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Tensile secondary creep rate analysis of a dental veneering porcelain

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ABSTRACT

Near-interface chipping of the porcelain veneer is now widely accepted as the primary failure mode of Yttria Partially Stabilised Zirconia (YPSZ) prosthesis. The origin of these failures is believed to be the complex interaction between YPSZ phase transformation, thermal expansion mismatch and other microstructural effects which induce high magnitude residual stresses within the first few microns of the interface. Recent studies have also provided evidence that these stresses, in combination with the applied sintering temperatures, are sufficient to induce tensile creep (and the associated voiding damage) within the near interface porcelain region.
In order to improve understanding of this creep rate behaviour, tensile creep has been performed on representative dental feldspathic porcelain (Vitablocs® Mark II for Cerec®) at the temperatures (650–800 °C in 50 °C increments) and stresses (50–125 MPa in 25 MPa increments) typically encountered in the near interface porcelain. Limitations on porcelain sample size meant that conventional ceramic secondary creep rate testing could not be implemented in this study and therefore a new approach based on applying multiple stresses and temperatures to a single sample was developed.
The four values of activation energy and stress rate exponent determined in this study were found to be consistent to within the 95% confidence intervals of each value. Average values and 95% confidence intervals for each parameter were determined to be $Q = 243.0 \pm 3.1$ kJ mol^{-1} and $n = 1.32 \pm 0.08$. These values compare well with published values of creep rate behaviour in silica and alumina based ceramics.
The quantitative values obtained in this study form a foundation for future studies into tensile creep induced voiding in porcelain as well as facilitating the development of improved models of the YPSZ-porcelain interface. The new single sample, tensile creep experimental approach also has potential for use in ceramic tensile creep testing of samples of limited size.

1. Introduction

Yttria Partially Stabilised Zirconia (YPSZ) is a phase transforming ceramic which has, in recent years, increasingly found use as a coping material in dental prosthesis [1]. The origin of this popularity lies in the combination of high compressive strength, biocompatibility, high toughness and chemical inertness of YPSZ along with its appealing aesthetic appearance [2]. During manufacture YPSZ copings are veneered with feldspathic porcelain which is applied using a manual multi-stage 'slurry-sinter' based process. The application of this porcelain coating serves to improve the aesthetic appearance of the prosthesis both in terms of colour and translucency, and also to reduce the hardness of the outer surface, thereby reducing wear and abrasion on existing teeth [3].

Despite these benefits, the application of porcelain to the YPSZ surface leads to the primary failure mode of these components; near-interface chipping of porcelain [4]. The origin of this failure has been the focus of extensive study [5–11] and is believed to be the combination of coefficient of thermal expansion mismatch and localised YPSZ phase transformation which results in a build-up of residual stress at this interface [12]. Both experimental measurement [13,14] and theoretical calculation [15] suggest that in the near-interface porcelain region, these stresses are tensile in nature and have magnitudes in the range of 10–200 MPa.

The combination between these locked-in forces and the 700–800 °C porcelain sintering temperatures applied to this system [16] have previously been shown to be sufficient to induce 4-point bending creep in porcelain [17]. In contrast to compressive creep, tensile creep of silica based ceramics is known to induce nano-cavitation [18] and recent transmission electron microscopy analysis of the near-interface region has demonstrated evidence for this behaviour within the first few

Abbreviations: (YPSZ), Yttria Partially Stabilised Zirconia; (RMS), Root Mean Square; (SNR), Signal to Noise Ratio; (SEM), Scanning Electron Microscopy.

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microns of the interface [19]. Despite the presence of constraining material, highly localised creep of this form is known to result in residual stress relaxation [20] which may reduce both the YPSZ-porcelain interfacial residual stress as well as the residual stress gradients across both materials.

This new insight into the near-interface porcelain behaviour requires improved understanding of the tensile creep response of dental feldspathic porcelain, which in the first instance takes the form of quantifying the secondary tensile creep rate parameters of this material. The analysis presented here has been performed over the temperature range 650–800 °C in 50 °C increments in order to be representative of the sintering temperatures applied to this system. Stresses within the range of 50–125 MPa in 25 MPa increments have also been applied in order simulate those stresses observed in porcelain at the near-interface region. This insight is necessary to facilitate improved micro-mechanical models of the impact of creep damage at this interface [21,22], along with future studies into the impact of tensile creep induced nano-cavitation on the material properties of this failure zone. These approaches will facilitate accurate quantification of the impact of the competing effects of residual stress relaxation and reduced fracture toughness on the likelihood of failure of the crept near-interface porcelain region.

The 'slurry-sinter' approach used to create veneers in which chipping has previously been observed [4] does not easily facilitate the manufacture of representative tensile creep rate specimens. To overcome this limitation, solid blocs of porcelain of almost identical elemental composition were found in the form of Vitablocs® Mark II for Cerec®. This similarity in composition is demonstrated by the comparison drawn with ZirLiner IPS e.max® Ceram, a typical near-interface veneering porcelain, as shown in Table 1. Despite the marginally different manufacturing techniques (automated layer build up vs manual layer by layer application) associated with these two types of porcelain, the matched composition ensures that similar creep rate responses should be observed in fully dense materials of the two types.

