

A comparative study on the processing parameters during fibre and CO₂ laser surface treatments of silicon nitride engineering ceramic

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Abstract This paper demonstrates a comparative study of a relatively novel fibre laser and a conventional CO₂ laser to surface-process silicon nitride (Si₃N₄) engineering ceramic. The objective of the research is to investigate the threshold of the novel fibre laser and compare it to the conventionally used CO₂ laser to process Si₃N₄ engineering ceramic and to produce a laser surface treatment free from major surface cracking without using any of the pre- or post heating techniques as this would increase the cost of the process and add more expense to the product when considering a bigger view point. The results showed that the fibre laser surface processing of the Si₃N₄ engineering ceramic differed to that of the CO₂ laser as the Gaussian beam modes, the beam quality factors, wavelength and the beam delivery systems were different between the two lasers. This consequently had a different effect on the surface of the Si₃N₄ engineering ceramic. The CO₂ laser wavelength when surface treating the Si₃N₄ engineering ceramic was being absorbed more than that of the fibre laser as higher power density and same traverse speed was used to reach the threshold for the Si₃N₄ engineering ceramic.

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1 Introduction

1.1 Research background and rationale

It is important to study the process parameters prior to any laser processing. This is because it allows one to understand the materials behaviour and the capacity of the material to withstand the thermal energy induced by the laser beam. This is specifically important for ceramics as they are prone to cracking when exposed to thermal shock which is generally introduced during the laser–ceramic interaction. The focus of this paper is on a comparative study of a conventionally used CO₂ laser with a mid infra-red (MIR) wavelength and the novel fibre laser which comprises of a wavelength in the near infra-red (NIR) region. From this, it enables one to understand the difference in material interaction between the two wavelengths. This in turn allows one to improve laser processing parameters for ceramics with a similar chemical composition to that used in this research as all ceramics exposed to intense heat source such as a laser beam tend to behave differently. In addition, the objective is to process the Si₃N₄ engineering ceramic which is free from major surface cracking without using any of the pre- or post heating techniques prior to the laser surface treatment, as this would increase the cost of the process and add more expense to the product when considering a bigger view point.

Despite ceramics comprising of low thermal conductivity and high resistance to withstand heat or thermal energy; there is still a limit or a threshold which it can resist before it fails by either shattering via producing brittle fractures or propagation

of sharp brittle cracks. In general, failure of ceramic under thermal loading occurs due to the introduction of the thermal shock, particularly during intense energy beam such as a laser. This is further justified in this paper but the most important aspect is to induce just enough energy into the ceramic so that it does not crack. For this to be successful, it is required that correct use of laser power, traverse speed, spot size and the gas flow rate are used. Although the literature review indicated some guidelines towards the range of parameters applicable to the various ceramics, it is still unclear with respect to the threshold of the ceramics used for this research. This is also because all ceramics are different due to their processing history as the material composition (additives) and processing methods used for the ceramics employed in this research are somewhat different in comparison to that of the previous investigations. Therefore, a systematic method was adopted and is presented in this paper which utilizes the approach of changing one factor (parameter) at a time to obtain the optimum parameter window for the Si_3N_4 engineering ceramics by employing the a relatively new fibre and the conventional CO_2 laser. Laser power, traverse speed, spot size and the appropriate power density required to process the Si_3N_4 engineering ceramics were mainly investigated in order to reach the material threshold.

1.2 Previous laser surface treatment of ceramics

Much work has been conducted within the field of ceramic processing by using various industrial lasers [1–13]. Nonetheless, limited investigations to date have been conducted with respect to achieving a ideal surface treatment of various ceramics with respect to the ceramic being crack-pore- and defect-free [1–6]. Murray et al. [7–10] performed several investigations by using the a CO_2 laser to cut ceramics which used a pre heating method of the ceramic substrate to temperature up to $1,500^\circ\text{C}$ in a furnace and then performed the laser cutting process. Ester et al. [1] conducted an investigation on Al_2O_3 and ZrO_2 based oxide ceramics by employing a high-powered diode laser (HPDL). Laser-irradiated area of $50\text{ mm}\times 7\text{ mm}$ was said to have crack-free surface. This was performed by controlling the laser power, traverse speed and the sample temperature by pre heating the surface of the ceramic. Triantafyllidis et al. [2–6] performed several investigations on laser surface treatment of mainly Al_2O_3 based refractory ceramics by employing the HPDL. Earlier work of Triantafyllidis et al. identified solidification cracking due to the generation of very large temperature gradients which occur within the ceramics. Triantafyllidis et al. further showed that the refractory Al_2O_3 ceramic can be treated with a combination of laser source (HPDL beam trailed by a CO_2 laser or vice versa) to eliminate the crack propagation by temperature control [2, 3]. However, such methods were

not always repeatable with ease and efficiency, since it required timely set-up and arrangement to take place. Further investigation showed that crack-free surfaces improved the surface properties of the ceramic such as corrosion resistance, contact characteristics and surface morphology, contact angle, wetting and water permeability [3, 4]. Another investigation by Triantafyllidis et al. [5] stands out from the others as it used the HPDL to process refractory Al_2O_3 ceramics by using none of the post of pre heating methods which are conventional ideas in laser processing of ceramics. Triantafyllidis et al. reported that a crack-free surface treatment was possible with the parameter window speed being 0.4 mm/s and a power density of $6\times 10^2\text{ W/cm}^2$. This led to crack-free surface treatment for the Al_2O_3 refractory ceramics. However, these parameters are unique for the refractory Al_2O_3 only with the particular composition. Other ceramics such as the ZrO_2 and Si_3N_4 are somewhat different due to their chemical composition which also changes the mechanical and thermal properties and has an effect during laser processing.

Lawrence and Li conducted an experimental investigation for the differences between beam interaction characteristics of $\text{SiO}_2/\text{Al}_2\text{O}_3$ ceramics by employing a CW CO_2 , Nd: YAG, Excimer and HPDL [11, 12]. Laser absorption, fluence threshold, thermal loading and the beam interaction at the melt-pool was investigated by using the Beer–Lambert’s Law and Stefan solution. Absorption length, thermal loading and melt-pool characteristics were determined using the values from the experimental work presented by Lawrence and Li [11]. It was concluded that an evidence of re-solidification was found on the $\text{SiO}_2/\text{Al}_2\text{O}_3$ ceramics when applying CO_2 , Nd:YAG, excimer and a HPDL’s. The ceramic had undergone some level of melting and then re-solidified when exposed to all the laser sources applied except the excimer laser. This was because of the lower wavelength of the excimer laser. It was further confirmed that the excimer laser was said to have no effect to the melting of the $\text{SiO}_2/\text{Al}_2\text{O}_3$ ceramic [11].

Furthermore, Lawrence and Hao [13] used ceramics for biomedical applications which involved improvement of surface properties of ceramics by using industrial lasers on the Al_2O_3 and ZrO_2 ceramics. Other work by Lawrence and Li showed improvement in the surface properties of the same ceramics [14]. A 60 W high-powered diode laser was used to investigate the adhesion characteristics of Al_2O_3 , $\text{SiO}_2\text{–TiO}_2$, clay tiles and other ceramic tiles. The main property investigated was the contact angle which was enhanced. The laser beam was fired with adding four types of different liquids to the ceramic for wetting the material surface. Those were human blood, human blood plasma, glycerol, and 4-octanol. The results showed that due to the improvement in the material surface roughness, the contact angle was reduced which resulted in the material exhibiting

better adhesion characteristics. Evidence of some oxidation was also found with the investigation with formation of glass element, indicating that the composition of the ceramic was changed. This resulted into the material comprising of better adhesion characteristics [14].

