

# CO<sub>2</sub> Laser Surface Treatment Of (Si<sub>3</sub>N<sub>4</sub>) Engineering Ceramic

Maryam Q. Jaleel, Saad A. Mohammed Salih

**Abstract**— This research has utilized CO<sub>2</sub> laser to process silicon (Si<sub>3</sub>N<sub>4</sub>) engineering ceramic. Several aspects of laser beam-ceramics interaction can be understood in order to establish real change in the morphology, microstructure, density, hardness, surface toughness and fracture toughness parameter (K<sub>1c</sub>). This work has succeeded in modifying the properties. This has been proved by different tests applied, e.g., SEM. The most appropriate equation identified for the determination of the fracture toughness parameter K<sub>1c</sub> among several equations is:

$$K_{1c} = 0.016 (E/Hv)^{1/2} (P/c^{3/2})$$

**Index Terms**— engineering ceramics, fracture toughness parameter, CO<sub>2</sub> Laser, silicon nitride (Si<sub>3</sub>N<sub>4</sub>).

## I. INTRODUCTION

Engineering ceramics is the science and technology of creating objects from inorganic, non-metallic materials, formed by heating or cooling and may have crystalline or semi-crystalline formation with long-range order on atomic scale. They are manufactured either by the behavior of heat, or at lower temperatures using deposition reactions from high-purity chemical solutions [1,2]. Therefore, the focus of this work is to investigate the feasibility of this type of imported available engineering ceramics, i.e., silicon nitride Si<sub>3</sub>N<sub>4</sub>. The importance of this type comes from their exceptional mechanical and thermal properties; their applications have gradually increased on account of the desirable and longer functional life which often gives them a commercial advantage over the conventional materials in use. This type of engineering ceramics in particular are mainly being used to fabricate components in the motorsports industries, aerospace, automotive and several industrial sectors.

For these aspects, fracture toughness is a very important property since the low fracture toughness compared to metals and alloys is considered as one of the disadvantages of this ceramics. Thus, an increase in the fracture toughness would therefore lead to an enhancement in their functional life, better execution which in turn leads to decrease in the maintenance time and cost of the component parts of the system.

Laser treatment of this engineering ceramic offers diverse advantages in comparison with conventional processing techniques and much research has been conducted to develop applications. Low crack resistance and fracture toughness in comparison to metals can limit the use of this type of engineering ceramic, particularly for demanding applications. Therefore, a growing interest is founded in developing ceramic materials with high fracture toughness (K<sub>1c</sub>) for constructional applications **Malshe et al. 2006**

[3] has examined the CO<sub>2</sub> laser processing of a Si<sub>3</sub>N<sub>4</sub> ceramic to remove imperfections within the ceramic and more conducted a three point bending strength study of the Si<sub>3</sub>N<sub>4</sub> after the CO<sub>2</sub> laser surface treatment. **Shukla and Lawrence 2009** [4] have examined hitherto by utilizing a fiber laser to process engineering ceramics. In addition, the fiber laser was chosen because of its shorter wavelength radiation compared to the traditional lasers formerly applied for ceramic processing. The section of the CO<sub>2</sub> laser was adjusted that a contrast of two different wavelengths would be seen. It would be important to investigate further the influence of shorter wavelengths on the surface properties of the ZrO<sub>2</sub> and Si<sub>3</sub>N<sub>4</sub> engineering ceramic. In addition, Shukla and Lawrence have investigated the effects on the K<sub>1c</sub> by applying a fiber laser to treat a ZrO<sub>2</sub> and Si<sub>3</sub>N<sub>4</sub> engineering ceramics surfaces that showed changes in the K<sub>1c</sub> of both ceramics. However, the fiber laser effects are different to that of the CO<sub>2</sub> laser due to the different wavelength, beam conditions and the beam delivery system in spite of using identical parameters. This is the reason of a broader investigation was carried out by applying the CO<sub>2</sub> and the fiber laser on the ZrO<sub>2</sub> and the Si<sub>3</sub>N<sub>4</sub> engineering ceramic. Moreover, in spite of the Nd:YAG laser wavelength being in the same region as that of the fiber laser, the Nd:YAG laser does.

