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Determination of Fracture Toughness of Zirconia Engineering Ceramics and its Effects from processing with fibre laser radiation

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Abstract

Vickers hardness indentation tests were employed to investigate the near surface changes in the hardness of the fibre laser treated and as received ZrO_2 engineering ceramic. Indents were created using 5 kg, 20 kg and 30 kg loads to obtain the hardness. Optical microscopy, white-light interferometery and co-ordinate measuring machine were then used to observe the, crack lengths and crack geometry within the engineering ceramic. Palmqvist and half-penny median crack profiles were found which indicated the selection of the group of equations used herein. Computational and analytical approach was then adapted to determine the ceramics K_{1c}. It was found that the best applicable equation for ZrO_2 was: $*K_{1c} = 0.016\ [E/H]^{½}\ x\ [P/C^{3/2}]$ and confirmed to be 42 % accurate in producing K_{1c} values within the range of 8-12 MPa $m^{1/2}$ for ZrO_2 ceramics. Fibre laser surface treatment reduced the surface hardness and produced smaller surface cracks lengths in comparison with the as received surface. The surface crack lengths, hardness and the indentation loads were found to be important particularly the crack length which significantly influenced the end K_{1c} value when using the equation*. Longer crack lengths proved lower ceramics resistance to indentation and moreover the end K_{1c} value. The hardness influenced the K_{1c} as softer surface was produced by the laser treatment which resulted in higher resistance to propagate a crack and enhanced the ceramics K_{1c}. Increasing the indentation load also varied the end K_{1c} value as higher indentation loads resulted in a bigger diamond footprint and the exhibited longer crack lengths.

Keywords: Fracture Toughness (K_{1c}), Vickers indentation technique, ZrO_2 Engineering Ceramics.

Introduction

Applications of ceramics have been limited due to their crack sensitivity and low fracture toughness (K_{1c}). Nevertheless, the use of ceramics has increased over the years. They are now considered as the new age materials used to manufacture components for the aerospace, automotive, military and power generation sectors. Engineering ceramics offer exceptional mechanical properties, which allows them to replace the more conventional materials currently used for high demanding applications [1-8].

Fracture Toughness (K_{1c}) is a very important property of any material and especially ceramics in particular due to their brittle nature. Ceramics in comparison with metal and metal alloys have a low K_{1c}, hence, it would be an advantage if the K_{1c} of ceramics could be improved. This could open new avenues for ceramics to be applicable to high demanding applications where metals and metal alloys fail due to their low thermal resistance, co- efficient of friction, wear rate and hardness in comparison with ceramics. K_{1c} is a measure of the materials resistance to fracture or crack propagation, it is the plane strain fracture toughness. Materials with high K_{1c} are much softer and ductile. Those types of material can resist cracks at higher stress levels and loading. Materials with low K_{1c} are much harder, brittle and allow crack propagation at lower stresses and loading. Unlike metals, it is difficult for dislocations to propagate with ceramics which makes them brittle. Ceramics also do not mechanically yield as well as metals in comparison which leads to much lower resistance to fracture. The measure of K_{1c} was carried out using the Vickers indentation method which calibrates the hardness of the material and induces a crack. Measured hardness and the crack lengths were then placed into empirical equations to calculate the materials K_{1c} after and prior to the surface treatment. K_{1c} of engineering materials can be determined using various different techniques. Single edge notched beam (SENB), shevron notched beam (CVNB) and double cantilever beam (DCB), as well as Vickers indentation method are all conventionally employed for industrial applications. The Vickers indentation test can be used to determine the K_{1c} of ceramics and glasses from empirical relationships derived by Antis *et al* [9-11]. The advantage of the Vickers hardness test is the cost effectiveness, ease of set up and is one of the most simple and least time consuming in comparison with the other techniques available to determine the K_{1c} of ceramics. The Vickers indentation test method is less responsive in comparison with other techniques, minimum preparation is requires with quick and cost effective set up and use. There are disadvantages to the Vickers indentation test such as the lack of accuracy to measure the length of the cracks which influences the final K_{1C} value [9, 10], the diversity of the use of the indentation equations and its accuracy. The crack lengths are also visualized after conducting the test by means of optical microscopy. Change in the K_{1c} has an influence on the materials functionality or diversity to its applications. Improving the K_{1c} of materials can enhance its functional capabilities such as longer functional life, improved performance under higher cyclic and mechanical loading particularly for demanding applications where engineering ceramics are applicable. This research illustrates a method to determine the K_{1c} using Vickers indentation method for laser treated CIP ZrO_2 ceramics. The test samples were investigated for their near surface hardness, generated crack profiles and surface finish from the diamond indentations prior to and after the laser treatment.

Background to determining the K_{1c} of ceramics

K_{1c} is considered to be one of the most important mechanical properties, particularly for engineering ceramics [12, 13]. Materials with high K_{1c} are much softer and ductile. Those types of materials can resist cracks at higher stress levels and loading [10, 14-16]. Materials with low K_{1c} are much harder, brittle and allow crack propagation at lower stresses and loading such as most ceramics. Unlike metals, it is difficult for dislocations to propagate with ceramics which makes them brittle [17-19]. Ceramics also do not mechanically yield as well as metals in comparison which leads to a much lower resistance to fracture in comparison.

A common method of measuring the materials K_{1c} is by using the Vickers indentation technique which measures the hardness of the material by inducing an indentation aided by a diamond indenter to produce a crack in the material surface [9, 10, 20]. Measured hardness and the crack lengths are then placed into an empirical equation to calculate the materials K_{1c} [9, 10, 14-16, 21, 22]. The results from the Vickers indentation test can then be applicable to the empirical equations which are derived by Ponton [9, 10], Chicot [21], Liang *et al* [23]. The equations derived by Ponton *et al* [9, 10] originate from various other authors [25-35]. However, they are modified by Ponton *et al* and applied specifically to hard and brittle materials such as ceramics and glass [9, 10]. The equations have a geometrical relationship with various ceramics. Different ceramics have various equations applicable to calculate the ceramics K_{1c}. Preparations of the samples involve polishing in order to create a reflective surface plane (this would mean that the surface has been well polished) [9, 10, 22, 36,] prior to the Vickers indentation process. There are still constraints with the Vickers indentation techniques as reported by Gong *et al* [13], over the more conventional technique applied such as single edge notched beam (SENB) and double –torsion (DT) method as mentioned elsewhere [11, 37- 39]. The constraints are: (a) the dependence of the crack geometry on the applied indentation load and the properties of the material; (b) indentation deformation (non-uniform fracture progression or rapid fracture growth) such as lateral cracking; and (c) unsuitable consideration of the effect of Young's modulus and the material hardness [13].

The procedure and steps which one must comply to in order to produces a genuine Vickers indentation test result and produce K_{1c} values that are genuinely valid [10]: Those namely are: (a) each indentation must be performed at a sufficient distance from one another. This would avoid the formed cracks to inter-connect and bridge with the other diamond indentations performed on the ceramic surface [40, 41], (b) a minimum load of 50 N must be used and recommended as the ceramic materials comprise of sufficient hardness requiring enough loading to produce an indent, (c) it is ideal to coat the test surface with gold so the performed indentations are visible. Post indentation coating may affect the crack tip and give an inaccurate reading, (d) the test samples should be near to 20c in thickness and have minimum porosity. The author also states that the adjacent indents should be no closer than 4c. The term “c” in the literature is defined as the crack length. (It is unknown what the term “c” is unless a Vickers indentation test is conducted. The indentation test is hence, required to be performed in order to determine if the process is valid.

