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CO2 Laser Surface Treatment Of (Si3n4) Engineering Ceramic

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Abstract— **This research has utilized co2 laser to process silicon (Si3N4) engineering ceramic. Several aspect of laser beam –ceramics intersection can be understood in order to establish real change in the morphology microstructure, density, hardness, surface toughness and fracture toughness parameter (K1c).This work has succeeded to modify the properties. This has been proved by different test s applied ,e.g., SEM . The most appropriate equation identified for the determination of the fracture toughness parameter K1c among several equation is :**

K1c=0.016 (E/Hv) 1/2 (P/c3/2).

Index Terms— **engineering ceramics, fracture toughness parameter ,CO2 Laser, silicon nitride (Si3N4).**

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 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 is imported available engineering ceramics, i.e., silicon nitride Si_3N_4 .The importance of this type come 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 (K1c) for constructional applications **Malshe et al. 2006 [3]** has examined the CO_2 laser processing of a Si_3N_4 ceramic to remove imperfections within the ceramic and more conducted a three point bending strength study of the Si_3N_4 after the CO_2 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_2 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_2 and Si_3N_4 engineering ceramic. In addition, Shukla and Lawrence have investigated the effects on the K1c by applying a fiber laser to treat a ZrO_2 and Si_3N_4 engineering ceramics surfaces that showed changes in the K1c of both ceramics. However, the fiber laser effects are different to that of the CO_2 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_2 and the fiber laser on the ZrO_2 and the Si_3N_4 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_2 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 K1c 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 (K1c) 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 (K1c) by using empirical equations from the literature for laser surface treated silicon

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nitride (Si_3N_4) 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 K1c values as an example for comparison from

Table (1) Fracture toughness values of Si_3N_4 engineering ceramics obtained by using the various indentation fracture methods as a comparison..

Indentation fracture method	Vickers	SENB	CN B	DCB
K1c of Si_3N_4 ($MPa.m^{0.5}$)	6.37	9.0	7.9	4.0

Determination of the K1c 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 K1c [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_3N_4 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 K1c modifications through the laser irradiated ceramics. to first determine the K1c of the as-received surfaces of the $Si_3N_{4,}$ then, the surfaces treated by laser . The selected equation applicable to calculate the K1c, by applying the Vickers indentation method is :
K1c = 0.016 (E/HV) 1/2 (P/c3/2) ……….(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_3N_4. The samples imported are shown in figures (1), with diameter 20 mm and thickness of 5mm.

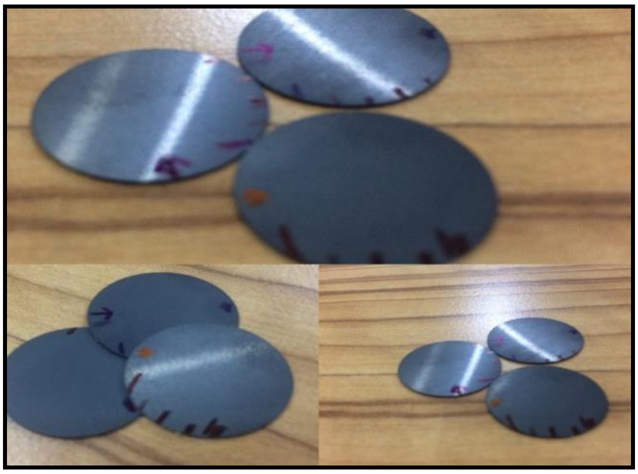


Figure (1): Si_3N_4 imported samples

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

Table(2) The value properties of si3o4 as receved

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

CO_2 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_3N_4 ceramics treated by CO_2 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_2 laser device (inside details)

EXPERIMENTAL TEST DEVICE:

Scanning Electron Microscopy (SEM)

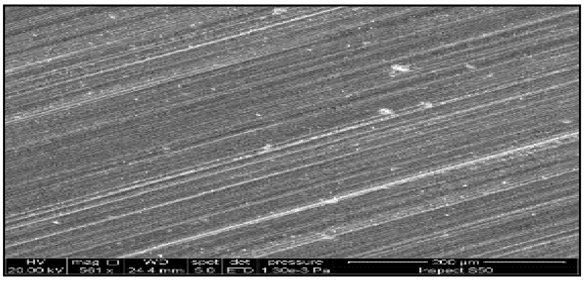
It is well known that Nano and Micro- structures need more high resolution microscope. Therefore, a scanning electron microscope of (model JSM-6460 LV, Japan) was used in this work to study the formed surface layer of applied type of engineering ceramic.