Fiber lasers also showed high brightness in comparison to the more traditional CO<sub>2</sub> and Nd:YAG lasers which mostly prevent deeper penetration, ability of showing finer spot sizes, longer depth of focus, and low cost per wattage which has been exhibited owing to its high brightness. As can be seen, this investigation is adequate as limited research has been conducted by utilizing fiber lasers to conduct the surface treatment of ceramics, especially for both engineering ceramics [5].

**Pratik Shukla 2010, 2011** [5,6] This research has specified the broader effects of different laser processing conditions, as well as characterization techniques, assessment and specification of a method to obtain the K<sub>1c</sub> and the thermal FEM of laser surface treated engineering ceramics. Also, the donating of laser-beam brightness as a parameter of laser processing and the effect on the engineering ceramics have been noticed from a basic viewpoint. The results of this research may now be adopted to develop ceramic fuel cell joining techniques and other applications that laser beam surface modification and characterization of engineering ceramics are needed [5].

## Calculating (K<sub>1c</sub>) depending upon the Indentation Method

Mechanical effects such as change in hardness were investigated in this work by employing the Vickers indentation method which was followed by determination of the fracture toughness parameter (K<sub>1c</sub>) by using empirical equations from the literature for laser surface treated silicon

nitride (Si<sub>3</sub>N<sub>4</sub>) engineering ceramic. Thereafter, scanning electron microscopy (SEM) was used to observe the integrity prior to laser surface treatment and (AFM) tests after the treatment. The hardness measurement was conducted in the standard manner at University of Technology. The Vickers indentation test has many advantages over other indentation techniques like the Rockwell test such as being simple, less time

consuming, cost effectiveness and easy setup. However, there are some constraints with the Vickers indentation techniques over the more conventional technique applied such as SENB and double-torsion (DT) method. These constraints as: (a) the dependence of the crack geometry on the applied indentation load and the properties of the material; (b) indentation deformation (non- using the SENB, CNB and DCB technique to determine the fracture uniform fracture progression or rapid fracture growth) such as lateral cracking [6]. Table (1) presents the literature K<sub>1c</sub> values as an example for comparison from

Table (1) Fracture toughness values of Si<sub>3</sub>N<sub>4</sub> engineering ceramics obtained by using the various indentation fracture methods as a comparison..

Indentation fracture method	Vickers	SENB	CN B	DCB
K <sub>1c</sub> of Si <sub>3</sub> N <sub>4</sub> (MPa.m <sup>0.5</sup> )	6.37	9.0	7.9	4.0

**Determination of the K<sub>1c</sub> by the Empirical Equation**

Hardness Measurement and the crack lengths from the Vickers indentation test are set into an empirical equation to calculate the engineering ceramic K<sub>1c</sub> [10]. Equations were modulated and used specifically to hard and brittle materials such as ceramics and glass by Ponton et al. [33]. The equations possess a specified empirical values particularly suitable for different ceramics. These equations were derived by the ceramics geometrical values which were determined from experimental means, of ceramics. However, this equation did not defined as applicable for a certain ceramic type.

Hence, the suitability of applying the various equations to the Si<sub>3</sub>N<sub>4</sub> engineering ceramics was not particularly defined. This is why it is required that an investigation must carried out in order to determine the most employable equation prior to investigating the K<sub>1c</sub> modifications through the laser irradiated ceramics. to first determine the K<sub>1c</sub> of the as-received surfaces of the Si<sub>3</sub>N<sub>4</sub>, then, the surfaces treated by laser . The selected equation applicable to calculate the K<sub>1c</sub>, by applying the Vickers indentation method is :

$$K_{1c} = 0.016 (E/HV) 1/2 (P/c3/2) \dots\dots\dots(1)$$

**Imported Samples**

For the purpose of this work, as fabrication did not fit, importing samples had to be done to pursue the target . The concentration was on importing one engineering ceramic type which is of distinctive importance in global industry for their special properties; This is silicon nitride Si<sub>3</sub>N<sub>4</sub>. The samples imported are shown in figures (1), with diameter 20 mm and thickness of 5mm.