Sun et al. [15, 16] investigated the effects of CO₂ laser surface processing on a Si₃N₄ and eliminated imperfections within the ceramic by applying the CO₂ laser beam. The researchers found that the fracture behaviour was considerably affected by surface treating the Si₃N₄ with a CO₂ laser. Fracture origins from the machining process and bending strength were improved. A four-point bending test, fractographic analysis and SEM micrographs were used to analyse the material's surface integrity. Sun et al. used a CO₂ laser to minimise the detrimental effects caused by grinding as the heat from the sliding motion created friction. Laser surface treatment was done after severe grinding of the ceramic in order to remove the mechanically induced cracks. Both high- and low-powered CW CO₂ lasers were used to treat the Si₃N₄ surface. It was found that the condition of grinding has a big influence on the fracture strength of the Si₃N₄. Longitudinal direction grinding in comparison with transverse directional grinding demonstrated much more resistance to fracture [15, 16]. The laser used in the work of Sun et al. was a square-shaped beam. This type of beam usually has a "top hat" end profile which means that the power distribution during its focus would be uniform. Although, this was not clearly defined in [15, 16], as the CO₂ laser beams are conventionally of TEM₀₀ Gaussian beam mode. The Gaussian configuration of TEM₀₀ (comprised of a ring-shaped end profile laser beam). So, the distribution of power for this type of beam would not be uniform throughout its surface area and this is desirable in order to obtain a uniform effect on the surface during the treatment. Results from the fractographic analysis showed that there were two types of cracks found; machine induced cracks and inherent flaws from the material (porosity). This should, however, be the case with Si₃N₄ due to its characteristic and material structure. The depth of the machining cracks was between 25 to 50 µm. Sun et al. also stated that due to the viscous flow of the glassy phase, the surface region of the material has undergone reorganisation and relaxation during the CO₂ laser processing. Compressive stresses were induced, as residual strain from machining process was released by the CO₂ laser processing. With longer laser processing time and higher power density, more relaxation of residual strain occurred. The important factor was the difference between the residual stresses in the treated samples to the samples which were untreated (as-machined). The difference was found within the material's surface morphology, microstructure and uniformity which were not further mentioned in any detail [15]. It was assumed that the surface integrity

was changed and improved by the secondary glassy phase (YSiAlON) which underwent a reflowing and rebinding process [15, 16]. This was because the temperature during the CO₂ laser processing was measured to be higher than the stable equilibrium upper temperature of the secondary glassy phase. It was found that softening (and possibly melting) of the secondary glassy phase caused infiltration in the surface defects. As the laser power density increased, the ability of the secondary glassy phase to rebind and flow.

Morita et al. [17] worked with Si₃N₄ to produce a crack-free surface by using an Nd:YAG pulsed laser processing of ceramics. The increase in peak output power caused the crack propagation and generation of a thick re-cast layer. The peak power was said to be kept low as possible and the pulse duration to be as short as possible for a crack-free processing of ceramics [17]. This also allows reduction in the thermal stress induced and into the material which justifies the elimination of crack generation during the process. The strength of the laser-treated samples was also compared with the strength of ground and polished surface by a diamond wheel [17]. The strength of the laser-treated samples was reported to be 10% to 20% reduced in comparison with the diamond polished sample due to the residual compressive stress layer being removed by the laser process [17].

Li et al. [18] conducted a three-dimensional numerical model for a convection–diffusion phase change process during laser melting of ceramic materials. The results showed that the both the numerical and the experimental models were in good agreement. Moreover, the absorption and liberation of latent heat of fusion during melting and solidification interfaces were more significant than the flow of fluid in affecting the temperature spread as well as the final shape and size of the molten area. Further analysis indicated that the temperature and velocity fields simulated involving both latent heat of fusion and fluid flow are to be considered. Several previous investigations have revealed interesting results when using industrial lasers (high powered) to surface treat refractory ceramics. Wang et al. [19–21] conducted studies using refractory ZrO₂ and Al₂O₃ ceramics to investigate the microstructural characteristics after melting of the ceramics by using a CO₂ laser. Surface cracking was found in the treated area, but changes to the laser parameters led to modified surface composition and morphology. Further work by Wang et al. [22] was on using an Nd:YAG laser to surface treat refractory ceramics by adding nano-particles to modify the surface density and the corresponding microstructure. The laser treatment was conducted prior to and after adding the nano-particles. Results linking to their previous investigations [19–21] showed dendrites that were much finer after the addition of the nano-particles.

Various aspects are now understood from having conducted this literature review. Those are: the use of a double laser beam technique, pre- or post heating of the ceramics prior to the laser process as well as the use of finite element and experimental model. The use of double laser sources (a leading and a trailing laser beam) as suggested from previous research will not be used. This is because it will complicate the experimental set-up, increase the length of time and because it is not always physically possible to attach two lasers together. Pre- or post heating of the Si_3N_4 will also be avoided as this will lengthen the laser surface treatment process.

2 Experimental methods

2.1 Details of the experimental material

A cold isostatic pressed (CIP) Si_3N_4 engineering ceramic was used with 90 wt.% Si_3N_4 , 4 wt.% Yttria, 4 wt.% Al_2O_3 and 2 wt.% other, unspecified content by the same manufacturer. Each of the samples was obtained in a bulk of $10 \times 10 \times 50 \text{ mm}^3$ (see Fig. 1) made from a specific rubber mould to a specified dimension from which all ceramics were CIPed. This was because the dimensions shown in Fig. 1 were best suited for the laser processing experiments. The surface roughness was $1.56 \mu\text{m}$ (as received from the manufacturer). The experiments were conducted in ambient condition at a known atmospheric temperature (20°C). Laser-induced surface cracks on the Si_3N_4 engineering ceramic were evaluated and the changes in the surface morphology were observed by employing an optical microscopy (Optishot; Nikon Ltd.) and assisted software for surface measurement. At each operating condition, the

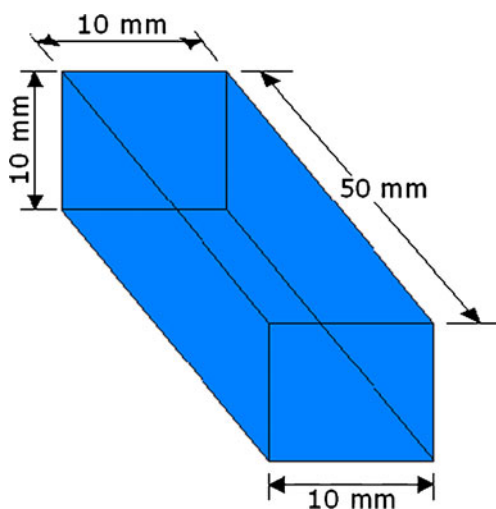


Fig. 1 A schematic diagram of the experimental work-piece of the Si_3N_4 engineering ceramic

crack geometry was checked after laser processing and then the laser process conditions were modified to achieve the best surface possible with minimal cracks. However, it is difficult to measure the inhibited surface cracks as they are continuous and or in various size and magnitude. Also, they often form bridges with other cracks in distance on the same surface profile. Owing to this, it is consistently very hard to define exact dimension of the surface cracks. Therefore, the use of the terminology of “very few cracks” to “high cracking” complimented by the use of colour key to define the crack type was implemented in the graphical figures. All surfaces of both the Si_3N_4 engineering ceramics to be treated were marked with black ink coating prior to the laser surface treatment to enhance the absorption and to allow the laser beam to further penetrate into the surface. Initial experiments showed that the black ink coating helped the beam to absorb better into the material. Furthermore, it was necessary to conduct like by like experiments with both lasers types and by using identical material surface conditions so a true comparison of the effects of the two lasers can then be further performed. The black ink coating was generally removed by the CO_2 or the fibre laser surface treatment and was not found to have any further effect on the Si_3N_4 engineering ceramic after the laser surface interaction had taken place. Despite the absorption for the two lasers being identical due to the coating, it is postulated that higher brightness (power per unit area) of the fibre laser would have led to better absorption in comparison to the CO_2 laser [23].

