

# **Micro-shot Peening of Zirconia Advanced Ceramic: An Examination of Surface Integrity**

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## Abstract

This paper presents the micro-shot peening of Zirconia ( $ZrO_2$ ) advanced ceramics applicable for bio-medical, dental, automotive and aerospace sectors. A  $ZrO_2$  advanced ceramic was micro-shot blasted with selected parameters as a first-step investigation focused on the topography, microstructure, surface hardness, and the surface fracture toughness ( $K_{Ic}$ ) characteristics. A new technique of micro-blasting was conducted using a portable shot blaster. A white-light interferometer (WLI), scanning electron microscopy (SEM), Vickers indentation technique were employed for the analysis. This was followed by determining the  $K_{Ic}$  using an empirical equation. Surface roughness was improved by 34% after micro-shot blasting treatment. No surface cracking was present which generally exists due to the brittle nature of the ceramic. The hardness, however, reduced by 5.6% with a reduction in the Vickers crack length of 9%. This improved the  $K_{Ic}$  by 3% when comparing the micro-shot peened surface to the original, as-received surface. It is difficult to conclude if the  $ZrO_2$  advanced ceramic has undergone plastic deformation and the movement of dislocations increased to strengthen the  $ZrO_2$  ceramic at this stage. However, based on the results, it can be predicted that a level of surface compression was induced beneath the micro-shot peened layer as indicated from the result of the surface topography and integrity. This would justify the hardness modification and the enhancement in  $K_{Ic}$ .

**Keywords:** Shot; Peening; Blasting;  $ZrO_2$ ; Hardness;  $K_{Ic}$ ; Surfaces; Ceramics

# 1. Introduction

## 1.1 Research Background

Over the last few decades, Zirconia ( $ZrO_2$ ) based ceramics have made in-roads into not just engineering but biomedical, transportation, information technology and power generation industries to state the least. The superior properties offered by ceramics, namely: electronic; mechanical; thermal; optical; magneto-resistive; piezoelectric and bio-compatibility allow the continuous implementation of novel applications in many different industrial sectors with the aid of technological advances and processing techniques to manufacture such materials. Having said that, there is still an increasing demand for tougher, stronger, harder but crack resistant and reliable ceramics in comparison to the industrial ceramics currently in use [1]. Increasing demand for tailored microstructure, excellent properties that could function in harsh, thermal and mechanical conditions mean that the cost of such property modification also rise. Often, it is a case where surface tailoring/engineering or modification to the ceramic material would bring about sufficient alteration to the material so that it adheres to the particular demand for its application. Amongst many ways, one way to alter the surface properties of materials is by shot peening/blasting, commonly applied to strengthen metals in particular [2]. This technique offers cheap and moderately less timely surface treatment.

Shot peening is a conventional process which enhances the performance of engineering components. Metals in particular are peened using conventional shot peening/shot blasting techniques to not only clean the surface but also to enhance their service life, provide wear resistance, avoids corrosion, fatigue and frictional

stresses. The effect of shot peening on the near surface layer of a material results to plastic deformation from the impact of the shot (balls) fired at high velocity. This occurs during the impact as a portion of the kinetic energy supplied by the shot impacts the material surface, creating a **zone of** local plastic deformation. This in turn results in a small increase in the temperature at the point of impact and the residual kinetic energy supplied by the shot allows the material to deflect it from the surface of the material [3 - 7]. The fired shots at high velocity causes plastic deformation, but this occurs by compression during the impact, whereby, the surface layer stretches and tightens which causes the surface beneath to compress – inducing a compressive zone beneath the impacted area. The compressive trapped stresses **consequently** prevent cracks and fractures to be formed when the material is stressed. **In turn, this** directly improves the functional life and performance of the component, and further resulting in lower maintenance costs.