Liang *et al* [23] followed an investigation on the K_{1c} of ceramics using the indentation method. He also used several equations by various authors as listed in [23]. It was stated by Liam *et al* that equations differ as the crack geometry changes (from Palmqvist to median half-penny cracks). He introduced a new equation stated elsewhere [23], which was said to be more universal as opposed to the pervious work conducted. However, in order to use the formula, it was required that it had to be manipulated for sufficient use. Ponton *et al*’s formula in comparison is much simplified and is easy to apply. Chicot *et al* [20] conducted further investigation by applying two other equations to produce results using materials such as tungsten carbide (Nickel phosphorus treated) and pure silicon. He uses the concept of median half-penny crack and a Palmqvist crack system to determine the most applicable equation [20]. It is stated that high indenter loads produce a median half-penny crack within the material which is on the edges of the diamond indentation (foot print produced). This type of crack will always remain connected. A Palmqvist crack is produced during low indenter loading and is of a smaller scale in comparison. The Palmqvist crack will always appear at initial stage of the crack generation during the indentation process, then, a median half-penny crack is produced once the impact of the indenter is exerted. It can be assumed that a median half-penny crack may be the result since the ceramics comprise of high hardness, indicating that high indenter loads are required in order to induced visible and measurable diamond foot prints.

Orange *et al* [37] investigated the K_{1c} of Al_2O_3-ZrO_2 by comparing the notched beam and the Vickers indentation techniques. Cracking behaviour was observed as Palmqvist and median half-penny crack geometries were found. Low indentation loading produced Palmqvist cracks and with increasing loading; median half-penny cracks were found. High micro-cracking was also found with Vickers indentation technique when a fine grain size (0 – 3 μm) ceramics were tested and with increasing grain size (0- 5 μm), the micro cracking was reduced. With the notched beams technique; a higher K_{1c} value was achieved and with increasing grain size [37]. This meant that the ceramics with smaller grain boundaries comprise of higher K_{1c} value and consists of higher resistance to fracture. From Oragne *et al*'s investigation it can be gathered that notched beam indentation technique produced better results in comparison with the Vickers indentation method, although, the reasons behind this were not justified in his work [37].

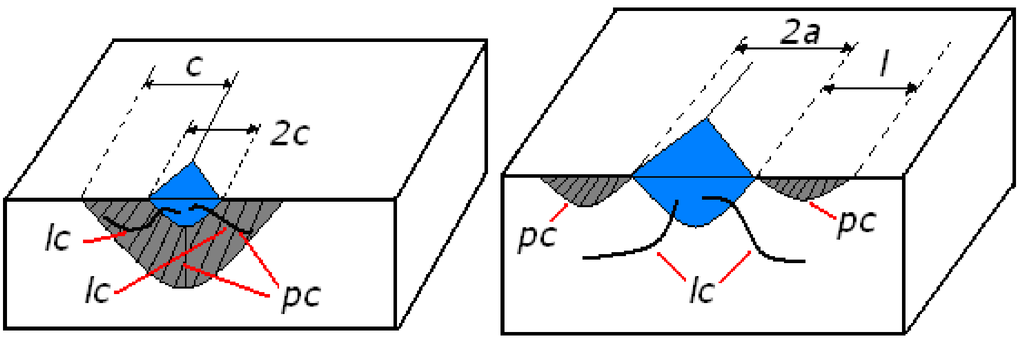
Equations for median half penny-shaped cracks as presented in Table 1 were used for high indenter load applications. One equation was selected to calculate the K_{1c} value for the treated and as received samples from applying the equations to real experimental values. The equations in the tables have been derived by the materials geometrical value that has been obtained from experimental means, of ceramics and glass [9-10]. The suitability of applying various equations to ZrO_2 is not particularly defined, so it was required that an investigation was carried out in order to determine the best employable equation for this study. There are 10 equations selected for this study from various equations discussed in [9-11], to first determine the K_{1c} of the as received surface of ZrO_2 and then the laser treated surfaces. The selected equations applicable to calculate the $K_{1c,}$ by using the Vickers indentation methods are [9]:

Table 1: Equations used to investigate the most employable equation significant for calculating the K_{1c} of the treated and as received ZrO_2 ceramics.

Equation Number	**Equations**	**Equation Number**
1	$K_{1c} = 0.0101\ P/\ (ac^{1/2})$	Lawn & Swain
2	$K_{1c} = 0.0824\ P/c^{3/2}$	Lawn & Fuller
3	$K_{1c} = 0.0515\ P/C^{3/2}$	Evans & Charles
4	$K_{1c} = 0.0134\ (E/Hv)^{1/2}\ (P/c^{3/2})$	Lawn, Evans & Marshall
5	$K_{1c} =0.0330\ (E/Hv)^{2/5}\ (P/c^{3/2})$	Niihara, Morena and Hasselman
6	$K_{1c} =0.0363\ (E/Hv)^{2/5}(P/a^{1.5})\ (a/c)^{1.56}$	Lankford
7	$K_{1c} =0.095\ (E/Hv)^{2/3}\ (P/c^{3/2})$	Laugier
8	$K_{1c} = 0.022\ (E/Hv)^{2/3}\ (P/c^{3/2})$	Laugier

9	K_{1c} =0.035 $(E/Hv)^{1/4}$ $(P/c^{3/2})$	Tanaka
10	K_{1c} = 0.016 $(E/Hv)^{1/2}$ $(P/c^{3/2})$	Anstis, Chantikul, Lawn & Marshall

Median half-penny shaped cracks occur when high indentation loads are applied [21, 22, 42]. The profile of a median half-penny shaped cracks are illustrated in Figure 1 (a). It can be predicted that the outcome for most of the crack profiles in this study would be of median half-penny shape. For cracks that are of median half-penny shape; the applicable equations differ and presented in this paper (equations 1-15) [15, 16]. The indention load at which the median half-penny crack occurs for most ceramics is 3 N [21]. This was lower for the loads applied for this study, hence, it could be assumed that the generated cracks would always be of half-penny median crack profile, so this indicates that only equation particularly applicable for median-halfpenny cracks should be utilised for this study in order to determine the K_{1c}. Figure 1 (b) illustrates a profile of a Palmqvist crack which tends to occur at low indentation loads [21, 42]. A Palmqvist crack is part of the median-half penny crack because when a load above 3 N is applied the indenter "pop in" occurs; a Palmqvist crack is already produced and further developed into a median half-penny crack [21, 42]. These cracks are shallow and lie in the axis of the indenter as there would be a small extension at the edge of the diamond indenter [42]. Up to 50 N of indentation loads were used for this work, hence, it is likely that a Palmqvist crack will occur leading to a half-penny median crack geometry.



(a) **(b)**

Figure 1: Schematic of the median Half–penny crack (a) and (b) Palmqvist crack system.

Where l is the surface crack length, 2c or 2a is the length of the diamond indent, c is the centre of the diamond to the end of the crack tip and pc is the load impact, lc is an interior crack.

Ponton *et al* [10] state that equation suggested by Chantikul *et al* [31] suggested that equation 10 has an accuracy of 30 to 40 % for ceramics that which are well behaved in their indentation response. However, it is first required that the propagation of the crack geometry is understood from performing the Vickers indentation test on the as received ZrO_2 ceramics as further justified in this paper. It is not made clear as to why this equation was particularly used for the ceramic. It was therefore, required that some of the relevant equations were applied to the tested values from this experiment to determine what sort of results are obtained. Hardness test was performed on the ceramics assuming that the resulting cracks were of half-penny median type (due to applying a sufficient indentation load applied). Ten equations were employed as previously stated to establish which particular equation type produces the K_{1c} value that is nearest to the known value for the as received ZrO_2 ceramics which is normally between 8 –12 MPa $m^{1/2}$.