The brittle nature of porcelain and associated statistical likelihood of failure of this material means that there are significant challenges associated with creep loading this material in tension. A range of tensile creep rate studies have previously been successfully performed on similar brittle ceramics and typically these can be grouped into two main categories, hot and cold grip based approaches. Cold grip approaches rely on relatively long specimen lengths and therefore minimise the temperature of the loading grips by ensuring that these are located at distances far away from the location of heat application [23–25]. In contrast, hot grip approaches are more suitable for smaller samples and typically require cooling to be applied to the grips, thereby necessitating the many complex geometries and sample shapes which have previously been implemented [26–29].

ASTM Standard C1291 [30] is the accepted procedure for high temperature (1073–2073 K) tensile creep rate analysis of ceramics of length greater than 76.2 mm. Due to the small bloc size of Vitablocs® Mark II for Cerec® ($12 \times 14 \times 18$ mm^3) this approach was not suitable for the present study. There is an increasing trend towards creep testing smaller ceramic samples, particularly in the 10–20 mm range associated with dental implants. This presents the need for a miniaturised technique capable of testing small samples. The technique developed here represents a step towards the necessary miniaturisation of the hot grip approach to allow ceramic creep testing over the temperature ranges typically applied to dental porcelain (less than 850 °C). This approach is based on producing a region of uniform thermal and mechanical loading and the use of rounded fillets on both the sample and grips, in the spirit of the approach successfully implemented in the ASTM Standard [30]. A reduction in the sample size also has the added benefit of reducing the influence of the gradients in material properties and temperatures typically observed between the bulk and surface regions in larger samples.

Based on the work of Post et al. [31], a single sample testing approach was chosen for this new testing technique. This methodology ensured that, despite the relatively high sample failure rates observed, representative strain rates were obtained across the range of temperatures and stresses previously outlined.

2. Experimental

2.1. Materials and methods

2.1.1. Sample preparation

As part of the preliminary testing into suitable sample preparation approaches, attempts were made to manufacture tensile creep specimens which were similar in form to those previously outlined in the literature [26–30]. The limited sample size of the Vitablocs® was found to present severe difficulties in effectively holding the sample during conventional shaping processes. This resulted in repeated fracture and brittle failure of the sample and thereby severely restricted the machining modes which could be implemented.

Following these initial trials it was found that diamond grinding of the material could effectively be used to shape the sample and therefore a procedure based on this approach was developed. Multiple tensile creep specimens were manufactured from the Vitablocs® Mark II for Cerec® using this three stage optimised process. Firstly an 8 mm diameter, 18 mm long cylindrical section was milled from the $12 \times 14 \times 18$mm^3 blocks using an Eternal Tools 8 mm inner diameter diamond core drill bit. This cylinder was then mounted into a lathe using specially manufactured grips and an Eternal Tools 4 mm diameter diamond cylindrical burr (rotating on an axis perpendicular to the lathe axis) was used to profile the central section of the specimen as shown in Fig. 1. An electronic read out was used to ensure that the positioning of the polishing tool was accurate to within 1 μm. The 'dog-bone' sample shape and gentle end radius shown in Fig. 1 were selected in order to minimise stress concentration during loading of the sample. A nominal gauge section length of 6 mm long and a diameter of 3.4 mm was selected in order to induce uniform stresses in the gauge volume.

Specimens of this type were initially loaded and were found to fail (through brittle fracture) at very low loads. Scanning Electron Microscopy (SEM) of the failed surfaces revealed that this failure was initiating at the sample surface at locations where relatively high roughness was observed (on the scale of ~50 μm). Therefore, in order to reduce the surface roughness of later samples, a polishing stage was introduced into the manufacturing process. This involved initially holding grinding paper of decreasing grain size (down to 2500 grade) against the rotating, shaped sample. A diamond polishing paste of grit size 10 μm was then applied to the surface using a polishing cloth in order to further improve the surface quality.

Table 1
Manufacturer published oxide weight % tolerances for the porcelain examined in this study (Vitablocs® Mark II for Cerec®) and a 'slurry' based porcelain (ZirLiner IPS e.max® Ceram) in which near interface chipping has previously been observed. It can be seen that both materials have similar oxide compositions for SiO_2, Al_2O_3, Na_2O and K_2O. The lack of comparable data means that no firm conclusions can be drawn on the relative weight percent of other named oxides.

Oxide (% by weight)	SiO_2	Al_2O_3	Na_2O	K_2O	Other Named Oxides
Vitablocs® Mark II for Cerec®[a]	56–64	20–23	6–9	6–8	0.4–0.7
ZirLiner IPS e.max® Ceram[b]	50–60	16–22	6–11	4–8	4–15.5

[a] VITABLOCS® for CEREC®/inLab® Working Instructions, https://www.vita-zahnfabrik.com/Products/CADCAM/VITABLOCS/en/VITABLOCS-Mark-II-16964,27568,11926.html, Accessed: 10/03/2015.
[b] IPS e.max® Ceram Scientific Documentation, www.roedentallab.com/downloads/emaxceramicdata.pdf, Accessed: 19/02/15.