## **1.2 Research Rationale**

This research is focused on micro-shot peening of ceramics. Published literature in this field in any main-stream journals and the current knowledge as it stands lack the understanding of shot-and-ceramic material interaction in general. Moreover, it is not yet clear as to which parameters are actually suitable to bring about a beneficial effect to hard brittle **materials such as a ZrO<sub>2</sub> advanced ceramics**. Thus, we attempt to present the effects of micro-shot peening a ZrO<sub>2</sub> **advanced** ceramic from a surface integrity view-point, in particular: topographical; microstructural and changes in the surface properties, namely: the surface hardness, and the surface fracture toughness (**K<sub>IC</sub>**). A modification in **K<sub>IC</sub>** of

ceramics could greatly improve their performance and allow them to be tailored for various industrial needs. Upon a carefully designed, precise and controlled micro-shot peening surface treatment of advanced ceramics, would enable them to not only enhance their performance, but also prove to be beneficial in dental restorations and for healing fractured ceramic based dental implants. In addition, micro-shot peening technique, could also benefit engineering applications where surface modification are necessary to enhance the performance of advanced ceramics.

### 1.3 Previous Research in Shot Peening of Advanced Ceramics

Shot peening for the use of material hardening has been around since the 1940's [8]. Much work covering various aspects of the process applied to metallic engineering components has been published [9 – 15]. However, very little applied research and literature in relation to shot peening of ceramics based materials can be found. The reason being is that ceramics are hard and brittle, and have a low possibility to plastically deform. In addition, improvement in strength, resistance to fracture and compressive residual stresses that could be induced into the metals are not evident with such hard, brittle and difficult to process materials using mechanical pre-stressing. Owing to this, it is predictable and inevitable that blasting ceramics using shots directed at high velocity would only yield to adverse effects. Notwithstanding this, research conducted by few authors have found some successes. Frey and Pfeiffer [16], and Pfeiffer and Frey [17] showed that  $\text{Si}_3\text{N}_4$  and  $\text{Al}_2\text{O}_3$  ceramics can be strengthened to comprise with high load capacity, residual stress and roughness using precise process control and parameters [17]. Other examination of results showed compressive residual stresses in the  $\text{Si}_3\text{N}_4$  ceramic

with increase in the load capacity by a factor of 9 [18]. Pfeiffer and Frey also reported that the dislocation of the shot peened surface could occur either by a localised microscopic surface deformation, and appearance of dislocations in the surface crystals. Or dislocation multiplication and local surface deformation which would increase the compressive residual surface stresses and create a strengthened surface of a  $\text{Si}_3\text{N}_4$  ceramic [18]. Although, no evidence of such features in relation to internal material aspect were presented. Furthermore, the  $K_{Ic}$  also improved in comparison to the untreated surface [18]. Pfeiffer and Wenzel [19] then demonstrated similar benefits to other materials and components, namely: cemented carbides and hard chromium plating and thin ceramic leaf springs [19 -21].

Tomaszewski *et. al.* [22 - 23], published papers on shot peening of alumina ceramics in which a ceramic peening process (Stressonic®) was introduced. The result showed grain refinement, crystalline structure as well as an increase in the hardness and  $K_{Ic}$  of alumina. Plastic deformation and an increase in residual compressive stress of 2GPa was also reported. However, if the hardness was increased, using the Vickers indentation technique, the  $K_{Ic}$  should have reduced rather than an increase as stated otherwise [22-23]. Similar results were also reported by Tomaszewski in another investigation for both the zirconia and alumina ceramics, however, the shot peening technique was rather conducted by an ultrasonic method [24]. Takahashi shot peened  $\text{Si}_3\text{N}_4/\text{SiC}$  ceramics of high crack healing ability. The results showed improvement in contact strength of the material and an increase in Weibull modulus [25]. Itoh *et. al.* [26] showed that a layer of aluminium can be deposited by shot blasting ceramics with aluminium