Experimental Methodology

Background of Test materials

The material used for the experimentation was cold isostatic pressed (CIP) ZrO_2 with 95 % ZrO_2 and 5 % yttria (Tensky International Company, Ltd). Each test piece was 10 x 10 x 50 mm^3 bars and comprised the surface roughness of 1.58 µm as provided by the manufacturer (Tensky International Company, Ltd.). This was to reduce the laser beam reflection as shinier surfaces would reduce beam absorption, although, rougher surfaces of the ceramics can often be more prone to cracking in comparison [9, 21]. The experiments were conducted in ambient temperature (20˚C). All surfaces of the ceramic to be treated were marked black prior to the laser treatment to enhance the absorption of the laser beam.

Hardness Indentation test and background of the Vickers indentation technique

An indenter of a specific shape made from a diamond material was used to indent the surface of the ceramics under investigation [8-21]. The diamond was initially pressed on to the as received surface of ZrO_2 ceramics and the load was then released. A diamond indentation was hence created onto the surface which was then measured in size. Thereafter, the surface area of the indentation was placed in to Equation 16 to calculate the hardness value:

$$HV = 2P \sin[\theta/2] / D^2 = 1.8544P / D^2 \qquad (16)$$

where P is the load applied in kilograms (Kg), D is the average diagonal size of the indentation in mm and θ is the angle between the opposite faces of the diamond indenter being 136° with less than $\pm 1^{\circ}$ of tolerance. Indentation load of 5 Kg, 20 Kg and 30 Kg, were applied. The indented surface and the resulting crack lengths were measured using the optical microscopy. This method was then implemented for the laser treated surfaces of the ceramics tested. The test samples were placed under the macro indenter and were initially viewed using the built in microscope to adjust the distance between the surface of the work-piece and the diamond indenter. This maintained a sufficient distance during each indentation and allowed a standardised testing method which complies to [11].

Calculating the crack lengths

Crack lengths generated by the Vickers diamond indentation test as presented in Figure 2 were measured using a Contact-less, Flash 200, CMM (Co-ordinate Measuring Machine). The ceramic samples are placed under the traversing lens (Optical Microscopy). The lens traverses in the Y direction and to adjust the magnification it is also able to move in the Z direction. Motion in the Y direction is provided by the bed on which the test-piece is mounted for analysing the surface. The image appears on the screen as the optical lens traverses above the surface of the test-piece. The diamond indentations and the resulting crack lengths were measured by moving the lever in the X and the Y direction and selecting a starting point on the screen where the crack ends (crack tip) and stopping on the symmetrical side of the other crack tip, which produced a measurement in both the X and the Y direction.

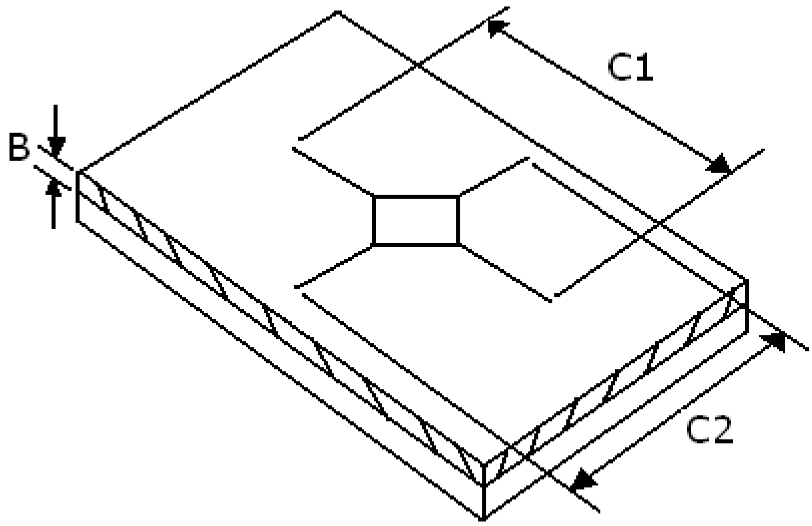


Figure 2: Schematic of a Vickers diamond indentation with propagation of the cracks.

Calculation of the Fracture Toughness (K_{1c})

Initial investigation used 15 equations to determine which equation type was best suited for calculating the K_{1c} [9, 10]. As received surface of ZrO_2 were first tested for its hardness. Fifty indentations were produced on one side of the particular surface of the ZrO_2 ceramics from various test samples. Calibrated hardness was then recorded and a mean average was measured of the as received surfaces. Each indentation was then viewed at microscopic level by the aid of the optical microscope to observe the surface morphology. The crack lengths were measured using the Flash 200 CMM and crack geometry was observed by a 3-D surface topography using the white-light interfrometry (Alicona Ltd., Infinite focus, IFM 2.15). The crack lengths, produced by the indentations were then placed into the various K_{1c} equations with its measured average hardness. In order to confirm that the cracks generated by the diamond indentation at 5 Kg were of median half-penny crack profile. This insured that the equations (1-15) used for median half-penny crack profile were the correct. Figure 3 and 4 presents an example of a typical surface profile produced of the Vickers diamond indentation using a 5 Kg (see Figure 3) and 20 Kg (see Figure 4) loads. Both showed evidence of median half-penny type crack profile where an indenter "pop in" indicated in Figure 3 and 4 is exerted and then a linear crack is produced. A Palmqvist crack profile which tends to occur with lower indentation loads has occurred (as indicated from the indenter "pop in") already in this crack geometry. The concept is more present with higher indentation loading as presented in Figure 4.

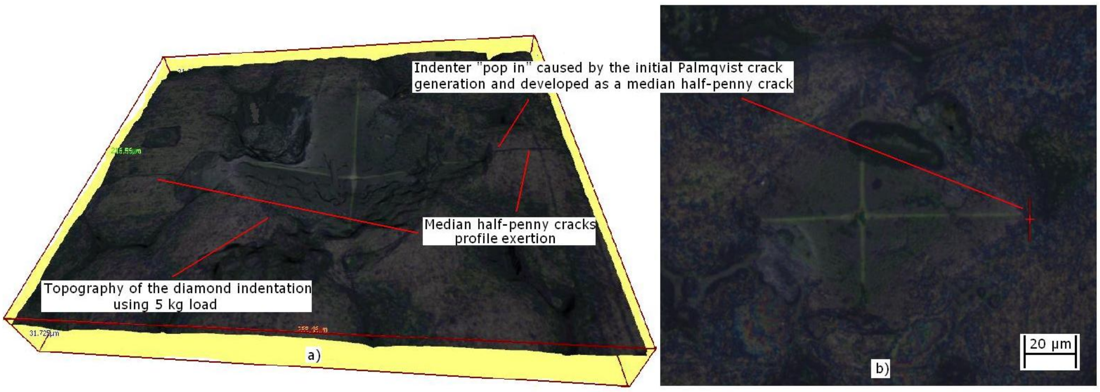


Figure 3: Topography of the Vickers diamond indentation of the as received surface of ZrO_2 ceramics indented at 5 kg, illustrating a median half-penny crack geometry.

