]. It has been shown that the microstructure of the cement construct, including pre-polymerised beads, matrix, radiopacifier particles and voids, is a factor in the development of fatigue cracks [3]. A clear understanding of the effects of each of these microstructural elements is therefore desirable if cement failure is to be minimised, whether by improved component control, mixing method, or physically-based performance prediction at the design stage.

The role of porosity in fatigue failure of cement has been widely reported [4-7]; however, the contribution of radiopacifier particles to failure of the cement mantle is less well studied, despite preliminary evidence linking particle agglomerates to crack initiation [8-9]. The present work describes a micromechanical investigation of the influence of barium sulphate on fatigue crack initiation processes using µCT and SEM techniques in conjunction with fatigue testing of conventional radiopaque and radiolucent cement formulations. Detailed three-dimensional characterisation of defect populations was performed to quantify the relative influence of barium sulphate agglomeration on cement fatigue *in vitro*.

MATERIALS AND METHODS

Two brands of conventional acrylic bone cement, CMW-1and CMW Original (Depuy CMW, Blackpool, UK) were used for the preparation of four point bend specimens. CMW-1 is a high viscosity poly(methylmethacrylate) based radiopaque cement containing 9.10 % w/w barium sulphate in the powder component. CMW Original is a high viscosity radiolucent cement manufactured to the same formulation but without the addition of a radiopacifier. Both cements are indicated for fixation of prostheses in primary and revision arthroplasty.

Each cement was mixed under vacuum (-70 KPa) using a CEMVAC integrated mixing and delivery system (Depuy, Blackpool, UK) and injected into polyethylene and PTFE moulds contained between steel plates. The assembly was clamped and the cement allowed to cure for 2 hours prior to removal. Seven bend bars, each measuring 8 x 8 x 45mm, were moulded from each 40g charge of cement.Specimens were then materialographically sectioned and polished to a surface finish of 3 µm, in order to minimise surface preparation defects.

Mechanical testing was conducted using an Instron servo-hydraulic testing machine (Instron, High Wycombe, UK). Due to the small sample size and corresponding accuracy required in positioning/loading, a self-aligning rig was developed for use in this study (Figure 1). Specimens were subjected to four-point bend fatigue testing at an R-ratio of 0.1 and frequency of 3 Hz. Tests were conducted at a maximum applied stress of 25 MPa or 30 MPa (CMW-1) and 35 MPa (CMW Original), informed by previous work [10] and the results of preliminary tests, to ensure consistent failure occurred within 10^5 fatigue cycles.

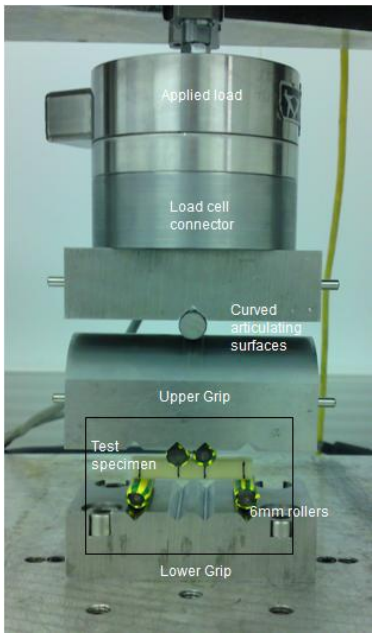

Figure 1: Self-aligning four point bend rig

The central 10mm gauge section of each specimen (Figure 2) was imaged prior to testing and post-failure using a laboratory µCT scanner (CT 160Xi, X-Tek Systems, Tring, UK) at a current of 98µA and accelerating voltage of 86kV, achieving a voxel size of ~8 µm (depending on sample width). Volume images of the specimens were generated in CT Pro Client; analysis of defects within the specimens was conducted using VG Studio Max 2.1 and Image J. This defect quantification was particularly performed to a depth of 1mm from the free surface loaded in tension (i.e. region of maximum fibre stress levels in bend) due to preliminary studies showing that crack initiation always occurred at defects present within this region.