powder. In addition, it was also reported that erosion process also occurred but **it was** unclear as to how the soft aluminium powder (peening media) had eroded a hard brittle ceramic material. Shot peening was also adopted for sample preparation prior to joining ceramic-to-metallic bonds. It is otherwise known as metallization and could be used to clean both surfaces which also **created** a highly adherent layer for effective ceramic-to-metallic bonding as demonstrated by Komarov and Romankov [27]. The existing literature in relation to shot peening ceramics has found some success, but from a theoretical view-point, particularly, the required parameter window for successful and beneficial effect to occur; as well as the shot-material interaction and that too for different grades of ceramics is still unclear and unreported. On this more, the results presented in these finding are somewhat shallow and require further justification. As a first-step investigation, we have attempted to address the gaps in knowledge with new findings on **particularly**, the surface effects herein this paper.

## 2. Experimental Procedure

### 2.1 Material Background

A cold isotatically pressed (CIPed) ZrO<sub>2</sub> **advanced** ceramic was used as an experimental material from Shanghai Unite Technology (China) with the dimension 50mm x 10mm x 5mm bar (see Figure 1). The **advanced** ZrO<sub>2</sub> ceramic comprised of 95 % ZrO<sub>2</sub> and 5 % Ytria CIPed at 455 bar pressure from all orientations and sintered at 1200°C for 5 hours. The ceramic was characterized prior to any experimentation from both the external and internal aspects. The average as-received finish was Ra 805 µm, surface hardness was found to be 1252 HV, a plane strain fracture toughness (**K<sub>Ic</sub>**) was **measured to be** 10.94 MPa.m<sup>1/2</sup>.

## 2.2 Almen Strip Intensity

In order to accurately set-up the micro-peening trials, N-type Almen strip was employed to measure the shot intensity of the micro-shot peening surface treatment. The thickness of the Almen strip was 1.25mm. The micro-shot peening process was first conducted on the Almen strip at 6.20 bar for an coverage duration of 5 min. Glass beads shown in Figure 2 and 3 were employed which were **executed** from a 4 mm diameter nozzle at 90° angle. Distortion of the Almen strip using the above parameters was found to be 873 µm arc height. **It is to be noted that since the work in this investigation was a first-step feasibility study, we do not have a required base-line, and so, it is not clear what Almen intensities were required as there is no particular specification to work towards.** However, a graph is presented in Figure 2(b) showing the peening pressures in relation to arc height based on the Almen intensity measurements that were undertaken. **In addition, Figure 2(c) also represents the variation in Almen intensity versus peening pressure. Both graph show somewhat a linear relationship which indicated that with increasing peening pressure, the arc height and ultimately the Almen intensity increased. In order to obtain a saturation curve, further exposure time and increasing pressure should be applied.**

## 2.3 Micro-Shot Peening Process Details

A portable shot blaster (Carsmetic Tools Ltd, Swansea, U.K.) was employed to conduct micro-shot peening surface treatment with fine glass bead media (Sealey Quality Machinery, Suffolk, U.K.) ranging from 140µm to 280µm diameter shown in Figure 3(a) and (b), and in the graph in Figure 4. The hardness of the glass media was 1161 HV. **A general tendency is to use a shot media that is of higher hardness**

to the material being shot peened. In this case, the hardness of the glass media was just under 7 % to that of the ZrO<sub>2</sub> advanced ceramic. It can be argued that this could be a vast difference, however, with a brittle material, it is very difficult to generate or increase the movement of dislocations within the structure of the ceramics *via* a mechanical pre-stressing technique. If a harder material was used as a media there is a likelihood of the ceramic generating micro-cracks on the surface. Therefore, to keep the surface damage low and to eliminate the surface defects, the option for shot media were either a same material or the one with a hardness value slightly lower. Glass beads were therefore suitable as it reduced the risk of surface fractures, and because they were a commercial product available in the market. Other media such as silicon carbide and alumina beads comprised of hardness somewhat higher than the ZrO<sub>2</sub> and so for practical reasons, the option was to conduct our trial using the aforementioned media. Future studies will solely focus on the effects of different media on the ZrO<sub>2</sub> and other ceramics to gain deeper insight into the subject. Since, this was a feasibility study, we attempted to go with the safer option of not damaging the material. This was successful as our microstructural study further presented in section 4.2 did not comprise of any surface flaws or micro cracking as initially expected from conducting the micro-shot blasting surface treatment.