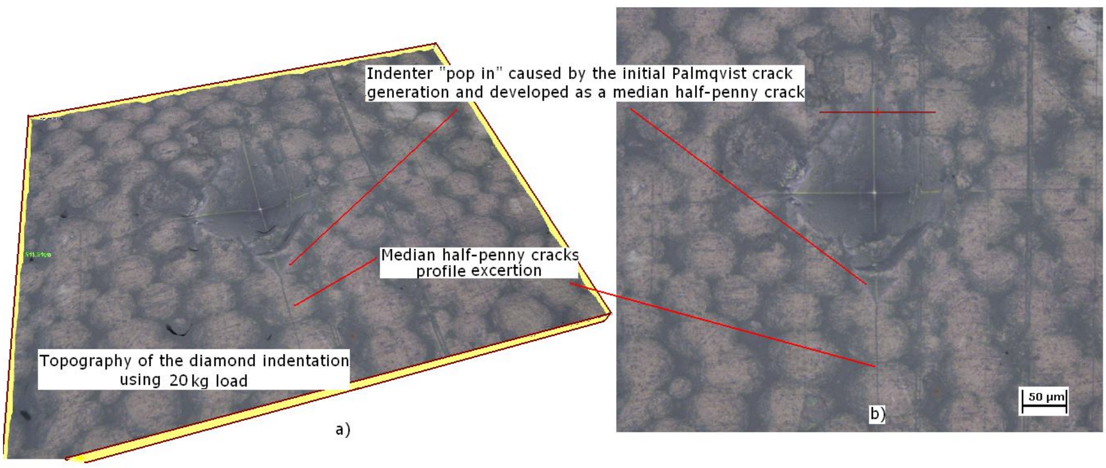


Figure 4: Topography of the Vickers diamond indentation of as received surface of ZrO_2 ceramics indented at 20 kg, illustrating a median half-penny crack geometry.

The equations used for this study are for half-penny median cracks. It was found that the cracks produced from the Vickers indentation test were half-penny median cracks so other equations illustrated for Palmqvist cracks were not used. Equations 1 to 10 were used to calculate the K_{1c} value for the as received surface of the tested ZrO_2 ceramics. The results have been tabulated and are as presented in Table 3 and 4. The equations were set up using Microsoft Excel which made it easy to be able to input three major parameters from the full equation. These values were hardness, crack length and the Vickers indention load. It can be seen that all the values which range between 8 to 12 MPa $m^{1/2}$ for ZrO_2 ceramics, allow the equation to be accurate and useable for calculating the K_{1c} for the laser treated and as received surfaces of the ceramics.

Table 2: Presents the end value of K_{1c} after applying the obtained hardness and the resulting crack lengths from the Vickers diamond indentation test conducted on as received ZrO_2 Ceramics.

Equation No.	**Equation Origin**	**Equation**	**Average K_{1c} (MPa $m^{1/2}$)**	**% accuracy (K_{1c} value within Range)**	**Status**
1	Lawn & Swain	$K_{1c} = 0.0101\ P/(ac^{1/2})$	0.90	0	Unacceptable
2	Lawn & Fuller	$K_{1c} = 0.0515\ P/C^{3/2}$	3.25	0	Under
3	Evans & Charles	$K_{1c} = 0.0824\ P/c^{3/2}$	5.20	0	Under
4	Lawn, Evans & Marshall	$K_{1c} = 0.0134\ (E/Hv)^{1/2}(P/c^{3/2})$	28.70	0	Unacceptable
5	Niihara, Morena and Hasselman	$K_{1c} = 0.0330\ (E/Hv)^{2/5}(P/c^{3/2})$	683.64	0	Unacceptable
6	Lankford	$K_{1c} = 0.0363\ (E/Hv)^{2/5}(P/a^{1.5})(a/c)^{1.56}$	783.93	0	Unacceptable
7	Laugier	$K_{1c} = 0.095\ (E/Hv)^{2/3}(P/c^{3/2})$	2024.98	0	Unacceptable
8	Laugier	$K_{1c} = 0.022\ (E/Hv)^{2/3}(P/c^{3/2})$	759.60	0	Unacceptable
9	Tanaka	$K_{1c} = 0.035\ (E/Hv)^{1/4}(P/c^{3/2})$	1208.44	0	Unacceptable
10	Anstis, Chantikul,	$K_{1c} = 0.016\ (E/Hv)^{1/2}$	12.66	42	Acceptable

	Lawn & Marshall	$(P/c^{3/2})$			

For all tested samples the indentation load is 5 Kg and 30 Kg (Vickers indentation test), E (Young's Modulus) is 210 GPa $m^{1/2}$ for ZrO_2. Range (required equation accuracy) is 8 to 12 MPa $m^{1/2}$ +/- 0.40 MPa $m^{1/2}$ for ZrO_2 ceramics. Average of the K_{1c} was obtained by using values from 50 different Vickers indentation tests. This allowed more consistency in calculating the K_{1c}, as values were used from a bigger pool of data (results).

Different values for K_{1c} were obtained. The K_{1c} value of untreated ZrO_2 was 8 to 12 MPa $m^{1/2}$, so the values that do not lie between the rang given for both ceramics were not considered as acceptable and therefore, those equations were discarded. The K_{1c} value using equation 10, were reasonable for both of the material and comply within the desired range so the equation was accurate and useable. Other equations were discarded and were not taken into consideration for use. Each of the equation was set up by the aid of an Excel spreadsheet. The experimental values obtained were an input into the equation such as the indentation load, crack length created by the Vickers diamond indentations and the measured hardness. The equation that generated the most accurate result was equation 10. Up to 42 % accuracy was found with using the same equation with the as received surface of ZrO_2 ceramics. Other equations applied were discarded as they proved to be of minimal use due to their results for this investigation. Values obtained using equations 10 were most accurate in comparison with the other equations. Hence, this equation was used for all as received ZrO_2 ceramics and laser treated samples to determine the K_{1c}. Where P = load (kg), N = load in Newton's (N), c = average flaw size, a = 2c, m = length in meters, Hv = Vickers material hardness value, E = Young's modulus. (Young's modulus for all untreated samples of ZrO_2 was kept to 210 GPa $m^{1/2}$.

The ceramic surfaces were first treated with the CO_2 laser and a fibre laser. The K_{1c} values were then calculated using equation 10. The reason for changing the Young's modulus from 210 GPa $m^{1/2}$ to 260 GPa $m^{1/2}$ for ZrO_2 ceramics was due to the ceramics being isotropic (meaning the Young's modulus of the material not being uniform around all orientations of the material). This may occurs due to certain manufacturing impurities and further modifications to have occurred during processing of the ceramics. As the ceramic is exposed to the laser beam (thermal energy); which leads to induce further changes within the material from the induced thermal stress which indicate that the Young's modulus value for all laser treated samples should ideally change for calculating the ceramics K_{1c}.

Fibre Laser Treatment

A 200 W fibre laser (SPI, Ltd.) was employed using continuous wave (CW) mode. The laser wavelength was 1.07μm. Trials ranged from 75 to 150 W by varying the traverse speed for the initial experiments to find that traverse speed of 100 mm min^{-1} was an ideal constant to maintain for all trials with only changing the laser power. Hence, all speeds were kept to 100 mm min^{-1} for the main set of experiments presented in Figure 1 and Table 1. Trials below 75 W for ZrO_2 ceramics at 100 mm min^{-1} showed no evidence of any influence on both of the ceramic the surfaces. Focal position was kept to 20 mm above the work-piece to obtain a 3 mm spot size for all trials. The processing gases used was compressed air at a flow rate of 25 l min^{-1}. Programming of the laser was conducted using an SPI software which integrated with the laser machine. A 50 mm line was programmed using numerical control (NC) programming as a potential beam path which was transferred by .dxf file. The nozzle indicated in Figure 2 was removed for all experiments.