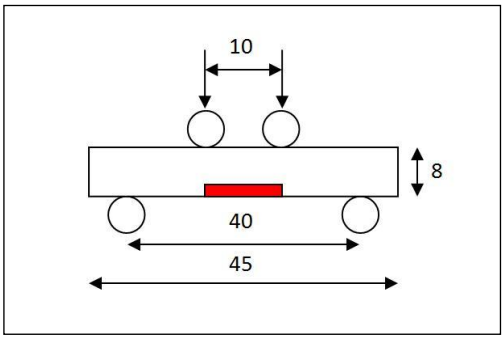

Figure 2: Schematic diagram of four point bend setup, showing centre 10 mm gauge section of interest for CT imaging.Dimensions in mm.

Fracture surfaces were imaged using a JEOL JSM6500F FEG-SEM. Specimens were sputter-coated with a thin layer (~15 nm) of gold prior to SEM imaging. A low accelerating voltage of 10 kV was used to minimise beam-induced polymer degradation.

RESULTS AND DISCUSSION

Analysis of μ-CT images allowed 3D visualisation of the distribution of microstructural defects within the gauge section of each specimen. Two types of microstructural defect were identified: voids, present in both CMW-1 and CMW Original, and barium sulphate agglomerates, present in CMW-1 specimens. Representative defect distributions for both cement types are shown in Figure 3. As individual barium sulphate particles are predominantly sub-micron in size [11], for the purposes of this study an agglomerate was defined as a cluster of barium sulphate particles with a diameter of at least 10 microns.

Agglomerates of barium sulphate particles in CMW-1 were present at spatial densities of approximately 6 per mm^3, and the largest agglomerates had an equivalent spherical diameter of over 0.3 mm. The agglomerates themselves were observed to be reasonably evenly distributed through the cement (Figure 3).This tendency of barium sulphate radiopacifier particles to form large agglomerates within the cement is consistent with the findings of other researchers [2, 8-11], although, to the authors' knowledge, no previous work has been reported detailing the frequency and size distribution of these structures.

Eleven CMW Original specimens were tested to failure at 35 MPa peak stress; failure occurred at 134,000-638,000 cycles. Crack initiation was not found to occur at defects visible at the 10 μm resolution of the CT volumes in this study. However,CMW Original demonstrated superior fatigue resistance to CMW-1, requiring a significantly higher maximum applied stress in order to induce failure within 10^5 cycles**.**

TenCMW-1 specimens were tested to failure; number of cycles to failure ranged from 73,000 – 1021,000. SEM imaging confirmed the presence of multiple large agglomerates on the fracture surfaces of CMW-1 (Figure 4), formed primarily from small particles of $BaSO_4$. The chemical composition of these features was verified using energy dispersive x-ray spectroscopy (EDX).

In all CMW-1 specimens, dominant crack initiation was found to have occurred at a large agglomerate of barium sulphate particles, close to the surface loaded in tension (within 1mm of free surface, as identified above). Detailed inspection of agglomerates indicated the initiation of multiple fatigue cracks in various directions at individual defects, with classical 'ratchet marks' running back towards the agglomerate (Figure 4).