Furthermore, a silicon carbide (SiC) nozzle with a diameter of 4mm was used. Compressed air gas pressure of 6.20 bar (90 Psi) supplied the rapid flow of glass media through the 4 mm diameter nozzle and onto the original, as-received work-piece. Micro-shot peening was conducted for 5 mins on the ZrO<sub>2</sub> advanced ceramic surface at a distance of 50mm from the nozzle to the sample using a 90° angle – as

the shot media was blasted perpendicular to the sample at a speed 4 sec/pass, which give a traverse speed of 12.5mm/sec. This in turn, over 5 min of micro-shot peening treatment resulted to 75 passes in total. **The divergence of the micro-shot media (executed through a 4mm nozzle diameter at a distance of 5mm) was sufficient to cover the whole width (10 mm) of the ZrO<sub>2</sub> advance ceramic. The white light interferometry (WLI) images in Figure 5 also confirmed that the micro peening media was impacting the whole 10 mm width of the sample.**

### **3. Analysis**

#### **3.1 Measurement of the Surface Topography, Integrity and Microstructure**

A white light interferometry was adopted (WLI) by (Zygo, Connecticut, USA) to investigate the surface topography of the micro-shot peened surfaces of the ZrO<sub>2</sub> **advanced** ceramic. A x2.5 lens was employed for the investigation to scan a 7mm **diameter** white-light beam, directed at the surface of the ZrO<sub>2</sub> at a focusing distance of 10mm from the sample surface to the edge of the focusing lens. A scan length of 1mm was measured at a time with a speed **150µm/32 sec**, whereby, the whole micro-shot peened and the original, as-received surfaces were scanned in the x-direction for both the as-received and micro-peened surfaces. This is because the surface morphology would be anisotropic and not uniform in orientations of the ZrO<sub>2</sub>, hence, the regime to undertake the measurements for both the treated and **untreated** samples were identical. An accuracy of **0.1nm** was available for the system employed. The surface analysis was processed using a MetroPro 8.2.0 software and the images were built using an associated package – ‘Vision’. The surface integrity of the ZrO<sub>2</sub> **advanced** ceramics were observed by Joel, NeoScope,

JCM-5000 (Japan) scanning electron microscope (SEM) at magnifications ranging from x100 to x2000.

### 3.2 Hardness Measurement and the Determination of Surface Fracture Toughness ( $K_{Ic}$ )

An automatic Vickers indenter (Wilson: 452.078 SVD, Wolpert Wilson Instruments, Shanghai, China) was employed to carry-out hardness measurements of both the as-received and micro-shot peened surfaces. An indentation load used was 30kg with a dwell time of 5 sec. The diamond foot-prints were then located and the automatic Vickers indentation system measured the size of the diamond foot-prints in both the x-and-y direction and ultimately the hardness values measured in HV. An error of  $\pm 10\%$  is generally expected with Vickers indentation method when measuring hardness of hard brittle materials such as advanced ceramics. The foot-prints of the Vickers diamond indentation were observed under an optical microscopy, Olympus (BX51, Tokyo, Japan) and the resulting crack lengths from the diamond foot-prints were measured using an associated software (Image Pro). To calculate the fracture toughness of the  $ZrO_2$  advanced ceramic, both the recorded hardness and crack length values from the same hardness measurement were applied to a plane-strain fracture toughness ( $K_{Ic}$ ) concept by using equation published Anstis and Chantikul's work [28]. This concept was successfully applied in our previous work on fracture toughness of ceramics [29 - 33]:  $K_{Ic} = 0.016 (E/HV)^{1/2} (P/c^{3/2})$ , where 0.016 is the materials geometrical value,  $E$  is the Young's Modulus;  $HV$  is the Vickers hardness,  $P$  is the load in kg and  $c$  is the average of the crack length of the diamond foot-print measured in the x-and-y direction. The parameters used for the determination of