2.2 Details of fibre laser surface processing

A 200-W fibre laser (SPI-200c-002; SPI, Ltd.) emitting a CW mode beam at a wavelength of $1.075 \mu\text{m}$ was used in this work. The fibre laser comprised of a Gaussian beam configuration of TEM_{00} with a beam quality factor of $M^2 = 1.2$. Due to the cost of ceramics being high, it was difficult to explore so many parameters in relation to one another as it required high number of ceramic sample utilization. Thus, only selected parameters were systematically explored due to limited availability of experimental samples. In any case, experiments were conducted by varying one parameter at a time and by keeping the other parameters constant. Therefore, the laser power was varied from 25 to 200 W (max laser power) and simultaneously, the traverse speed was varied from 25 to 500 mm/min by keeping the focal position constant for the Si_3N_4 engineering ceramic. From these experiments, it was found that laser power of 143.25 W, traverse speed of 100 mm/min for the Si_3N_4 with a spot size of 3 mm was found to be ideal constant to use in order to conduct the surface treatment. Furthermore, identification of the range of laser power and power density required to reach the material threshold was achieved and are represented by graphical

means. The processing gas used for this set of experiment was compressed air which was supplied at a flow rate of 25 l/min. Initial experiments showed that the flow rate in excess of 25 l/min was blowing off excessive material during the laser–ceramic interaction. This was significant during surface cracking of the ceramic as the cracked surface was eventually blown away and produced a large crater. Lower gas pressure would result to lower interaction between the laser beam and the ceramic. Hence, 25 l/min was found to be appropriate for this study as this prevented the ceramic debris from travelling upwards into the lens and also protected the optics. Programming of the laser was conducted using SPI software which integrated with the laser system. A 50-mm line was programmed using numerical control (NC) programming as a potential beam path which was transferred by a .dxf file. The nozzle indicated in Fig. 2 was removed for all experiments. This was to obtain a wider spot size based on the fact that laser welding in general is conducted with a wider spot diameter which distributes the power on a bigger surface area. This is required for conducting the surface treatment also where the beam is focused on a larger surface area and has restricted depth of penetration but has a larger surface distribution. Therefore, a larger-diameter laser beam was applied for the study herein. This was more suitable for performing the laser surface treatment as opposed to laser cutting, drilling or hardening where high power density is required in the smaller surface area to penetrate through the

material. In this case, a defocused beam allows the energy to distribute over a wider surface area which prevent thermal shock and sever cracking of the ceramics. Nevertheless, the defocused beam yet remained Gaussian.

2.3 Details of CO₂ laser surface processing

A 1.5 kW, Everlase S48, Coherent, CO₂ laser was employed to conduct experiments on both the ZrO₂ and the Si₃N₄ engineering ceramics as presented in Fig. 3. The CO₂ laser comprised of a Gaussian beam configuration of TEM₀₁ with a beam quality factor of $M^2=1.3$. Likewise to the fibre laser surface treatment, one parameter was changed at any one time in order to determine the ultimate parameter window for CO₂ laser surface treatment of the Si₃N₄ engineering ceramic. The trials ranged from 50 to 200 W of laser power with a CW beam applied with a 10.6 μm wavelength while the beam spot size was kept constant at 3 mm with a gas flow rate of 25 l/min by using compressed air as the processing gas. The traverse speed ranged from 25 to 700 mm/min to determine the ultimate speed required to process the Si₃N₄. Programming of the laser was conducted by using an independent software which integrated with the laser machine. A 50-mm line was programmed by using NC programming as a potential beam path transferred by a .dxf file. Stand-off distance between the nozzle and the work-piece was kept to 16 mm in order to obtain a focal spot size of 3 mm. Parameters used for the CO₂ laser surface treatment were not directly comparable to those of the fibre laser surface treatment due to the difference in the wavelength and the nozzle shape and diameter as well as the high-powered laser not being able to execute stably at lower laser powers.

3 Results and discussion

3.1 Fibre laser surface processing

A systematic approach was used in order to find the threshold of the Si₃N₄ engineering ceramic. The laser power was changed whilst other parameters such as the spot size of 3 mm, traverse speed of 100 mm/min and gas flow rate of 25 l/min were kept constant during the fibre laser surface treatment. Table 1 show the range of powers used from 25 to 200 W and from 694 to 4861 W/mm² along with other associated parameters. During each stage, the effects of the laser–ceramic interaction were recorded were it was found that the 143.25 W or 3,979 W/mm² was ideal for producing a surface treatment that comprised of very few cracks on the surface of the Si₃N₄ engineering ceramic. As more laser power was introduced at a constant speed, the surface of the Si₃N₄ began to show some effects. Those

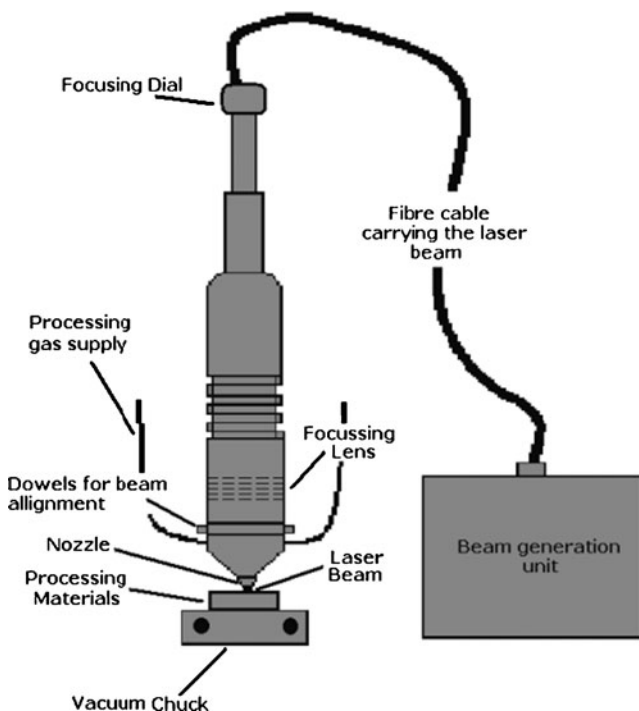
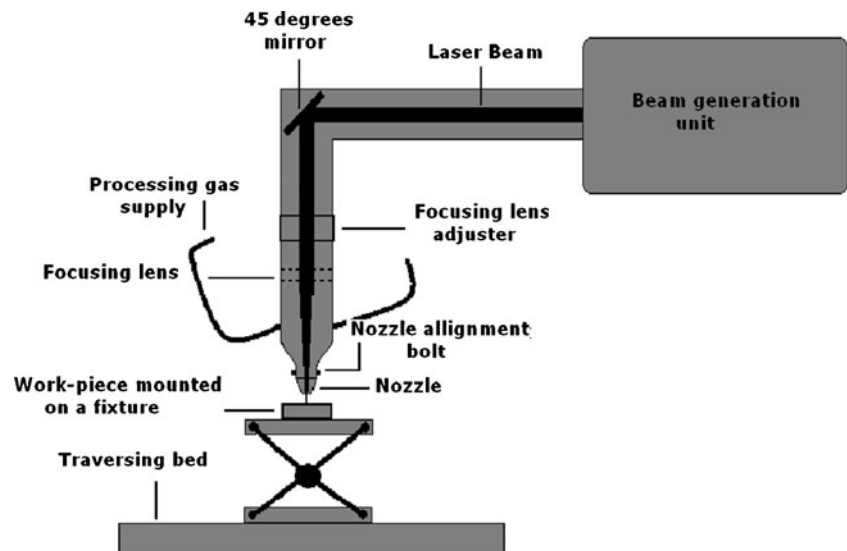