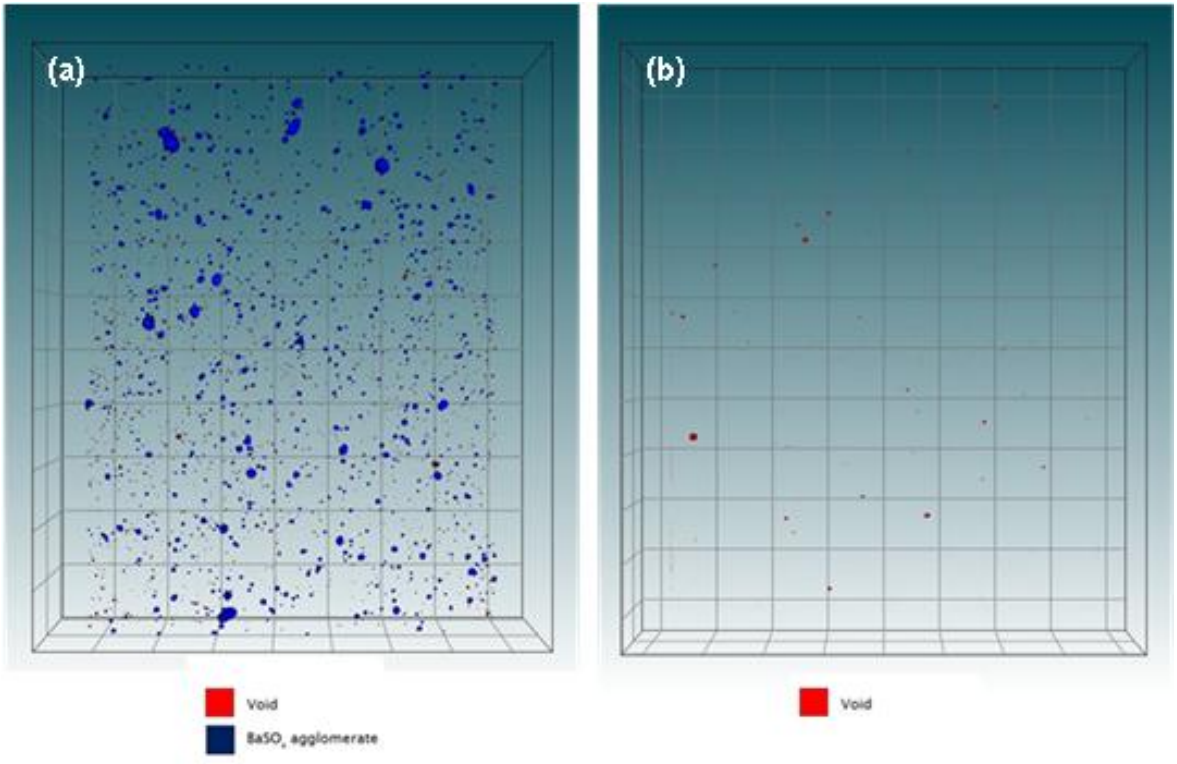

Figure 3: Representative microstructural defect distributions in (a) CMW-1 and (b) CMW Original

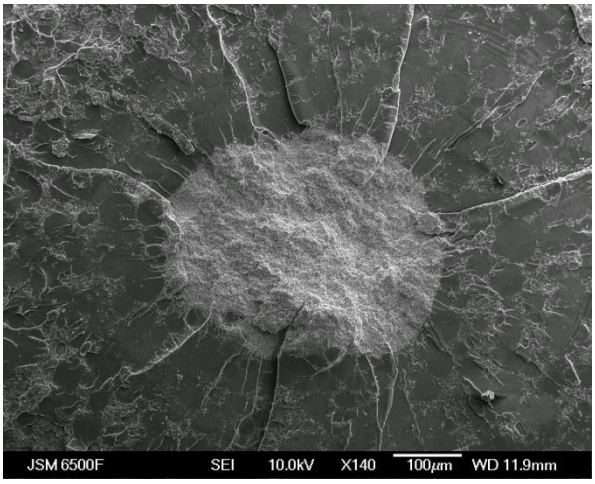

Figure 4:FEG-SEM micrograph showing multiple fatigue crack initiation from a $BaSO_4$ agglomerate in CMW-1.

CT data obtained from specimens prior to testing was used to characterise the specific crack-initiating agglomerates (CIAs), as evidenced in the post-failure FEG-SEM images (Table 1).

In each specimen, the CIA was always found to be one of the 3 largest agglomerates within the defined region of interest. Plotting fatigue test results against the defect size showed that increasing defect volume, expressed as equivalent spherical diameter, was clearly correlated with reduced fatigue life for specimens tested at either stress level (Figure 5).