plane strain  $K_{Ic}$  of both the original as-received and micro-peened surfaces were Young's Modulus of 230 GPa.m<sup>1/2</sup> as specified by the ceramic manufacturer; 30kg indentation load; Vickers hardness values (HV) and the resulting crack lengths (m). Expected error of  $\pm 10\%$  would be normal using this method of determining the  $K_{Ic}$  of hard brittle ceramics but no other method proves to be cheap easy and less time consuming.

## 4. Results and Discussion

### 4.1 Topography

The surface topography of both the as-received and micro-shot peened ZrO<sub>2</sub> are illustrated in Figure 5 (a) and (b) measured in the x-direction. It is obvious that evidence of surface improvement was found as the average roughness for the as-received surface was 805nm, whereas, the average roughness of the micro-shot peened surface was measured to be 534nm. This showed an improvement in the surface finish by up to 34% after conducting the micro-shot peening surface treatment on the ZrO<sub>2</sub> at the set conditions. Table 1 illustrates other parameters of the surface finish as an average of the measured surfaces for both the as-received and the micro-shot peened surfaces of the ZrO<sub>2</sub> advanced ceramics. Other parameters shown in Table 1 for both the original, as-received surface in comparison to the micro-shot peened surface show that Rt value (the height between the deepest valley and the highest peak), for the as-received surface was just over 2% higher than the micro-shot peened surface. This was due to the fine glass bombardment flattening the sharp kinks and troths of the machined striations present on the as-received surface. The value Rq, corresponds to the standard deviation of the height distribution for the measured area and provides the same information as Ra. Although, the values of Rq differ from the Ra for both

the surfaces, the regime of surface improvement is still evident from the micro-shot peened ZrO<sub>2</sub> advanced ceramic.

When comparing the work herein with the work in previous investigations of Pfeiffer and Frey [16 - 18], the results do not seem to correlate with their findings with respect to the surface finish. Their surfaces were much coarser after peening and surface roughening was reported. Nevertheless, the ceramics used in those studies were much harder than the ZrO<sub>2</sub> used herein, as well as the shot diameter and shot type used in their studies were bigger in size and harder. Hence, the discrepancies in the result have occurred. In addition, the surface modification could be considered very subjective as the original, as-received surface would have been somewhat different to the one in this study. Nevertheless, improvement in surface finish is a beneficial effect, as surface smoothing could be employed for ZrO<sub>2</sub> based dental implants, whereby, surface cleaning and improvement/restoration to existing (used) dental materials could be adopted and applications where friction and wear reduction are a necessity.

#### **4.2 Microstructural Evaluation**

In order to evaluate any modified surfaces, it is first important to compare the modifications with that of the original, untreated (as-received) surface. Micrographs in Figure 6(a) and (b) illustrate the as-received surface of the ZrO<sub>2</sub> advanced ceramic at x200 and x1000 magnification. The coarse asperities or striations that were visible from the topographical images of the WLI can also be observed at microstructural level. Such features can be attributed to the machining process, post CIPing and sintering of the ZrO<sub>2</sub>. The original, as-received

microstructure of the material at both high and low resolution showed not only the machined striation marks but also some porosity, surface flaws, and inclusions as indicated by the arrows on the micrographs.