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

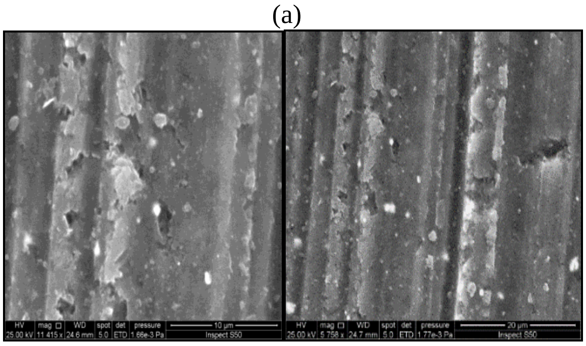
II. RESULTS AND DISCUSSION

Si3N4 ceramics treated by CO2 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_3N_4 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_3N_4 ceramics.

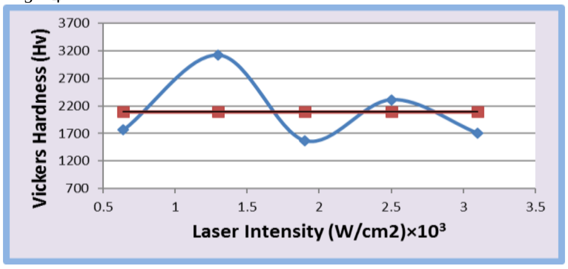


Figure (4): Hardness of Si_3N_4 ceramic treated by different CO_2 laser intensities.

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_2 laser surface treatment has seemed to be slightly large in both in width and depth of the CO_2 laser treated surface as shown in Figure (5).

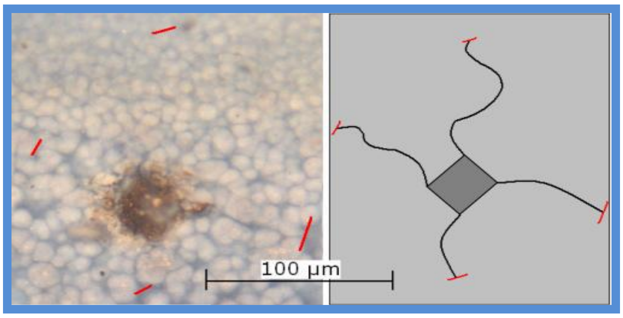


Figure (5): OM image (left) and the crack shape(right) of CO_2 laser processed surface of Si_3N_4 engineering ceramic.

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_3N_4 engineering ceramics.

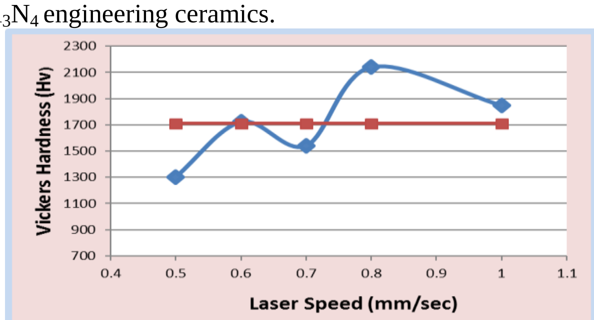


Figure (6): Hardness of Si_3N_4 ceramic treated by different CO_2 laser speeds. The brown line is the average value.

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 $m^{0.5}$) for the Si_3N_4 samples

For Varied CO_2 laser intensities

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

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

For Varied CO_2 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$^{1/2}$
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_2 laser interaction with Si_3N_4 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_3N_4 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.

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Nomenclature:

Symbol	Definition	Units
A	Water Absorption	/
C	Crack Length	µm
D	Bulk Density	g/cm^3
E	Elastic Modulus	G Pa
HV	Hardness	G Pa
K1c	Fracture toughness	MPa.m$^{0.5}$
P	Load	N
V	External Volume	Cm^3
W1	Dry Weight	G
W2	Weight of ceramics body in water	G
W3	Weight of the water_ saturated ceramics body	G