Table 1: Parameters used for fibre laser treatment of ZrO_2 ceramics.

Trial No	Power (W)	Comments
1	75	No visual effect
2	100	Small change in colour
3	125	Small cracks apparent
4	130	Small cracks on the edges.
5	150	Large crack apparent
6	137.5	Crack –free
7	143.25	Crack-free
8	150	Apparent cracks

Results and Discussion

Analysis of the as received surfaces

The average surface finish of the as received surface was found to be 1141 Hv for ZrO_2 as illustrated in Figure 10. The values provided by the supplier (manufacturer) for the as received surfaces are 800-1200 Hv for ZrO_2. The ceramics were manufactured using the CIP method which may have left porosity and surface flaws into the ceramic in comparison with the HIP (Hot Isostatic Pressing). The deviation of the hardness values from its mean. The average surface hardness of ZrO_2 is 889 Hv with the highest value of 1129 Hv and lowest being 757 Hv. This was when an indentation load of 30 Kg was applied. This fluctuation has occurred due to several factors such as porous structure, the ceramics response to the diamond indentation, surface flaws and micro-cracks pre-existing on the ceramic, operator and machine accuracy in measuring the sizes and footprints of the diamond indentations.

The fluctuation found in the mean hardness from the results of this study were over 11 % in comparison with the values for ZrO_2 given by the manufacture which is 1 % higher for ZrO_2 from the ± 10 % range given in the literature [16] and can be an except from being a non- conformance.

The results for the crack lengths produced by the Vickers diamond indentations ZrO_2. The average crack length 276 µm for ZrO_2 ceramics. Results from 50 indentations present that the crack lengths range from 221 µm as the lowest and 335 µm being the maximum for ZrO_2 ceramics. The variation from its mean value is wide due to the micro-cracks pre-existing on the materials surface. If the surfaces were well polished the results of the crack lengths would be much lower as the surface would be less prone to cracking after grinding and fine polishing of the ceramic. However, a smother surface would prevent the laser from being absorbed sufficiently into the material surface and often has the tendency to reflect more than absorb, hence, the surfaces were not polished and were tested as received from the manufacturer.

From applying a 30 Kg load, it was found that the cracks were significantly large due to the amount of force exhibiting on the surface area of the ceramic. An example of such crack profile is shown in Figure 5. It was therefore, interesting to investigate the crack lengths produced with a lower indentation load which predictably would have a smaller effect on the end value of the ceramics K_{1c}. Hence, a 5 Kg of indentation load was used due to the force over the surface area being much lower which produces a smaller footprint of the diamond and the resulting crack lengths. This would therefore, result to producing a lower K_{1c} value than the literature and the manufacturers range given for the ZrO_2 ceramics. However, with this particular investigation, hardness, crack length and the K_{1c} value of the near surface layer was only determined as the depth of the diamond indentation from a 5 Kg load was much smaller in comparison with the depth of the diamond produced by s 30 Kg load.

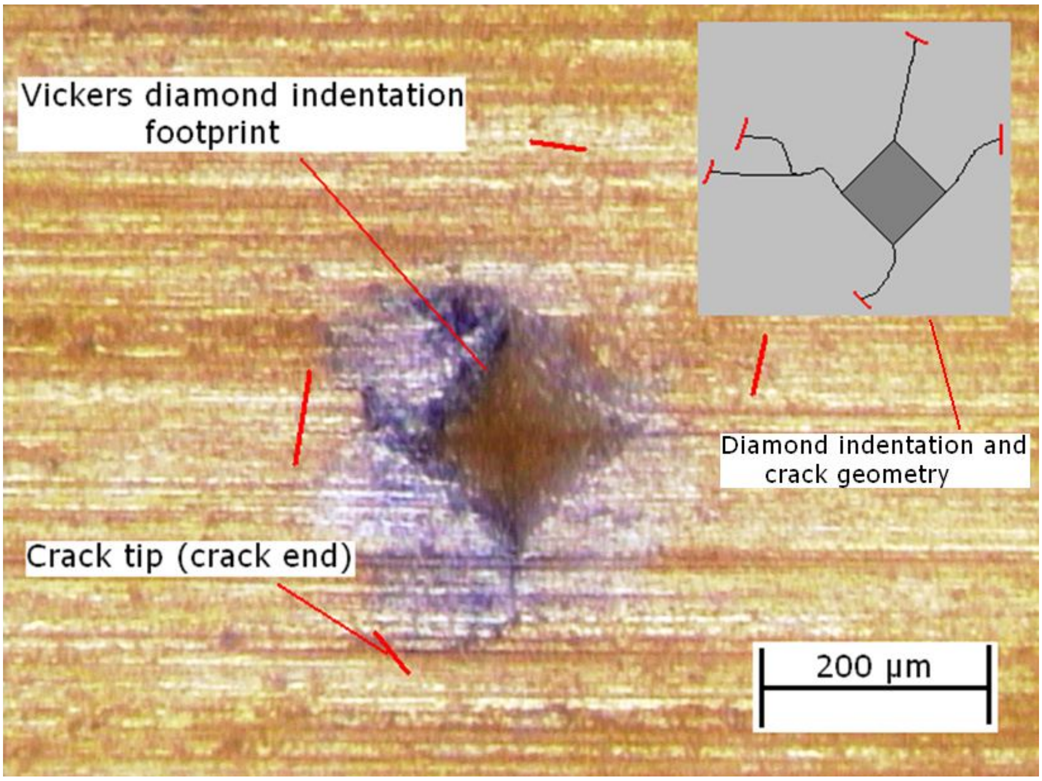


Figure 5: as received surface of ZrO_2 ceramics indented with by a 30 Kg load (hardness =926 (Hv), crack length = 437 µm, K_{1c} = 6.94 MPa $m^{1/2}$).

The K_{1c} for as received surfaces after applying an indentation load of 30 Kg as presented in Figure 6. The values obtained from conducting the indentation test complies with the values given in the literature and the values given by the manufacturer [1, 16]. The average K_{1c} for ZrO_2 was found to be 12.7 MPa $m^{1/2}$. It is indicative from the graph in Figure 8 that there is a significant level of fluctuation for the values above and below the mean range.