This is consistent with experimental evidence of the effect of increasing pore size on fatigue life of metals [12]. In an infinite volume, the stress concentration around a smooth defect is of course independent of the defect size [13]. Near a free surface however, a larger defect would be expected to cause a high stress concentration and hence facilitate crack initiation. In addition, a larger defect will increase the rate of crack propagation, as it generates a greater effective crack length and thus a higher stress concentration factor, *K* for a given extent of physical crack growth from the defectThe scaling of fatigue life with agglomerate size observed in this study is therefore consistent with the notion that in CMW-1, barium sulphate agglomerates are the largest microstructural defects present and are thus the controlling defects in terms of the fatigue performance of the cement.

CMW-1 specimen no.	Applied stress/MPa	No. cycles to failure/1000s	Agglomerate ESD/mm	√Area in axial plane/mm	Distance from tensile surface/mm
1	25	424	0.43	0.38	0.5
2	25	73	0.34	0.31	0.0
3	25	215	0.29	0.29	0.0
4	25	236	0.31	0.29	0.0
5	25	1021	0.24	0.22	0.2
6	30	210	0.28	0.22	0.2
7	30	124	0.29	0.25	0.0
8	30	348	0.20	0.17	0.1
9	30	120	0.26	0.22	0.0
10	30	170	0.24	0.22	0.3

Table 1: Characteristics of crack-initiating agglomerates in CMW-1

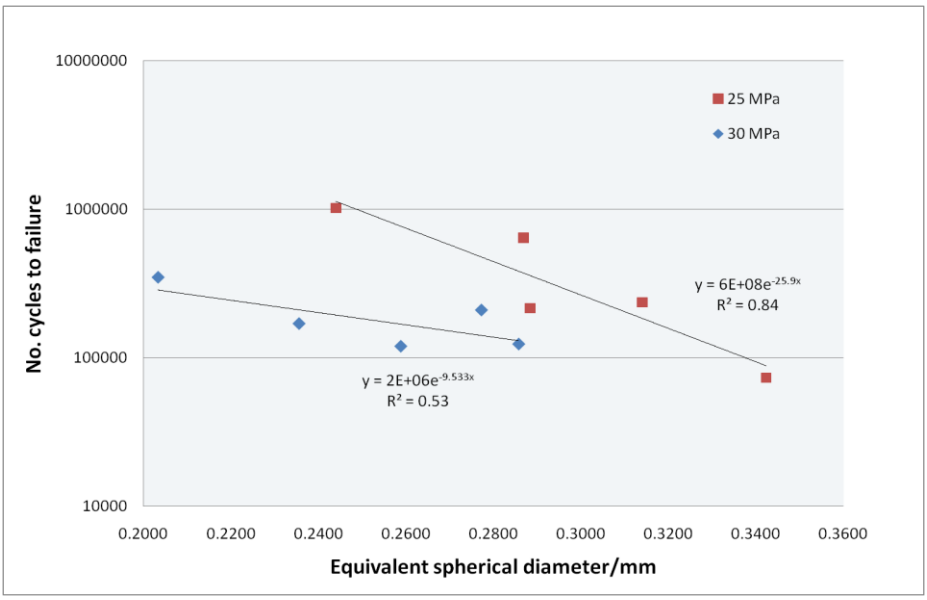

Figure 5: Relationship between defect size and fatigue life in CMW-1

CONCLUSION

The consistent initiation of fatigue cracks from $BaSO_4$ agglomerates (N = 10), despite their proximity to both large and small voids, may have significant implications for the fatigue life of bone cement, and can be viewedin terms of competition between stress concentration sites.As improved mixing methods are employed to inhibit void formation, alternative micro-structural features (such as $BaSO_4$ agglomerates) are likely to dominate failure. In formulations where these features are not present, e.g. CMW Original, failure continues to be dominated by the presence of voids. A reduced number of stress concentration sites results in a generally longer but less predictable fatigue life, which also has significant implications for the performance of cement *in vivo*.

Optimization of mixing techniques and/or cement formulations containing $BaSO_4$ may be advantageous in order to reduce the formation of agglomerates and their potential effects in-vivo. Further statistical analysis of initiation probability *vs.* size effects for all defect types may also be of considerable value.

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