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Domain :

Biomaterials

Mechanical testing at the micro- and nano-scale

Micropillar Splitting for Microscale Spatially Resolved Fracture Toughness Determination

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Introduction

Yttria Partially Stabilized Zirconia (YPSZ) is a high toughness ceramic which in recent years has found increasing use in the manufacture of dental prostheses [1]. In dental applications, YPSZ copings are veneered with porcelain in order to produce an aesthetically pleasing finish. However, this leads to the primary failure mode of YPSZ prostheses; near interface chipping of the porcelain veneer [2]. The origins of this failure have been poorly understood. Recent studies have indicated that chipping is linked to the mechanical property variation and residual stress state within a few microns of the interface [3, 4]. Insights into the microscale spatial variation of fracture toughness, K_C, is therefore crucial for understanding failure.

A microscale fracture toughness approach based on nanoindentation and fracture of micropillars has recently been proposed by Sebastiani et al. [5, 6]. This approach benefits from a well-defined microscale gauge volume and can be implemented in short time, thereby facilitating high resolution fracture toughness profiling and mapping. This technique exploits the speed and precision with which FIB milling can be used to mill arrays of micropillars. The six key steps of this technique are outlined in Figure 1.

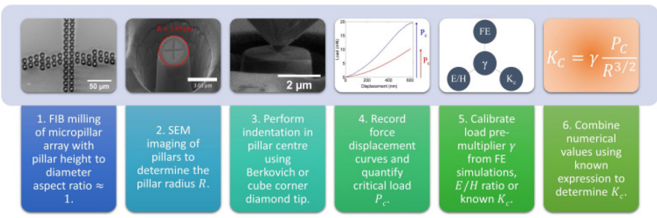

Figure 1. Flow diagram of the micropillar splitting approach

Experimental

A low speed diamond saw and multi-stage polishing process was used to prepare a cross section of the YPSZ-porcelain interface (Figure 2). FIB milling was then used to generate an array of micropillars with nominal diameter and height of 5 µm. An increased number of pillars were milled near the interface in order to obtain higher density of measurements at the location where most variation was expected. SEM imaging was then performed to determine the pillar radius and distance from the interface.

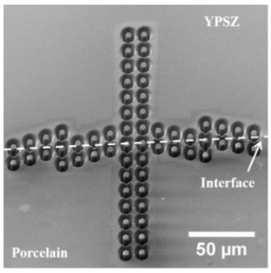

Figure 2. Micropillars across the YPSZ Porcelain interface.

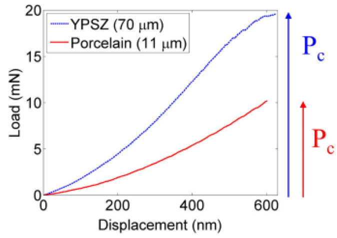

Figure 3. Typical load-displacement curves in the porcelain and YPSZ.

Nanoindentation was performed using an Alemnis Indenter (Alemnis AG) in displacement control mode in a scanning electron microscope to record the force-displacement curves as shown in Figure 3. Berkovich indentation was performed on 12 pillars far from the interface, at the location where fracture toughness was expected to be nominally uniform to measure the pre-multiplier value. In order to improve the precision of the indentation positioning, the remaining 48 indentations were performed using a cube corner tip as this provides a larger viewing angle.

Results and Discussion

The Berkovich pre-multiplier values (γ_B) for YPSZ and porcelain were determined using the bulk values of Young's modulus and hardness in order to provide estimates for the bulk fracture toughness of each material (K_B) as shown in Table 1. These results were then compared with the cube corner indentation performed at the same location, to quantify estimates of the cube corner pre-multiplier (γ_C) for each material. The remaining analysis was performed using these estimates, with an average value of porcelain and YPSZ being used for the pillars located at the interface.

Table 1. Mechanical properties and indentation load pre-multipliers for YPSZ and porcelain.

Material	E (GPa)	H (GPa)	γ_B	γ_C	K_B (MPa.m$^{1/2}$)
YPSZ	210 [7]	12.75± 0.96 [7]	0.31± 0.02	1.10± 0.05	4.79± 0.24
Porcelain	60 [8]	5.40± 0.20 [9]	0.22± 0.01	0.41± 0.04	2.65± 0.27

The distribution of fracture toughness as a function of distance from the interface is shown in Figure 5. The porcelain pillars were found to exhibit a highly brittle failure with no noticeable deviation in the loading curve before failure (Figure 3). In contrast, a gradient decrease was observed in the loading curve of YPSZ before failure. SEM videos of the indentation revealed that this was associated with small amounts of pre-cracking in the pillar as shown in Figure 4.

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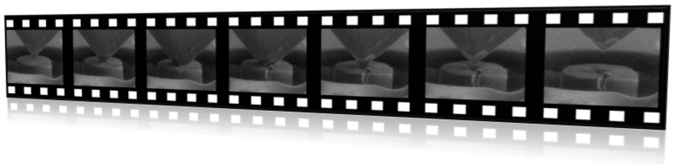

Figure 4. Stills from micrograph video of YPSZ indentation showing crack growth in the pillar before complete failure.

Post-fracture SEM imaging of the pillars revealed distinct differences in the pillar failure response as shown in Figure 5. The porcelain micropillars were found to fully fracture leaving only the highly faceted pillar stub. Preferential half-pillar cracking in a direction parallel to the interface was observed in pillars containing both YPSZ and porcelain. Differences were also observed between the cube corner and Berkovich YPSZ indentation response, with the former exhibiting highly faceted central cracking and the latter primarily displaying edge cracking.

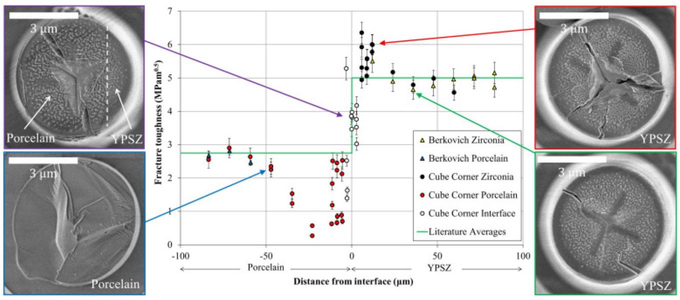

Figure 5. Fracture toughness variation across the YPSZ – porcelain interface. SEM imaging of the different types of pillar failure are shown.

Examination of Figure 5 reveals that both YPSZ and porcelain exhibit a 'bulk' fracture toughness response at large distances from the interface. At the interface, an increased scatter is observed which is likely to be associated with the porcelain nanovoiding present at this location [4]. Close to the interface, YPSZ shows a ~15% increase in the fracture toughness. This effect is related to the larger YPSZ grains at this location [4], and the associated increase in K_C [7].

Within 50 µm of the interface, porcelain shows a clear and consistent reduction in toughness of up to 90% at the location where chipping is known to originate. This result is the first to reveal this significant reduction in toughness and identifies the likely origin of persistent failure. This new finding can be used for directed optimisation of the manufacturing routine aimed at increasing the toughness at this location and thereby reducing prosthesis failure rates.

Conclusions

This study is the first demonstration of microscale spatially resolved fracture toughness assessment using the micropillar splitting technique. The experimental technique can be readily implemented in relatively short time, and overcomes many of the limitations associated with fracture toughness techniques of comparable resolution. The new method is a powerful tool for microscale spatially resolved mechanical property analysis and paves the way for a broad range of applications.

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