After the micro-shot peening surface treatment was conducted, it was possible to not only feel the difference in the modified surface of the as-received  $ZrO_2$  with finger nails but also visually see the difference. The microstructural images showed the same modification that was visual and by using the WLI. The rough striation marks from the finishing process were no longer present as the surface was tailored smooth and the roughness improved which was present from the microstructural image in Figure 6 at x 200 magnification in (a) and x 1000 in (b). Not only this, but when comparing the original, as-received surface with that of the micro-shot peened surface have a considerable change to the surface structure as the machining marks were no longer apparent in Figure 7 (a) and (b), as well as the pre-existing porosity was also not present. At the same time, it **was** clear that the kinks and troughs that existed on the original surface layer were somewhat compressed to become smoother and balance-out from these micrographic images.

One of the critical factors of shot peening is the percent of coverage over the material that is being treated. From both the WLI images, and the micrographs it can be suggested that after 75 passes of glass media over the  $ZrO_2$  ceramic surface. **Hence, a practical coverage of at least 98% would have occurred as the whole surface area of the  $ZrO_2$  was bombarded by the glass beads at least once from the 75 passes, and so, it was evident from the micrographs that the presence of**

striations from machining were no longer heavily present (see Figure 8 (a) and (b)). In addition, the images from the WLI in Figure 5(b) also represent this effect. The reason for the 98% coverage taking place was also because a shot peened surface of a material is considered to be completely covered when only 2% or less of the as-received surface was left untreated by the impacting shots. A full coverage in turn, is therefore, only 98% rather than 100%. Further study focused on the effects of different peening times and pressures upon the ceramics will gather more understanding about the level of coverage that can be obtained. Thus, our future investigations will involve a complete demonstration on the effects of different parameters upon ceramics such as a  $ZrO_2$ .

A  $ZrO_2$  advanced ceramic exists at least in 3 crystalline structures in most of cases: cubic, tetragonal (quadratic), and monoclinic. To understand the original as-received structure and the structural evolution after micro-peening process requires an independent study which is not within the scope of this investigation, as herein, we attempt to evaluate and examine only the surface issues. Having said that, the change in phase of the crystals before and after shot-peening should be considered for future investigations as it is possible that the advanced  $ZrO_2$  ceramic would have some phase transformation due to the mechanical effects caused from the micro-shot bombardment. Moreover, it is also possible to have a microstructure gradient from shot-peening surface to sub-layer and so, require an additional investigation focused on the aspects through the cross-section of both the micro-peened and as-received surface.

### 4.3 Effects of Micro-shot Peening on the ZrO<sub>2</sub> Surface Properties

#### 4.3.1 Hardness

Figure 9 presents a graph of the hardness values obtained from the surface of the micro-shot peened region of the ZrO<sub>2</sub> advanced ceramic. As one can see, the average hardness of the original as-received surface (after 28 indentations) was 1252 HV. In comparison, the average hardness of the micro-shot peened surfaces was 1182 HV. A reduction of 5.6 % which could be negligible and one could argue that the error between both the as-received and the micro-peened samples is well within the expected  $\pm 10\%$  region. However, when the diamond foot-print in both the x-and-y direction were analysed for the two aforementioned samples, it was found that the foot-print for the as-received surface was 211  $\mu\text{m}$ , whereas, the one for the micro-shot peened surface was 217  $\mu\text{m}$  on average - an increase in size just under 3%. Thus, the 5.6% reduction in the surface hardness is certainly not negligible because the foot-print produced by the modified micro-shot peened layer were slightly larger and rightly so, due to the top surface considered to be softer after being micro-shot peened. The softening effect could have occurred from increase of plastic strain during the micro-shot peening process of the ZrO<sub>2</sub> advanced ceramic. In doing so, the material achieved its maximum hardness but with increase in strain, more-and-more dislocations could have been generated, whilst equivalent dislocations may have annihilated - forcing +ve and -ve dislocations together, but, further work softening could be predicted which inherently produced large areas of softer surface [34]. Figure 10 show the Vickers diamond foot-print of both the as-received surface in (a) and the micro-shot peened surface foot-print in (b). It is important to consider the possibility of