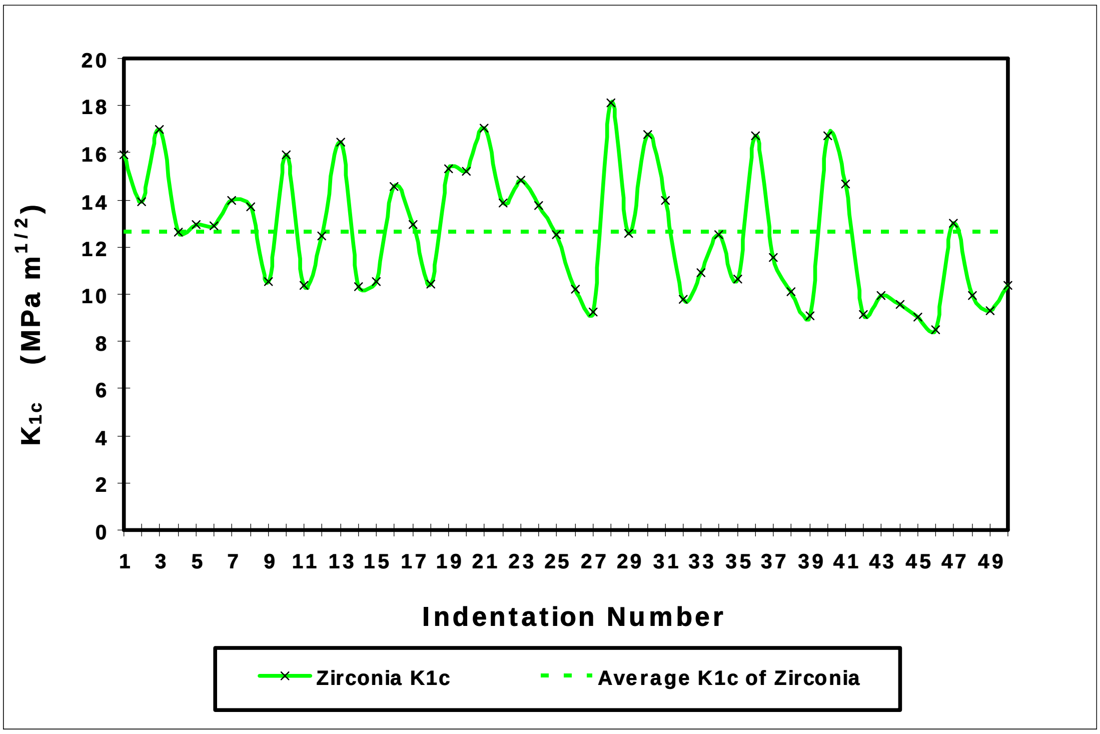


Figure 6: K_{1c} of the as received surfaces ZrO_2 ceramics after applying a load of 30 Kg.

The highest value above the mean was found to be 18.11 MPa $m^{1/2}$ and the lowest value above the mean was 8.52 MPa $m^{1/2}$. This has occurred due to the following factors: (a) a change in the material hardness influences the end K_{1c} value. The change in the hardness by ± 100 Hv resulted into a change in the final K_{1c} value by ± 0.34 MPa $m^{1/2}$, (b) change in the crack length (being the major parameter in the equation as used in this work (equation 10)) by ± 100 µm results into change in the end K_{1c} value over ± 6.31 MPa $m^{1/2}$ if the hardness was up to 1250 Hv as a particular input parameter in the calculation. Hence, the crack length has a bigger influence on the K_{1c} end value in comparison with the hardness, (c) the surface micro-cracks and porosity pre-existing on the ceramic surface making it prone to cracking and reduces the ceramics resistance to fracture, (d) the response of ZrO_2 ceramics to diamond indentation as some of the areas within the ceramic produced fluctuating values to other areas from the view point of the crack length, porosity and the surface flaws.

The hardness for ZrO_2 ceramics from applying a 5 Kg load was much lower than the hardness values obtained after applying a load of 30 Kg. This is because of the 5 Kg load applied to the material's surface area resulted into lower penetration of the diamond indentation into the ceramic as well as the surface area of the diamond footprint also being smaller in dimension resulting in a lower calibration of the hardness value. The average hardness value for ZrO_2 ceramics was 983 Hv with the highest value being 1330 Hv above the mean and lowest being 707 Hv below the mean. The hardness values of ZrO_2 using a 5 Kg load comply with the hardness values provided by the manufacturer, however, they were found to be towards a bottom limit. A possible cause of this vast fluctuation in the values may have occurred due to the material being much softer on its top (near surface) layer in comparison with the bulk hardness and due to the surface being a much porous structure and comprising of cracks which often produced non-uniform results.

The results showed minimal difference in the generated crack lengths for ZrO_2 ceramics from applying a 5 Kg load in comparison with the results from applying 30 Kg load. The average crack length was 279 µm. Despite the indentation load and the applied force being much smaller in comparison with the 30 Kg load; the material was yet cracking equivalently compared to the results of the trials conducted using a higher load. This clearly indicates that the surface did not exhibit a good response during the indentation test. This could mean that a smoother surface finish is required for the indentation test in order to overcome this problem so that the surface scaring and micro-cracks pre-existing on the ceramics are minimized and the strength of the top (near) surface layer is further enhanced. This has a possibility of increasing the surface hardness yet at the same time also reduces the resulting cracks from the Vickers diamond footprints.

Ponton and Rawlings *et al* [9] suggested that a minimum loading of 50 N must be indented in order to produce a diamond indent which some way or another agrees to the work in this study although, the loading herein is 49.05 N and we yet see a diamond indentation in Figure 7 with a median half-penny shape profile. Initial experiments using lower indentation loads such as 24.5 N and 9.8 N also presented a sufficient indentation footprint from the Vickers hardness test. The diamond indentation in Figure 7 is smaller in size when compared with the indentation created by the 30 Kg load. However, the crack lengths found from using a 5 Kg indentation load were equally the same size as that of the 30 Kg. The difference between the two tests results were 3 % and less when considering a larger pool of data. From this it can be gathered that macro hardness the indentation test may be more stable at higher indentation loads particularly with hard brittle materials such as engineering ceramics.

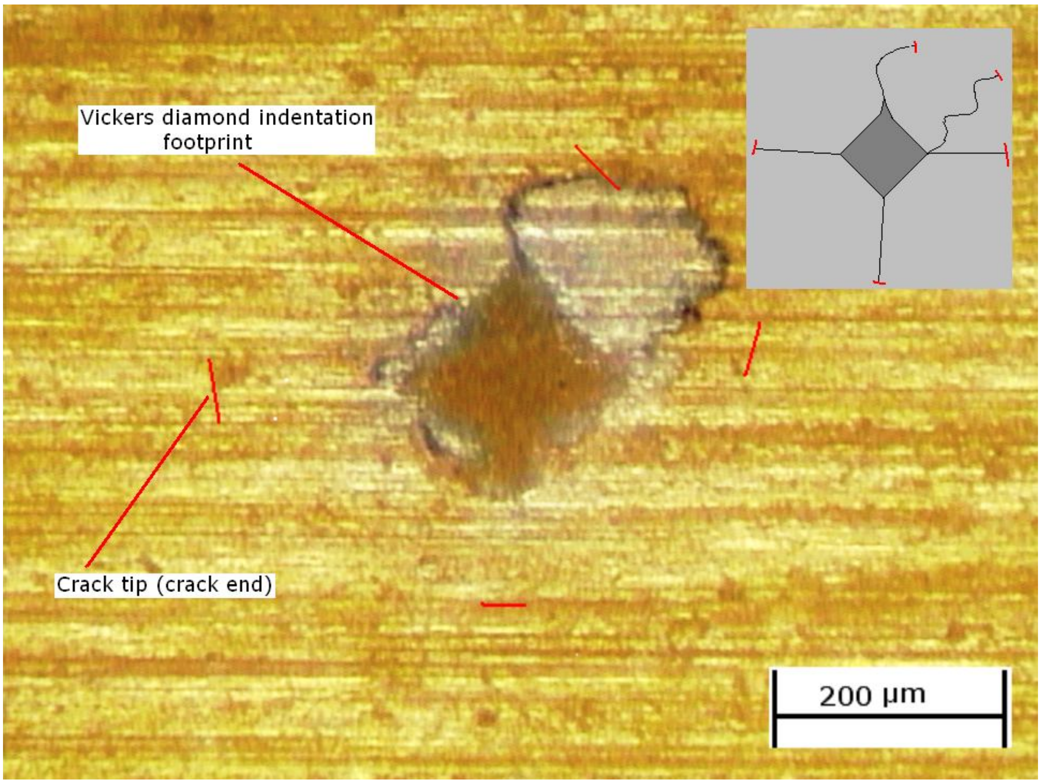


Figure 7: as received surface of ZrO_2 ceramics indented with by a 5 Kg load (hardness =1120 (Hv), crack length = 425 μm, K_{1c} = 1.10 MPa $m^{1/2}$).