Fig. 2 Schematics diagram of the experimental set-up of the fibre laser treatment of the Si₃N₄ engineering ceramic

Fig. 3 Schematic diagram of the experimental set-up of the CO₂ laser surface processing of the Si₃N₄ engineering ceramics



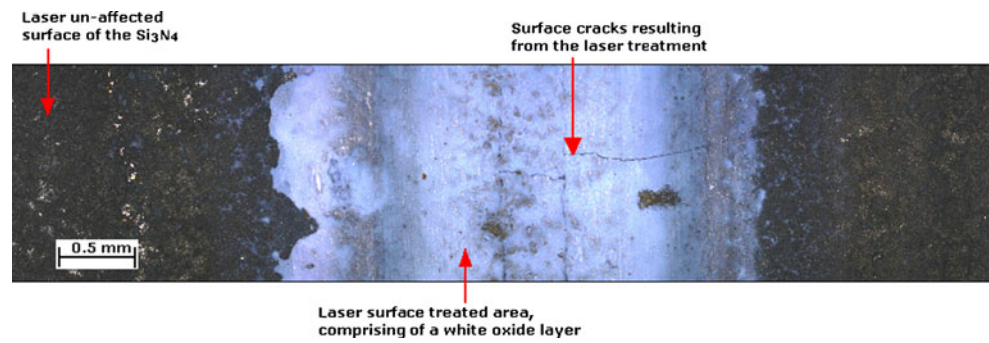
are a change in colour (see Fig. 4), appearance of few surface cracks to medium and then produced high cracking profile. The change in colour resulted from the laser–Si₃N₄ interaction as the surface of the Si₃N₄ was oxidised as result of the thermal exposure at elevated temperature. Owing to this, a formation of a new surface layer was produced which changed the surface characteristics as seen in Fig. 4. Further, it can be said that a change in composition has taken place as the fibre laser-irradiated surfaces of the Si₃N₄ was altered to form SiO₂ [24, 25]. This finding was also confirmed to be true by the work of Lysenko et al. [24]. Lysenko et al. reported that the SiO₂ results from heating silicon at temperatures up to 1,600°C. The fibre laser processed Si₃N₄ had changed composition as seen from Fig. 4 after being exposed to the laser beam at temperatures above 1,600°C which are confirmed from the previous investigations by the authors of this paper [26, 27]. The effects are somewhat similar for both the fibre and the CO₂ laser surface treatments also due to the temperature being above that previously mentioned.

It was found from the experimental investigation that laser power of 143.25 W at a speed of 100 mm/min was the most ideal and produced the lowest cracks during the fibre laser surface treatment. In addition, the traverse speed was then investigated and varied from 25 to 500 mm/min whilst keeping the laser power of 143.25 W and a spot size of 3 mm constant. The effects were then observed in terms of the surface cracks generated as result of the fibre laser–Si₃N₄ interaction. Lower traverse speeds resulted to more laser-material interaction time which inherently produces high surface cracking. As the traverse speed increased, the laser–ceramic interaction time also increased. Owing to this, lower thermal energy into the ceramic was induced and hence, reduced the cracks propagation. At a speed of 25 mm/min, evidence of high cracking was found as opposed to 500 mm/min. This is where there were no effects found on the Si₃N₄ engineering ceramic. The most ideal speed as shown in Table 2 was 100 mm/min and generated very few cracks during the fibre laser surface treatment.

Table 1 Effects of varying of laser power by using a 3 mm constant size spot at the traverse speed of 100 mm/min and gas flow rate of 25 l per min after fibre laser surface treatment of the Si₃N₄ engineering ceramic

Experiments	1	2	3	4	5	6	7	8	9	10
Laser Power (W)	25	50	75	100	125	137.5	143.25	150	175	200
Power Density (W/mm ²)	694	1389	2083	2278	3472	3819	3979	4167	4556	4861
Speed (mm/min)	100	100	100	100	100	100	100	100	100	100
Effects										

Fig. 4 A optical microscopic image of the surface morphology of the CO₂ laser-treated Si₃N₄ engineering ceramic showing the laser unaffected surface, the laser-treated oxidized surface and the geometry of the laser-induced surface cracks



It was found that the most ideal power for the fibre laser surface treatment of Si₃N₄ ceramic was 143.25 W which was applied at 100 mm/min. Therefore, the effects produced by varying the spot size needed to be explored. Hence, the beam diameter of the fibre laser was varied from 0.5 to 4.5 mm whilst keeping a constant laser power, traverse speed and gas flow rate as presented in Table 3. In general, the smallest spot size produces high power density in comparison to a wider spot diameter. This would affect the laser processing in a detrimental way. This is also true for the results in this study as the smallest spot size used (0.5 mm) resulted to producing the highest surface cracks and the largest spot size used (4.5 mm) barely changed the colour of the Si₃N₄. A spot size of 3 mm used with a laser power of 143.25 W had produced 3979 W/mm² of laser power density with a traverse speed of 100 mm/min had generated the most desired fibre laser surface treated zone in terms of producing a lowest cracked surface.

The effects of the laser parameters can be further presented with respect to the crack propagation of the Si₃N₄ during the fibre laser surface treatment. Figure 5 shows that the increase in traverse speed results to the laser beam having lesser effect. Evidence of high crack propagation as found with traverse speed under 100 mm/min.

With increasing traverse speed, the laser-induced cracks had began to reduce until there was no physical effect on the surface of the Si₃N₄ beyond 450 to 500 mm/min. Figure 6 showed the effects of the laser power and the power density on the fibre laser-irradiated surface which have a close relationship. This is because the power density is dependent on the laser power and the diameter of the laser spot used. Also, with increased laser power and increased power density, the propagation of cracks and the effects on the ceramic would increase due to high energy being induced and therefore, would increase the temperature at the laser–ceramic interface. This in turn would also increase the thermal shock and would characteristically lead to propagation of a crack.