measurement errors that could occur when measuring the crack lengths of ceramics in general. Not only the brittle nature of the ceramic but the porous structure also makes it prone to cracking under the influence of a sharp load. This has the tendency to vary the hardness resulting from the indentation test by about 10% and like-wise for the resulting crack length produced from the diamond shape indentation [31 -33]. Thus, a variation in the crack length may not always be of the same length in all four corners of the diamond indentation. What is more, the surface after being micro-shot peened cannot be polished so the error in measuring the crack lengths off the computer screen will still be present and will depend on the accuracy of a human eye. It is therefore suggested that an error between 5  $\mu\text{m}$  to 10  $\mu\text{m}$  may occur on average per a diamond indentation test, due to the aforementioned constraint as mentioned.

#### 4.3.2 Change in Crack Length and Surface $K_{Ic}$

In order to determine the  $K_{Ic}$ , Vicker's diamond indents were induced into the  $\text{ZrO}_2$  and their respective crack lengths were measured as presented in Figure 11. The average crack length for the original, as-received surface was 658 $\mu\text{m}$ . This in comparison to the average crack length of the micro-shot peened surface was 639 $\mu\text{m}$ . This is a reduction of almost 9% after the micro-shot peening surface treatment and can be attributed to the fact that a reduction in hardness of 3% increased the size of the diamond foot-print. The average  $K_{Ic}$  as shown in Figure 12 for the as-received  $\text{ZrO}_2$  advanced ceramic was 10.94  $\text{MPa}\cdot\text{m}^{1/2}$ . In comparison, the average surface  $K_{Ic}$  for the micro-shot peened surface was 11.71  $\text{MPa}\cdot\text{m}^{1/2}$ . This is another indication that micro-shot peening surface treatment created work softening of the  $\text{ZrO}_2$  advanced ceramic, and so, a reduction in hardness had

occurred, followed by a decrease in the resulting crack length on the edges of the diamond foot-prints and directly increased the surface  $K_{Ic}$  by over 6 % compared to the original, as-received surface. Attention should be given to the potential errors arising from the measurement of surface hardness and more importantly the crack lengths. **This inherently**, would both influence the end  $K_{Ic}$  value. Due to **the** nature of the ceramics being hard, brittle and porous, it is therefore possible that the crack lengths produced from the Vickers indentation technique may extend leading to a value which may reduce the end  $K_{Ic}$ . In addition,  $ZrO_2$  **advanced** ceramic would have an anisotropic morphology that will also lead to differences in hardness over its surface area which **consequently**, will also lead to variation in the resulting crack lengths and consequently produce variation in the  $K_{Ic}$  values.

Previous investigation by Vahey [15], Pfeiffer *et. al.* [16, 19], Tomaszewski *at al.* [22 - 24] have reported the increase in hardness,  $K_{Ic}$  and also increase in the induced residual compressive stress within ceramics. However, unless the crack length as result of the Vickers indentation foot-print were reduced, then it is not possible to increase the hardness of a ceramic material and still have an increase **in the  $K_{Ic}$  as** was reported. This is because the hardness is a parameter of  $K_{Ic}$ , and upon an hardness increase of ceramics; the material becomes brittle. At the same time, it is also prone to cracking, indicating that the  $K_{Ic}$  of the ceramic would have reduced as it loses its ductility by gaining hardness. **Hence**, we suggest a more thorough cross-sectional residual stress analysis to be conducted **in future studies**. **Moreover**, it would **also** be beneficial to understand the surface compression as well as a thorough microstructural investigation to reveal any increase in dislocation movement that were reported in previous research. We have focused

mainly on the surface issues in this paper, but an investigation of residual compressive stress state through the surface and sub-surface of the **micro-shot peened** ZrO<sub>2</sub> **advanced** ceramic as a future study is encouraged and **merits** a special attention. From this, further understanding of dislocation movement, the effect of work softening, and the stress state of ceramics along with any phase change could also be understood.