Figure (1): Si<sub>3</sub>N<sub>4</sub> imported samples

The values of the as received properties of engineering ceramic are listed in tables (2)

Table(2) The value properties of si3o4 as received

Item	Sintering
Rockwell Hardness (HAR)	91-92
Volume Density (g/cm <sup>3</sup> )	3.0-3.2
Breaking Tenacity(MPa.m <sup>1/2</sup> )	5-6
Elasticity Modulus(G Pa)	290-320
Thermal Expansivity(m/K*10 <sup>-6</sup> /°c)	600
Thermal Conductivity(W/M K)	15

**CO<sub>2</sub> Laser Treatment**

Every ceramic piece been parted into five sections to be treated individually. The attempts were ranged from 20 to 100 W of laser power with a CW beam applied with a 10.6µm wavelength. The traverse speed ranged from 0.5 up to 1.7 mm/sec to determine the ultimate speed required to process engineering ceramic during the circumstances of this work. One or two parameters, for instance, power or speed or spot diameter were change one variable with fixed other parameters to determine the ultimate parameter window with fixed other parameters

**Experimental Steps:**

The samples were treated as follows:

- i. The experiments were conducted in ambient condition at known atmospheric temperature (27°C).
- ii. Preparations of the samples involve polishing in order to create a reflective surface plane prior to applying the Vickers indentation process.
- iii. For the Si<sub>3</sub>N<sub>4</sub> ceramics treated by CO<sub>2</sub> laser : every part of the individual sample have been treated as follows :
  - Varied laser power (20-100) W , with fixed laser speed (1.7 mm/sec)and laser spot diameter (2 mm).
  - Varied laser speed (0.5-1 mm/sec) with fixed laser power (100 W) and laser spot diameter (2 mm).
- iv. fixed parameter in order to maintain the sample and keep it as a whole uninterrupted and not to smash.

All the samples were coated with a black ink adopted from different references. This would assist in

reducing the reflection of the laser beam and would improve the absorption particularly. Treatment were continued as follows :



Figure (2): The CO<sub>2</sub> laser device (inside details)

EXPERIMENTAL TEST DEVICE:

**Scanning Electron Microscopy (SEM)**

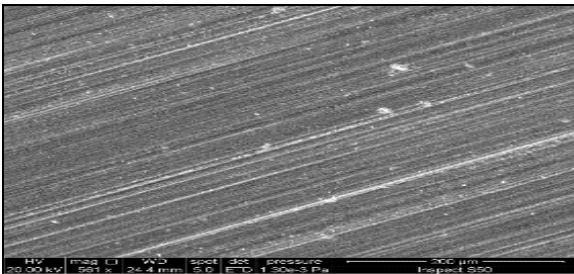
It is well known that Nano and Micro- structures need more high resolution microscope. Therefore, a scanning electron microscope of (model JSM-6460 LV, Japan) was used in this work to study the formed surface layer of applied type of engineering ceramic.

This device was used to test the morphological properties and the identification of the ceramic layer composition .

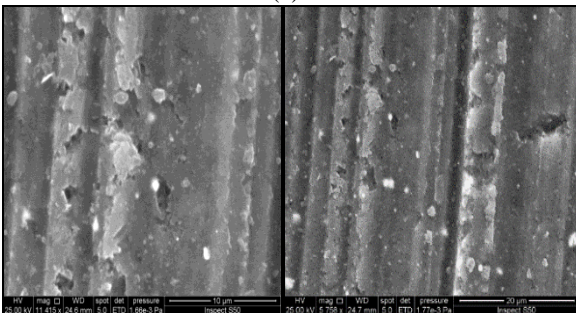
II. RESULTS AND DISCUSSION

**Si<sub>3</sub>N<sub>4</sub> ceramics treated by CO<sub>2</sub> laser**

In this Figure (3) shows the SEM images of the sample before and after treatment by the carbon dioxide laser.



(a)



(b1)

(b2)

Figure (3): SEM a top view image of the Si<sub>3</sub>N<sub>4</sub> sample: (a) before treatment (as received ),(b1) varied laser intensity after treatment and (b2) varied laser speed after treatment.

**Varied laser intensities**

Figure (4) shows the relation between different laser intensities applied and the resulted in Vickers's hardness for the Si<sub>3</sub>N<sub>4</sub> ceramics.