Figure 7 presents the effects of the laser power and the traverse speed with respect to cracking on the fibre laser-irradiated surface of the Si₃N₄ and demonstrates that with increasing speed and increasing power the effects also becomes distinct. But, high surface cracking begins to appear when applying high laser power and low traverse speed. Therefore, a combination of the power and speed was important and 100 mm/min at about 143.25 W was ideal to use in terms of generating a fibre laser-treated surface and comprised of the lowest surface cracks. In

Table 2 Effects of varying the traverse speed by using 143.25 W of constant laser power, a 3 mm spot size and a gas flow rate of 25 l/min after fibre laser surface treatment of the Si₃N₄ engineering ceramic

Experiments	1	2	3	4	5	6	7	8	9	10	11	12
Laser Power (W)	143.25	143.25	143.25	143.25	143.25	143.25	143.25	143.25	143.25	143.25	143.25	143.25
Power Density (W/mm ²)	3979	3979	3979	3979	3979	3979	3979	3979	3979	3979	3979	3979
Speed (mm/min)	25	50	75	100	150	200	250	300	350	400	450	500
Effects	Red	Red	Red	Light Blue	Blue	Blue	Blue	Green	Green	Green	Green	Grey

Table 3 Effects of varying the spot size by using 143.25 W of constant laser power, traverse speed of 100 mm/min and a gas flow rate of 25 l/min after fibre laser surface processing of the Si₃N₄ engineering ceramic

Experiments	1	2	3	4	5	6	7	8	9
Spot Size (mm)	0.5	1	1.5	2	2.5	3	3.5	4	4.5
Power (W)	143.25	143.25	143.25	143.25	143.25	143.25	143.25	143.25	143.25
Power Density (W/mm ²)	143250	35813	15917	8953	5730	3979	2923	2238	1769
Speed (mm/min)	100	100	100	100	100	100	100	100	100
Effects									

addition, as the power density is increased and the traverse speed is decreased, the propagation of the surface cracks also increased. Traverse speed of 100 mm/min and the power density of 3979 W/mm² was found to be appropriate to generate the lowest surface cracks on the Si₃N₄. Lower spot size generally produces high power input and larger power density which in relation to this work consequently propagates high surface cracks on the Si₃N₄ ceramic. It was found that at constant laser power, and traverse speed, the spot size below 3 mm had generated high surface cracks as the fibre laser beam diameter was altered from being large to small.

3.2 CO₂ laser surface processing

A similar approach was used to obtain the most ideal parameters for the CO₂ laser surface processing as to that of the fibre laser. Table 4 shows that the traverse speed was initially selected at constant parameter of 100 mm/min to

determine the most ideal laser power. Along with that, the supply of the processing gas was kept as a constant to 25 l/min. The power density was changed characteristically, as the laser power was changed. At 25 W, no significant effects were found. As the power was raised, the CO₂ laser had began to create some effect by producing discolouration of the Si₃N₄ which was identical to that shown in Fig. 4. Moreover, small cracking had then begun to occur beyond 200 W.

Table 5 shows the use of constant laser power (200 W), power density of 5,556 W/mm², whilst the traverse speed was varied from 25 to 400 mm/min. The effects here were similar in comparison to the fibre laser as the traverse speed below 100 mm/min showed evidence of high cracking. Between the traverse speeds of 100 to 125 mm/min, cracking was reduced to very few cracks. At the same time, discolouration was found until 350 mm/min where only some effects can be seen of the CO₂ laser. Beyond

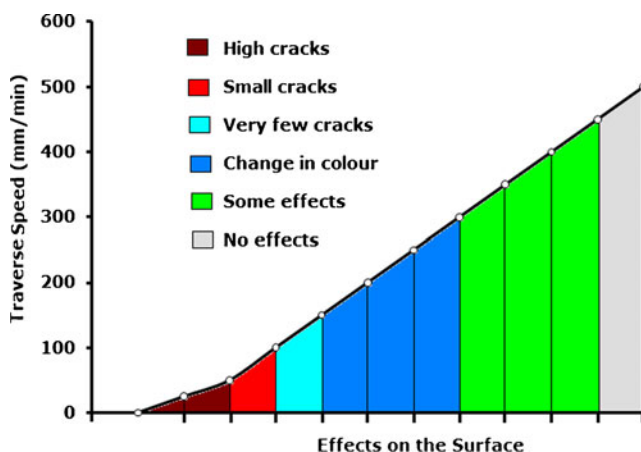


Fig. 5 Effects of the traverse speed on the surface of the Si₃N₄ engineering ceramic after fibre laser surface processing

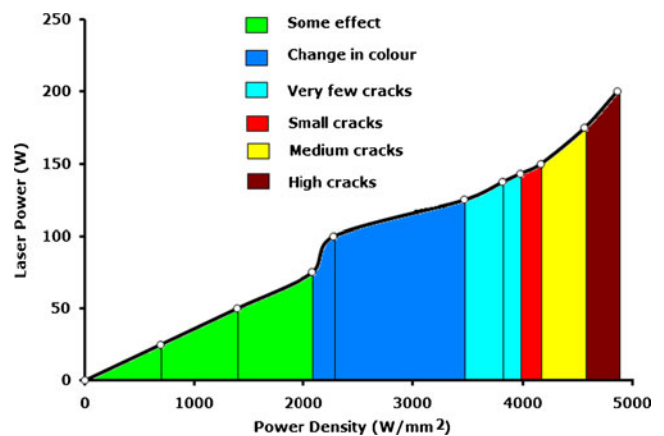


Fig. 6 Relationship between laser power and power density which shows the effects of the fibre laser surface treatment of the Si₃N₄ engineering ceramic by keeping a constant traverse speed of 100 mm/min, 3 mm spot size and 25 l/min gas flow rate

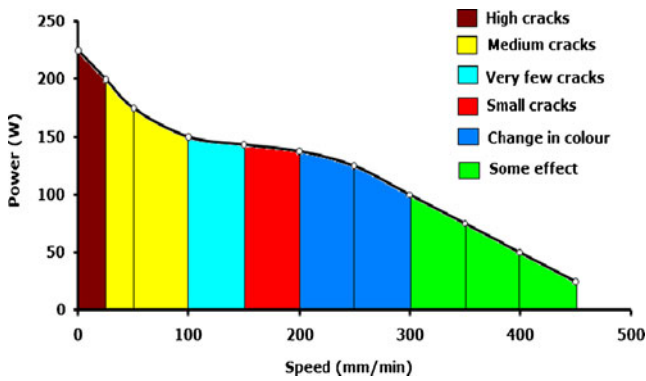


Fig. 7 Relationship between laser power and traverse speed which shows the effects of the fibre laser surface treatment of the Si₃N₄ engineering ceramic by keeping a constant spot size of 3 mm and 25 l/min gas flow rate

250 mm/min, no effect was to be seen on the Si₃N₄ engineering ceramic.

The relationship of power density and laser power with the crack propagation during the CO₂ laser surface interaction is presented in Fig. 8. This shows that smallest cracks occurred above 175 to 200 W. Laser power below 175 W only influenced the Si₃N₄ ceramic as there was some discolouration. Evidence of high cracking was found beyond 200 W. Surface treatment comprising of smallest surface cracks was found at the traverse speed of 100 and 150 mm/min as shown in Fig. 9. High level of cracking was seen under the traverse speed of 50 mm/min. As the traverse speed increased, the cracks begun to reduce. The smallest cracks were seen at 100 mm/min and beyond this speed showed only discolouration and small effects on the surface of the Si₃N₄ engineering ceramic were found (Table 6).