#### **4.4 Shot-ZrO<sub>2</sub> Ceramic Interaction**

The surface integrity analysis of a material would reveal several unknown which in most cases cannot be observed from a naked eye [35]. During shot peening of materials in general, the typical events that occur are several. In particular, the material undergoes considerable surface dimpling, a change in surface hardness and the surface **K<sub>IC</sub>** which could be attributed to a possible induction of compressive residual stress [34], although, it would be ideal if this could be proven by further analysis on residual stress state which is the focus of our next study as well as the possibility of material transfer from the micro-shot to the **surface of the** ZrO<sub>2</sub> **advanced** ceramic.

As proven from the results from the WLI, that the micro-particles of glass (glass bead shot media) have impacted the sharp machined striations present over the original ZrO<sub>2</sub> advanced ceramic and compressed the kinks into a flatter, smoother surface. But to broaden the understanding of the shot/material interaction (micro-glass bead interaction with ZrO<sub>2</sub>), it is important to study the state of shots after the bombardment. Figure 13 presents this by the optical images in which a powder glass bead and its diameter is shown at microscopic level. On average the

powder was compacted to a diameter of 11.38 $\mu\text{m}$  and ranged between 5 $\mu\text{m}$  to 24 $\mu\text{m}$ . This was because the glass bead shot media was a soda-lime glass material with a Vickers hardness of 1161HV. On the other hand, the ZrO<sub>2</sub> advanced ceramic surface hardness was just under 8% harder. This meant that the high shot deformation would have occurred whilst the ZrO<sub>2</sub> surface was bombarded, thus, compressed at microscopic level to smoothen and flatten the striations as shown in Figure 14. At the same time, the glass beads were shattered into smaller pieces due to its hardness being somewhat lower. It is generally obvious that the correct shot type for peening would be to use a slightly harder material than that of the material being treated. However, it is clear from this investigation that a like-by-like hardness shot would prove to be considerably effective. We suggest further studies in relation to the influence of shot type on ceramics also.

## 5. Conclusions

This paper presents an in-depth investigation on the micro-shot peening of ZrO<sub>2</sub> advanced ceramics as a first step study. Surface integrity and microstructure were first investigated prior-to and after the micro-shot peening surface treatment and measured were the surface hardness and surface fracture toughness ( $K_{Ic}$ ) characteristics. The following results were found from **this** investigation:

- The results showed enhancement in the surface roughness by 34% after conducting the micro-shot blasting surface treatment.
- SEM analysis revealed that no surface cracking was present which generally would exist due to the brittle nature of ceramics.
- The hardness was reduced by 5.6% compared to the as-received surface and the resulting crack lengths (from the Vickers indentation tests) were reduced by 9% which directly enhanced the  $K_{Ic}$  by 6% under the set-condition applied.

It is unclear to conclude if the ZrO<sub>2</sub> **advanced** ceramic has undergone plastic deformation so that the movement of dislocation would have increased and in turn led to a strengthened top surface layer. Nevertheless, on account of the findings, it can be predicted that a level of surface compression should have occurred beneath the micro-shot peened surface layer by a possible phase change, which justifies the enhancement in surface  $K_{Ic}$  and the improvement in the surface hardness of the ZrO<sub>2</sub> **advanced** ceramic under the set conditions applied.

Implementation of the micro-shot peening with further process refinement and a thorough parametric analysis of various range of ceramics could allow further

understanding of the wider effects of such process/material interactions. With careful process control and selected parameter window would create a cheap, easy and a rapid surface treatment process that would be applicable and highly beneficial to surface treat components from many sectors ranging from automotive; aerospace; bio-medical and dental applications where **ceramics can be** employed.

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Authors declare that the submitted work is their own and that copyright has not been breached in seeking this publication to author's knowledge.

### **Originality**

Authors declare that the submitted work has not previously been published in full, and is not being considered for publication, elsewhere.

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