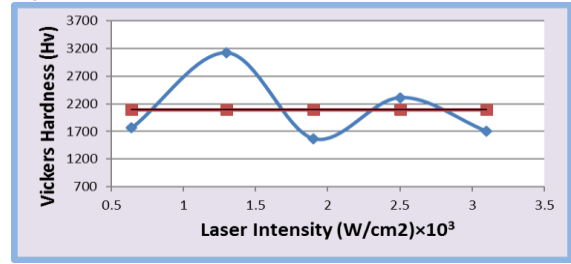


Figure (4): Hardness of Si<sub>3</sub>N<sub>4</sub> ceramic treated by different CO<sub>2</sub> laser intensities.

Intensities (blue line), The brown line represents the average hardness. The average hardness deduced by using ~ 1 kg load is ~ 2094 Hv. The wide fluctuation in the hardness values is not unusual. They already expected to be occurred due to several factors as :

- i. surface pre-existing micro-cracks.
- ii. occurred porous structure.
- iii. the fabrication process.
- iv. ceramics reaction to the diamond indentation.
- v. Defects and impurities on the next surface layer in comparison with the bulk hardness .
- vi. operator and machine finesse in measuring the sizes of the diamond indentations.
- vii. Not evenly oxide layer deposited after the CO<sub>2</sub> laser surface treatment has seemed to be slightly large in both in width and depth of the CO<sub>2</sub> laser treated surface as shown in Figure (5).

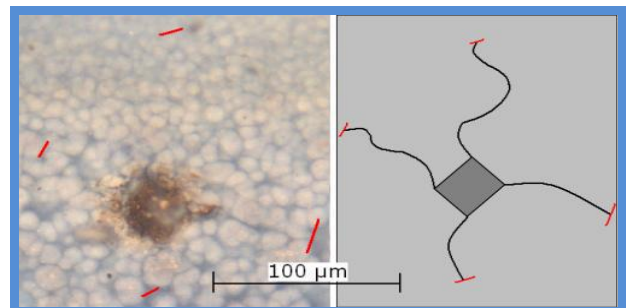


Figure (5): OM image (left) and the crack shape(right) of CO<sub>2</sub> laser processed surface of Si<sub>3</sub>N<sub>4</sub> engineering ceramic.

**Varied laser Speed**

In this Figure (6) shows the relation between different laser speeds applied and the resulted in Vickers's hardness for the Si<sub>3</sub>N<sub>4</sub> engineering ceramics.

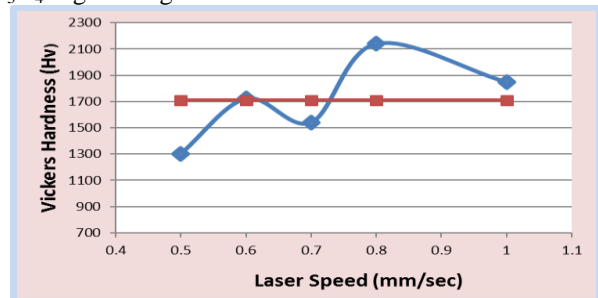


Figure (6): Hardness of Si<sub>3</sub>N<sub>4</sub> ceramic treated by different CO<sub>2</sub> laser speeds. The brown line is the average value.

The proportionality between the hardness values and laser speeds is obvious even for evident fluctuation of the hardness. The average fluctuation value is ~1710 Hv for 1 kg load. This proves the effect of speed with certain values on hardness, then, on K1c values. However, it needs to be careful to choose the right suitable speeds.

**K1c Results**

By applying Equation (1), K1c values can be calculated depending on data measured experimentally for every treated engineering ceramics, i.e., the Vickers hardness (converted to GPa), crack lengths and their Young Modulus value. The load used for all cases was 9.8 N stands for 1 kg which is the highest load available at the lab. These applied values and results would be depicted in separated tables. Recall that the as received K1c = 5-6 (M Pa m<sup>0.5</sup>) for the Si<sub>3</sub>N<sub>4</sub> samples

**For Varied CO<sub>2</sub> laser intensities**

Table (3): K1c results for every intensity applied with their hardness, Young Modulus and crack lengths values.