Figure 9 shows the relationship between the laser power and the traverse speed where high cracking occurred with traverse speed below 100 mm/min and beyond where few cracks were found during the CO₂ laser interaction of the

Si₃N₄ ceramic. The cracking effect became less as the traverse speed was increased. The variation in spot size during the CO₂ laser surface treatment of the Si₃N₄ surface had begun to produce high cracking at spot size of 2 mm despite the power density being low. However, with increasing spot size, the cracking had reduced and very few cracks were generated by the CO₂ laser from using a spot diameter of 3 to 4.5 mm, with power density being raised to 6,000 W/mm². However, beyond 4.5 mm of beam diameter, there was only evidence of change in colour despite the laser power density increasing to 7000 W/mm². This showed that the spot size had considerable contribution to influence the surface treatment as it controlled the thermal energy being induced in to the ceramic.

3.3 Mechanism of crack propagation, potential impact and significance

The most difficult aspect during the laser surface treatment is that the material tends to undergo a degree of thermal shock. This is the main reason for the occurrence of failure within ceramics. Ceramics are hard and brittle, prone to cracking, with low thermal conductivity, high thermal expansion and low toughness. Due to this, ceramics have the tendency to generate cracks during exposure to high temperature. Moreover, it is the exposure to temperature change which eventually leads to failure. Thermal shock is produced when high temperature is introduced to the ceramic whilst the ceramic is in state of ambience (see Fig. 10). This is particularly the case during laser surface treatment as the high power density from laser beam focused on a small surface area interacts with the ceramic which body is at 20 to 25°C temperature. During the laser–ceramic interaction, the processing temperature around almost above 2,000°C (depending on the laser parameters and the type of laser used). Most probably, the rise of temperature at the ceramic surface would be from ambient

Table 4 Effects of varying of laser power by using a 3 mm constant spot size at the traverse speed of 100 mm/min and gas flow rate of 25 l/min after the CO₂ laser surface treatment of the Si₃N₄ engineering ceramic

Experiments	1	2	3	4	5	6	7	8	9
Laser Power (W)	25	50	75	100	125	150	175	200	225
Power Density (W/mm ²)	694	1389	2083	2778	3472	4167	4861	5556	6250
Speed (mm/min)	100	100	100	100	100	100	100	100	100
Effects									

Table 5 Effects of varying of the traverse speed by using a 3 mm constant size spot with the laser power of 200 W, 3,819 W/mm² power density and gas flow rate of 25 l/min after the CO₂ laser surface treatment of the Si₃N₄ engineering ceramic

Experiments	1	2	3	4	5	6	7	8	9	10	11	12
Laser Power (W)	200	200	200	200	200	200	200	200	200	200	200	200
Power Density (W/mm ²)	5556	5556	5556	5556	5556	5556	5556	5556	5556	5556	5556	5556
Speed (mm/min)	25	50	75	100	125	150	175	200	250	300	350	400
Effects	High cracking	High cracking	High cracking	Very few cracks	Very few cracks	Change in colour	Change in colour	Change in colour	Change in colour	Change in colour	Some effects	No effects

to about 2,000°C within a second and then is made to cool as the laser beam has moved to another area. In this case, a temperature difference has occurred already which in turn would expand the ceramic as it tries to absorb the heat. During this expansion, the areas within the bulk are somewhat cooler in comparison, which are working against the induced thermal energy and attempts to contract the ceramic (see Fig. 10). However, the high thermal energy needs to escape, hence, the expansion (which is in form of a tensile stress) of the ceramic acts as a force towards contraction (which is in form of a compressive stress) by the bulk (cooler areas). If the tension is sufficient enough and is inhibited to certain level then it will overcome the compression. This in turn will cause the ceramic to fail by cracking and often shattering the ceramic in pieces. For the study herein, it ideal to create shorter numbers of cracks in terms of producing a defect-free surface or a surface with minimal cracking. In other words, it is more desirable to produce a laser-treated surface with as minimal surface cracks as possible. But more importantly, it is more desirable to produce many small cracks in comparison to few larger surface cracks. By comparing the results herein with those of the previous workers it can be said that the

difference is mainly in the terminology of crack definition as the crack-free surface as described in their research is more realistically described in this research as surface comprising of minimal or very few cracks.

Since cracking occurs due to the difference in the thermal gradient then cracking can be avoided by reducing the thermal gradient such as changing the temperature of the ceramic more slowly which means heating the ceramic before the laser surface treatment or possibly post heating (if need be) [3, 6] or by using a dual laser beam technique [7–10]. This would avoid rapid thermal gradient and the clash of hot and cold surfaces occurring at the ceramic surface and within the body. Pre- or post heating can be performed as an additional process and could compliment the laser surface treatment as shown by previous workers. Or the concept of using a combination of dual laser beams to perform the surface treatment can also be adopted to increase the cooling rate by the trailing laser beam. However, from a

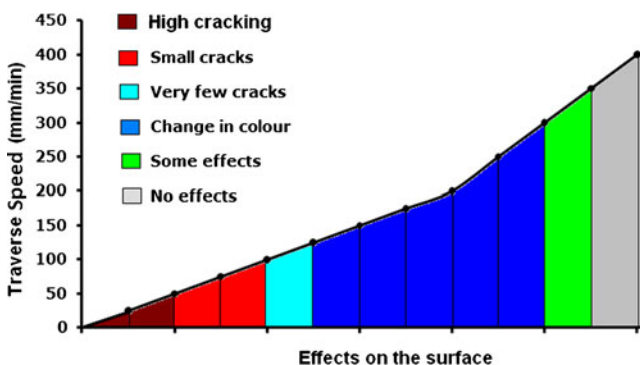


Fig. 8 Effects of the traverse speed on the surface of the Si₃N₄ engineering ceramic after CO₂ laser surface processing

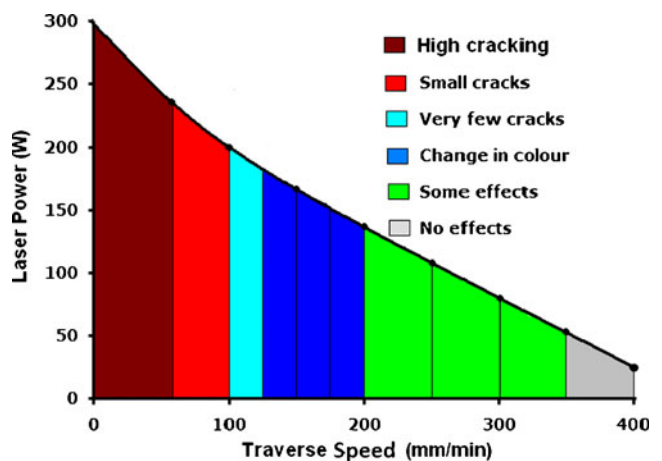


Fig. 9 Relationship between laser power and traverse speed which shows the effects of the CO₂ laser surface treatment of the Si₃N₄ engineering ceramic by keeping a constant spot size of 3 mm and 25 l/min gas flow rate

Table 6 Effects of varying the spot size by using 200 W of constant laser power, traverse speed of 100 mm/min and a gas flow rate of 25 l/min after the CO₂ laser processing of Si₃N₄ engineering ceramic

Experiments	1	2	3	4	5	6	7	8	9
Spot Size (mm)	0.5	1	1.5	2	2.5	3	3.5	4	4.5
Power (W)	200	200	200	200	200	200	200	200	200
Power Density (W/mm ²)	5556	5556	5556	5556	5556	5556	5556	5556	5556
Speed (mm/min)	100	100	100	100	100	100	100	100	100
Effects									

Colour key used to show the effects in Tables 1 to 6 are also shown in Figs. 5 to 9

broader view point, both the dual laser beam processing technique and by using the heating furnace to pre- and post heat the ceramic are additional processes that add extra value to the total cost of processing rather than the product itself. This includes extra process time, tooling and/or man power, more energy required for pre- and post heating technique to take place as well as doubling the cost of laser processing if two lasers are used as leading and trailing heat source. Owing to this, the research herein attempts to process the Si₃N₄ engineering ceramic without using any additional heat source. It is rather economic and less time-consuming to use single laser source as attempts were made to generate the most desirable and ideal surface which comprises of minimal cracking, flaws, porosity, and defects so further analysis can be conducted to elucidate the physical effects of laser–ceramic interaction. Despite the fact that considerable work have been done with processing various ceramics, there is still very little work available with processing engineering ceramics by using the novel fibre laser. The work reviewed in this paper shows various modifications to the material property or surface characteristics. Nonetheless, there is still very little evidence of the first-order effects that occurs during the laser–ceramic interaction

when processing with no additional pre- or post heating or by applying dual beam technique. This is what makes the work herein somewhat unique and novel.