This result found for hardness herein when employing a 30 Kg indentation load match with the values provided by the manufacturer and proves that the method used for the hardness calculation and measurement of the crack lengths is valid. Although, the values for the hardness are much smaller than the values provided in the manufacturer specification when using a 5 Kg load. This was due to the fact that the indentation load was much smaller and produced smaller footprints of the diamond which exerted lower force to the surface area and reduced the end value of the K_{1c}. The average K_{1c} was found to be 2.53 MPa $m^{1/2}$ for ZrO_2 ceramics as presented in Figure 8. The highest value K_{1c} value was 2.53 MPa $m^{1/2}$ with the highest value being 6.02 MPa $m^{1/2}$ and the lowest being 0.88 MPa $m^{1/2}$. A possible occurrence of this has led due to the as received surface of the ceramic being scared and comprising of micro-cracks during its processing. The hardness can become much if the surfaces were ground and polished prior to the Vickers indentation test. This would minimizes the surface micro-cracks and results in obtaining a better consistency in achieving the hardness value and the resulting crack lengths. The surfaces were tested as received due to the comparison made with the laser treated surface as the ground and polished surfaces would enhance the materials reflectivity to the laser beam and would minimize the laser beam absorbing into the ceramic.

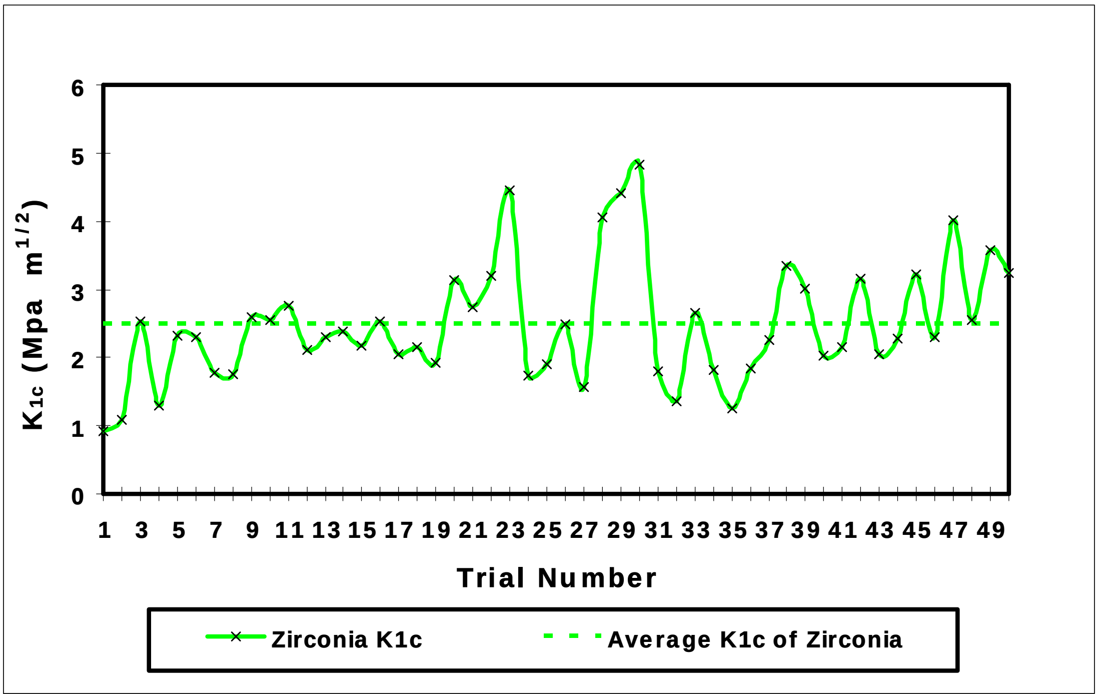


Figure 8: K_{1c} of the as received surfaces of ZrO_2 ceramics from applying a 5 kg indentation load.

Analysis of the fibre laser treated surfaces

The mean hardness found was 941 (Hv) for the ZrO_2 ceramics after conducting the fibre laser treatment. The highest value above the mean was 1089 (Hv) and the lowest being 826 (Hv). The average surface hardness of the as received surface of ZrO_2 was 983 (Hv). There is a 4.5 % difference between the hardness values obtained for the fibre laser treated ceramic in comparison with the hardness values obtained by the as received surface. The fibre laser has decreased the hardness in comparison to that of the as received surface of ZrO_2. The average crack length of the fibre treated ZrO_2 ceramics was 171 µm. The crack length was much reduced in comparison with the crack length of the as received surface being 277 µm. The fibre laser treated surfaces also comprised of much smaller cracks in comparison with the as received surface (see example in Figure 9). Reduction in the surface hardness indicated that the laser surface treatment had softened the top (near) surface layer of the ceramic. From this it can be assumed that some degree of melting and solidification may have taken place during the laser/ ceramic interaction. Through this would have caused a localised ductile surface to have formed along with change in the surface composition. Further study is being undertaken to determine this effect.

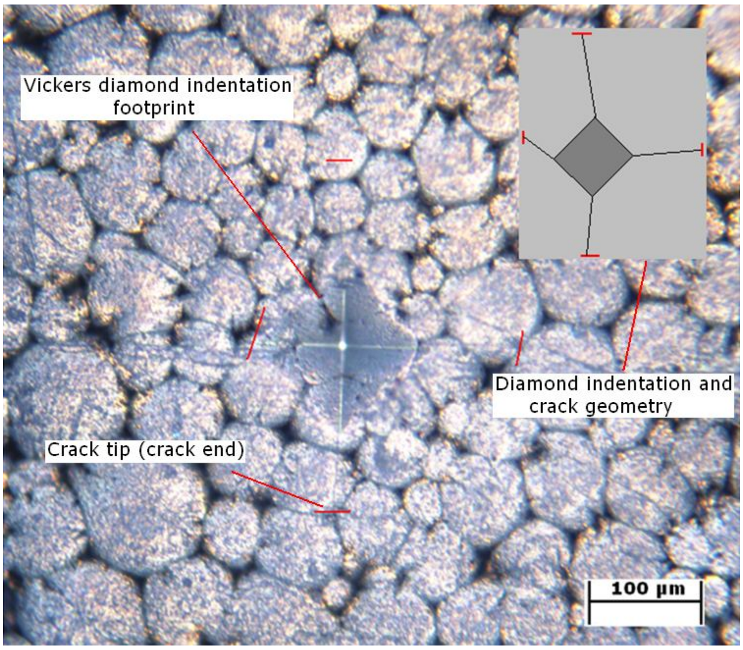


Figure 9: Fibre laser treated surface of ZrO_2 ceramic indented by a 5 Kg load, laser power = 150 W, 100 mm min^{-1}, 3 mm post size, (hardness = 654 (Hv), crack length = 232 µm, K_{1c} = 3.97 MPa m$^{1/2}$).