Intensity W/cm <sup>2</sup>	Hardness GPa	Young Modulus GPa	Crack length μm	Fracture toughness MPa.m <sup>1/2</sup>
0.64×10 <sup>3</sup>	17.3	310	21	6.631
1.3×10 <sup>3</sup>	30.6	310	32	2.74
1.9×10 <sup>3</sup>	15.4	310	19	5.851
2.5×10 <sup>3</sup>	22.7	310	31	3.25
3.1×10 <sup>3</sup>	16.7	310	24	5.45

**For Varied CO<sub>2</sub> Laser Speeds**

Table (4): K1c results for every laser speed applied with their hardness, Young Modulus and crack lengths values.

Laser speed mm/sec	Hardness GPa	Young Modulus GPa	Crack length μm	Fracture toughness MPa.m <sup>1/2</sup>
0.5	12.75	310	18.03	9.734
0.6	16.88	310	24.05	5.483
0.7	15	310	18.6	8.562
0.8	21	310	18.4	7.325
0.9	18	310	21.6	6.224

III. CONCLUSIONS

This work has elucidated diverse key issues related CO<sub>2</sub> laser interaction with Si<sub>3</sub>N<sub>4</sub> engineering ceramics over surface treatment.

The drawn conclusions that being summarized are :

1. The greater laser traverse speed and least intensity have the minimum effect on the surface of applied engineering ceramic.
2. For evaluating the fracture toughness property K1c of the as received this engineering ceramic, it can be specified that equation:  
 $K1c = 0.016 (E/Hv)^{1/2} (P/c^{3/2})$  was the most appropriate to employ for engineering ceramic.
3. A variation in the hardness and the crack length as results of the Vickers indentation was detected to be an effective parameter. This leads to a variation in the average value of the K1c (for the utilized conditions).
4. For Si<sub>3</sub>N<sub>4</sub> engineering ceramic, the spot diameter effects result in the highest values of K1c, followed by the speed change effects and the varied laser intensities. This indicates that choosing a certain intensity with suitable speeds and spot diameters would have the best influence.

REFERENCES

- [1] Reza Rowshan, “ Process Control during Laser Transformation Hardening”, PhD Thesis, UNIV. OF MISKOLC, Hungary, 2007.
- [2] Philippe Boch & Jean-Claude Niepce, “ Ceramic Materials. Processes, Properties and Applications”, Library of Congress Cataloging-in-Publication Data, 2006.
- [3] Malshe A, Sun Li, Jiang W, McCluskey WP. Effect of CO<sub>2</sub> Laser Surface Processing on Fracture Behaviour of silicon Nitride Ceramic. Journal of engineering materials and technology 2006; **128**: 460 – 467.
- [4] Shukla PP, Lawrence J, Wu H. On the Fracture Toughness of a Zirconia Engineering Ceramic and the Effects thereon of surface processing with fiber laser radiation. Proceedings of the I Mech E Part B 2009; 224.
- [5] Shukla PP, Lawrence J, Fracture Toughness modification by using a Fiber Laser Surface Treatment of a Silicon Nitride Engineering ceramic. Journal of Materials Science; Vol. 45 (21), 2010.
- [6] Pratik Shukla, “Viability and Characterization Ceramics”, PhD Thesis, Wolfson School of Mechanical and Manufacturing Engineering, Loughborough Univ., 2011.
- [7] George D. Quinn, “Fracture Toughness of Ceramics by the Vickers Indentation Crack Length Method: A Critical Review”, Ceramic Engineering and Science Proceedings Cocoa Beach, Jan. 2006.
- [8] D. Marinescu, Handbook of Advanced Ceramic Machining, CRC Press, (2007)
- [9] Lawrence, J. The characterization and feasibility of a two-stage ceramic tile grout sealing process using a high power diode laser, PhD thesis, Institute of Science and technology: University of Manchester 1999.
- [10] George D. Quinn, “Fracture Toughness of Ceramics by the Vickers Indentation Crack length Method: A Critical Review”, National Institute of Standards and Technology, 2006.

Nomenclature:

Symbol	Definition	Units
A	Water Absorption	%
c	Crack Length	μm
D	Bulk Density	g/cm <sup>3</sup>
E	Elastic Modulus	G Pa
HV	Hardness	G Pa
K1c	Fracture toughness	MPa.m <sup>0.5</sup>
P	Load	N
V	External Volume	cm <sup>3</sup>
W1	Dry Weight	g
W2	Weight of ceramics body in water	g
W3	Weight of the water_saturated ceramics body	g