In terms of producing a laser-irradiated surface with minimal or no surface cracks, the most suitable parameters were are shown in Table 7. The fibre laser-treated surface of the Si₃N₄ engineering ceramic was somewhat different to that of the CO₂ laser due to a difference in the wavelength and the beam delivery system. This was because the fibre laser was delivered from a fibre cable and the CO₂ laser being delivered by mirrors and galvanometers. Particularly, the laser beam brightness is more influential rather than the laser power. Consequently, this could have led to both lasers producing different results on the surface of the Si₃N₄ engineering ceramic. From comparing the two lasers, it was found that the Si₃N₄ engineering ceramic was more resistive towards the MIR wavelength of the CO₂ laser as opposed to that of the NIR wavelength of the fibre laser.

The depth of penetration varied for the two lasers on the Si₃N₄ as the CO₂ laser was having a bigger effect on the top surface but was only penetrating through the sub-surface. In comparison the fibre laser had penetrated much deeper but produced a narrow surface track [26, 27]. This was due to

Fig. 10 Effect of the thermal energy on the surface and within the bulk of the ceramic after laser surface processing

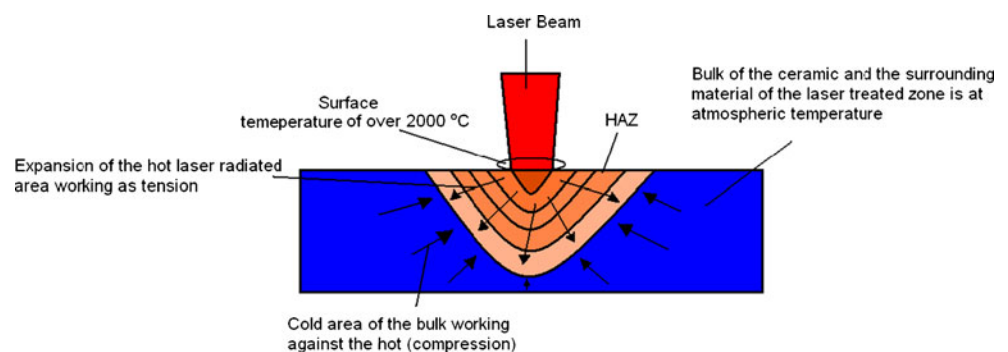


Table 7 Selected parameters from the CO₂ and the fibre laser experimental investigation of the Si₃N₄ engineering ceramic by using a constant spot size of 3 mm, gas flow rate of 25 l/min by employing compressed air assist gas

Lasers types	Parameters		
	Traverse speed (mm/min)	Laser power (W)	Power density (W/mm ²)
CO ₂ laser	100	200	5556
Fibre laser	100	143.5	3979

its higher quality, acceptance of the NIR wavelength and higher brightness value [23, 27]. On the other hand, the CO₂ laser produced a larger surface profile in comparison to that of the fibre laser (see Fig. 11a and b). It can be

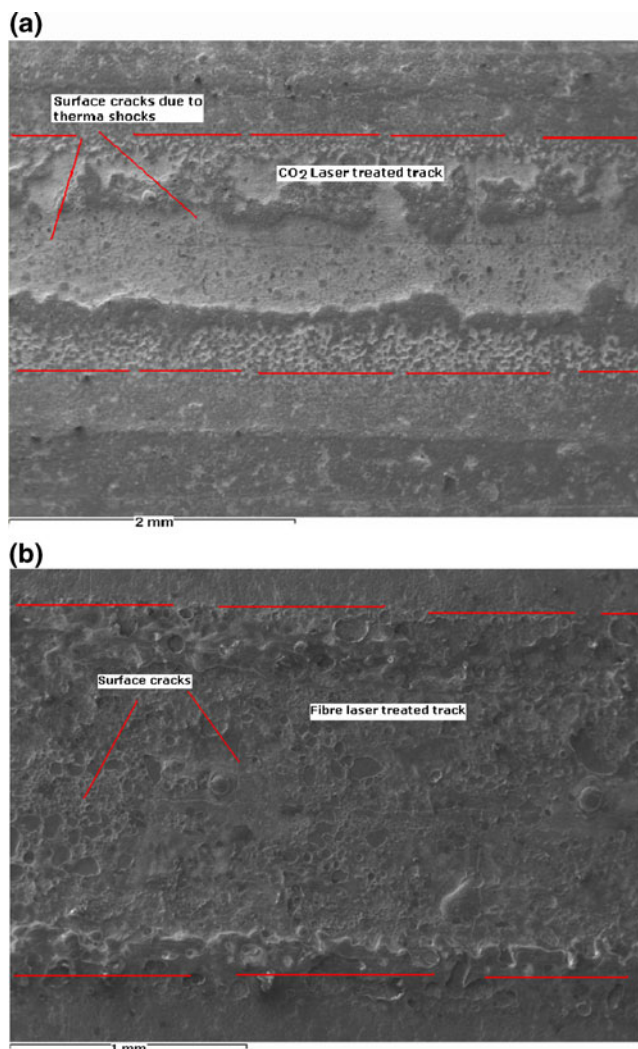


Fig. 11 SEM images of the CO₂ laser-treated surface in (a) and the fibre laser-treated surface in (b), both showing the laser-treated track and the induced surface cracks on the Si₃N₄ engineering ceramic

measured from the SEM images in Fig. 11 that the fibre laser-induced track was about 1.30 mm wide whereas the one for the CO₂ laser was 1.45 mm in width. This meant that the CO₂ laser was producing a larger footprint of the beam whereas the fibre laser was much narrower and produced a finer footprint. At the same time, the depth of penetration of the CO₂ laser was around 100 to 150 μm and the fibre laser was around 150 to 200 μm [27, 28]. This showed that the CO₂ laser was having a bigger interaction zone (on the surface) and the sub-surface but the fibre laser in comparison penetrated deeper which can also be confirmed by previous investigations [27, 28]. On account of this, it is indicative that the NIR wavelength of the fibre laser had better absorption in comparison to the MID wavelength of the CO₂ laser. This had occurred despite using the same spot size for both lasers and the cause of this can be postulated by the high brightness fibre laser also comprising of better beam quality. This in turn led to the fact that a fibre laser with a NIR or a diode laser would be better suited for the surface treatment of the Si₃N₄ engineering ceramic.