The final K_{1c} value for ZrO_2 after the fibre laser treatment was 5.62 MPa $m^{1/2}$. The highest K_{1c} value obtained for above the mean was 9.85 MPa $m^{1/2}$. The lowest value below the mean for was 2.97 MPa $m^{1/2}$ for ZrO_2 ceramics as presented in Figure 10. The K_{1c} values of the fibre laser treated ceramics were enhanced by 56 % for ZrO_2 in comparison with that of the as received surfaces. The values in Figure 10 fluctuate due to the softening of the treated surface that would have generated lower cracks during the indentation test. In those areas where the K_{1c} is high, indicate that the localised near surface layer has more resistance to crack propagation under cyclic loads or during the onset of any tensile stresses. The Young's modulus being another factor which also influenced this change in the ceramics K_{1c}. The Young's modulus was increased from 210 GPa (as received surface) to 260 GPa (laser treated surface) whilst determining the K_{1c}. This was because of the ratio of stress and strain being higher after conducting the laser treatment. Due to the way in which the Young's modulus contributing to the K_{1c} equation used; it is likely that the influence of the Young's modulus was significant in end value of the K_{1c} found in this investigation.

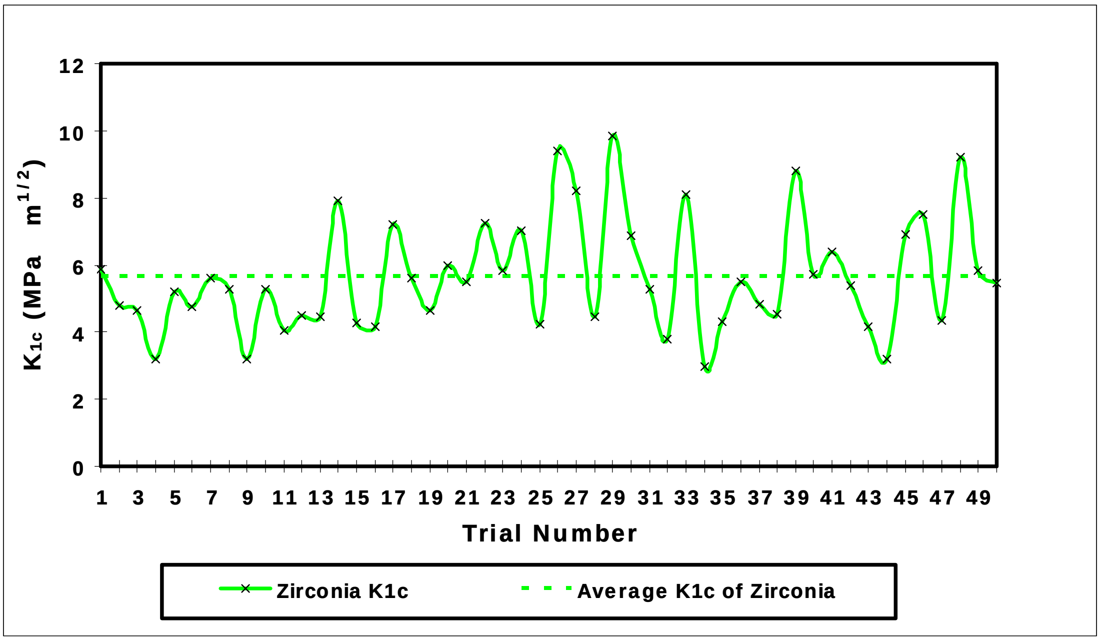


Figure 10: K_{1c} of the fibre laser treated surfaces of ZrO_2 ceramics from applying 5 kg indentation load.

Conclusions

Empirical equations as derived (Antis *et al* 1981) were used on the as received surfaces of the ZrO_2 ceramics to investigate the most suitable equation for calculating the K_{1c}. Palmqvist cracks were produced leading to half-penny median type cracks which confirmed the use of for the group of equations applied for the investigation. The results showed that equation 10 (K_{1c} = 0.016 $(E/Hv)^{1/2}$ $(P/c^{3/2})$ by Anstis, Chantikul, Lawn & Marshall was the most accurate and produced 42 % accuracy with ZrO_2 ceramics. The most influential parameter in calculating the K_{1c} was crack length as is it proved that longer cracks produced by the diamond indentation led to lower resistance for the ceramic to propagate a crack. Shorter cracks lengths exhibited higher resistance to indentation further resulting to improved K_{1c}. Hardness also influenced the ceramics K_{1c} as the results showed that high ceramic hardness produced bigger crack lengths which reduced the K_{1c} value. From varying the indentation loads; it was found that higher indentation loads produced bigger diamond footprints and generated higher crack lengths. It was also found that from increasing the Young's modulus had effected the K_{1c} value due to the ratio of stress over strain possibly increasing after the laser treatment. This resulted in producing a higher K_{1c} value. Despite the advantages, the Vickers indentation method to calculate the ceramics K_{1c} comprises of many flaws such as the results obtained from the hardness test heavily depend on operators ability to detect the crack lengths and its geometry, the ceramics ability to indentation response and the surface conditions that are used during the indentation test as smother surfaces would result to higher surface strength and influence the hardness value and hence, the resulting crack length. The K_{1c} results could be much accurate if a consistent material hardness value was obtained along with its crack geometry which could be found from employing other indentation techniques. Various other methods by using several other equations from the literature would produce variation in the K_{1c} value.

Comparison of the as received surface with the fibre laser treated surface as presented in Table 3 showed improvement in the of the K_{1c} value of the top (near) surface layer of the fibre laser treated ZrO_2 ceramics. This was due to the hardness and the crack lengths produced by the Vickers indentation were lower than that of the as received surface to increase the K_{1c} value. From this it was indicative that the laser treatment had softened the localised surface layer whilst melting the surface melted and solidified. Further investigation is being undertaken to elaborate this effect.

Table 3: Summery of the results illustrating an increase or decrease in the parameters tested for calculating the K_{1c} of the laser treated ZrO_2 ceramics.

	Average Surface Hardness (Hv)		**Average Surface Crack length (µm)**		**Average Surface K_{1c} (MPa $m^{1/2}$)**	
As received surface	983	0	277	0	2.48	0
Fibre laser treated surface	940	4 % decrease	171	38 % decrease	5.62	56 % rise

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Notations

Fracture Toughness	$\mathbf{K_{1c}}$
Hardness	**Hv**
Young's Modulus	**E**
Newtons	**N**
Average Flaw Size	**c**
Load (Kg)	**P**
Load Impact	**Pc**
Interior Cracks	**Ic**
Metre per minute	$\mathbf{m\ min^{-1}}$
Hot Isostatic Pressed	**HIP**
Cold Isostatic Pressed	**CIP**
Oxygen	$\mathbf{O_2}$

Zirconia Oxide	$\mathbf{ZrO_2}$
Alumina	$\mathbf{Al_2O_3}$
Silicon Nitride	$\mathbf{Si_3N_4}$
Kilo gram	**Kg**
Mega Pascal	**MPa**
Giga Pascal	**GPa**
Micro Metre	**µm**
Meters	**m**
Milimeters	**mm**
Litres	**l**
Meter Cubed	$\mathbf{m^2}$
Co-Ordinate Measuring Machine	**CMM**
Delta	**δ**
Beta	**ß**
Degrees Centigrade	**˚C**
Numerical Control	**NC**
Theta	**θ**
Average diagonal size	**D**