4 Conclusions

Experimental investigation was conducted on the Si₃N₄ engineering ceramics by using the CO₂ and the fibre laser. Threshold of the Si₃N₄ was found by using two different wavelengths. The fibre laser surface processing of the Si₃N₄ engineering ceramic was somewhat different to that of the CO₂ laser. This was due to the difference in the Gaussian beam modes (TEM₀₁) and the beam quality factor (M^2) of 1.3 for the CO₂ laser TEM₀₀ and an M^2 value of 1.1 for the fibre laser. Also, the difference in the wavelength between the two lasers was significant as the fibre laser wavelength was 1.075 μm and the CO₂ laser being 10.6 μm, the beam delivery system also being different as the fibre laser was delivered from a fibre cable and the CO₂ laser being directed by mirrors and galvanometers. Consequently, each laser produced surface cracking at different laser parameters during the laser surface treatment of the Si₃N₄ engineering ceramic. Furthermore, the comparison showed that the CO₂ laser wavelength when processing the Si₃N₄ was absorbed less than that of the fibre laser as higher power density and same traverse speed was used to reach the threshold for the Si₃N₄ in comparison to the fibre laser. This was because the fibre laser penetrated deeper, whereas the CO₂ laser produced sufficient interaction at the surface and the sub-surface which in turn produced the sufficient surface cracks. Minimum cracking was observed with using the parameters which is why such parameters were further adopted to conduct experiments for this research. It can be

suggested that the fibre laser surface treatment by using the NIR wavelength would be more suitable when processing the Si_3N_4 engineering ceramic.

References

- Ester FJ, Merino RI, Pastor JY, Martin A, Llorca J (2008) Surface modification of Al_2O_3 - ZrO_2 (Y_2O_3) eutectic oxides by laser melting: processing and wear resistance. *J Am Ceram Soc* 91(11):2552–3559
- Triantafyllidis D, Li L, Stott FH (2002) Surface treatment of alumina-based ceramics using combined laser sources. *Appl Surf Sci* 186:140–144
- Triantafyllidis D, Bernstein JR, Stott FH (2003) Dual laser beam modification of high alumina ceramics. *J Laser Applications* 15 (1):49–54
- Triantafyllidis D, Li L, Stott FH (2004) Surface properties of laser-treated ceramic materials. EMRS Spring Conference. Strasbourg: France. *Thin Solid Films* 453:76–79
- Triantafyllidis D, Li L, Stott FH (2005) The effect of laser-induced modification of surface roughness of Al_2O_3 -based ceramics on fluid contact angle. *Material science and Engineering A-structural Material Properties Microstructure and Processing* 390(1–2):271–277
- Triantafyllidis D, Li L, Stott FH (2006) Crack-free densification of ceramics by laser surface treatment. *Surf Coat Technol* 210:3163–3173
- Murray AJ, Tyrer JR (1998) Nd:YAG Laser cutting and drilling of PSTZ- influence of substrate heating temperature on recast layer microcracking. *Journal of Laser Applications* 11(3):128–135
- Murray AJ, Tyrer JR (1998) Pulsed CO_2 and Nd: YAG laser drilling of PSTZ—a study into the wavelength, effects on recast layer micro-cracking. *Lasers in Engineering* 9:23–37
- Murray AJ, Tyrer JR (1998) Nd: YAG laser drilling of 8.3 mm thick partially stabilized tetragonal zirconia—control of recast layer micro-cracking using localized heating techniques. *Journal of Laser Applications* 11(4):179–184
- Murray AJ (2001) The reduction of micro-structural damage during the drilling of ceramics by high power laser. PhD Thesis: Loughborough University.
- Lawrence J, Li L (2002) On the difference between the beam interactions characteristics of CO_2 , Nd:YAG Excimer and high powered diode lasers with a $\text{SiO}_2/\text{Al}_2\text{O}_3$ ceramic. *Lasers in Engineering* 12(2):81–93
- Lawrence J (2002) An analysis of the bonding mechanism active in a high powered diode laser generated two stage ceramic tile grout. *Lasers in Engineering* 12(4):289–309
- Lawrence J, Hao L (2005) *Laser Surface Treatment of Bio-implant Materials*. Wiley, United Kingdom
- Lawrence J, Li L, Spencer JT (1999) Diode modification of ceramic material surface properties for improved wettability and adhesion. *Appl Surf Sci* 138–139:388–393
- Sun L, Malshe AP, Jiang W, McCluskey PH (2006) Effect of CO_2 laser surface processing on fracture behaviour of silicon nitride ceramic. *J Eng Mater Technol* 128:460–467
- Sun L, Malshe AP, Wen Ping J, McCluskey PH (2006) Experimental investigation of laser surface processing of flexure silicon nitride ceramics. *Transaction of Nonferrous Metals Society of China* 16:558–565
- Morita N, Watanabe T, Yoshida Y (1991) Crack free processing of hot-pressed silicon nitride ceramics using pulsed YAG laser. *JSME International Journal Series III* 34(1):149–153
- Li JF, Li L, Stott FH (2004) A three-dimensional numerical model for a convection–diffusion phase change process during laser melting of ceramic materials. *International Journal of Heat and Mass Transfer* 47:25
- Wang HA, Wang WY, Xie CS, Song WL, Zeng DW (2003) Microstructural characteristics of Al_2O_3 -based refractory containing ZrO_2 induced by CO_2 laser melting. *Appl Surf Sci* 221:291–301
- Wang HA, Wang WY, Xie CS, Song WL, Zeng DW (2003) CO_2 laser-induced structure changes on a zircon refractory. *Appl Surf Sci* 223:104–113
- Wang HA, Wang WY, Xie CS, Song WL, Zeng DW (2004) CO_2 laser-induced structure changes on an alumina–mullite–zirconia refractory. *Appl Surf Sci* 223:244–251
- Wang HA, Wang WY, Xie CS, Song WL, Zeng DW (2005) Nd: YAG laser surface densification of a zircon refractory by adding AlN nano-particles: material characterization. *Appl Surf Sci* 256:227–231
- Shukla PP, Lawrence J, Paul A (2011) Influence of laser beam brightness during laser surface treatment of ZrO_2 engineering ceramics. *Lasers in Engineering*. Accepted on 6th December 2010: (in press)
- Lysenko VS, Nazarov AN, Lokshin MM, Kaschieva SB (1977) Effects of laser irradiation on the structure of silicon dioxide film with implanted phosphorus ions. *Fiz Tekh Poluprovodn* 1 (11):2254–2257
- Shukla PP, Lawrence J (2010) Surface characterization and compositional evaluation of a fibre laser processed silicon nitride (Si_3N_4) engineering ceramic. *Lasers in Engineering* 20(5–6):359–370
- Shukla PP, Lawrence J (2011) Evaluation of fracture toughness of ZrO_2 and Si_3N_4 engineering ceramics following CO_2 and fibre laser surface treatment. *Optics and Lasers in Engineering* 49:229–239. doi:10.1016/j.optlaseng.2010.09.010
- Shukla P (2011) Viability and Characterization of the Laser surface treatment of Engineering Ceramics, A doctoral thesis: Loughborough University
- Shukla PP, Lawrence J (2009) Laser surface treatment of engineering ceramics and the effects thereof on fracture toughness. The 27th International Congress on Applications of Lasers and Electro-Optics (ICALEO 2009): Laser Materials Processing, 2–5 November 2009, Orlando, FL, USA, 102: 109–115