SURFACE MICRO TEXTURING OF SINTERED CERAMIC BY KrF EXCIMER LASER

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Abstract: In the current investigation the surfaces of a sintered Al_2O_3 and TiC ceramic composites are irradiated in air with a KrF Excimer laser. The surfaces are irradiated with pulse energy varying from 300 to 750mJ, pulse frequency varying from 2 to 10Hz, and number of pulses ranging from 300 to 10,000. The surface morphologies, removal depth and roughness of the treated spots are subsequently examined. Very fine agglomerate of ceramic structures, which are near conical in shape, are found to be formed all over treated zone. The roughness and ablation rate considerably increase with number of pulses and beam energy. Set of experiments is carried out using Taguchi methodology to find out optimum conditions for roughness and depth of machining. The analysis gives the optimum condition at 650mJ pulse energy, 3000 number of pulses and 8 repetition rate with expected roughness (R_a) value of 1.97µm and expected removal depth of 132.92µm.

Key words: KrF Excimer laser, Sintered ceramics, Surface micro texturing

1. INTRODUCTION

The prominent characteristics of ceramics include wear resistance, corrosion resistance, temperature resistance, controlled surface behavior, dimensional stability, engineered behavior, heat recovery, and filtration. The monolithic ceramic materials, which are entirely ceramic, typically have low porosity, and comprise of a complete component. Examples of such ceramics include dense forms of aluminum oxide (Al_2O_3) , silicon nitride (Si_3N_4) , silicon carbide (SiC), zirconium oxide (ZrO₂), transformation-toughened zirconium (TTZ), transformation-toughened alumina (TTA), and aluminum nitride (AlN) (David & Douglas 2003). Ceramics has the potential to help the industries to reach their visions of increase machining capabilities, decreased maintenance, optimized recycling, increased efficiency, and decreased pollution.

Conventional methods for surface texturing of ceramic materials are very difficult similar to glass machining. Traditionally, diamond-tool, hydrodynamic (water jet) or ultrasonic machining are used to create complex surface geometries in ceramic materials, but these processes are very time consuming and expensive.

The advantage of laser sources in industries includes fast, flexible and contact free processing. The heat input is localised causing less distortion than other methods. Lasers used for surface texturing and micro structuring is UV Excimer, diode pumped Nd: YAG lasers (DPSS), copper vapour lasers (CVL) and Ti: Sapphire lasers. The wavelengths of Excimer laser are 157nm (F_2), 193nm (ArF), 248nm (KrF), 308nm (XeCl) and 351nm (XeF) depending on gas mixture used. In the Nd:YAG lasers such wavelength as 1064nm, 532nm, 355nm and 266 nm are available. In CVL there are two wavelengths 511nm (green) and 578nm (yellow). The wavelength in the Ti:Sapphire is usually about 800nm. Pulsed Excimer lasers are the most commonly used lasers for micromachining, fine surface texturing and precision machined components in industry. All laser micromachining techniques use the process of laser ablation, where the interaction of the laser energy with the sample leads to material removal. Laser ablation usually relies on the strong absorption of laser photons by the sample material which means that the wavelength of the laser has to be chosen carefully for maximum absorption. The use of ultra fast lasers, however, has circumvented this approach since ablation takes place as a result of multi-photon absorption at high peak intensities, which means that even materials normally transparent to the laser wavelength can be machined (Tonshoff & Gonschior 1993). The pulse length of Excimer laser typically ranges from a few nanoseconds to just over 100 nanoseconds (Yilbas et al., 1990).

Due to the potential of Excimer laser and importance of ceramics in industry, laser surface texturing is selected as an area of research in the current investigation. The surfaces of a sintered Al_2O_3 and TiC ceramic composites are irradiated in air with a KrF Excimer laser. These sintered ceramic composites are used as cutting tool inserts in practice and their 'as received' surface has a high quality finish by diamond grinding. One possible application of such surface texturing is to engineer appropriate roughness at selective places of the inserts so as to make them suitable for adhesive epoxy bonding. This can be an attractive alternative to join the inserts by vacuum brazing.

Laser texturing of any material is a complex process involving many different parameters that need to work in tandem to produce a quality texturing operation. Such parameters include laser beam energy, number of pulses, repetition rate, stand off distance and thermo-physical and opto-physical properties of the workpiece. Excimer laser surface treatment is a newly developed method for surface modification. When UV radiation is directed onto the ceramic surface the material absorbs the UV radiation strongly in the outmost layer, not exceeding a few micrometers deep, causing some components to be ablated and melted, as well as surface contaminants to be removed (Ricciardi et al., 1998). In the present paper interaction between KrF Excimer laser radiation and sintered ceramics is discussed. The laser energy density, pulse repetition rate and number of pulses are used as the input parameters. The effects of pertinent process parameters on achieved texturing depth, surface topology, edge quality and evolved surface microstructure are studied. Overall material removal mechanism in relation to the interacting parameters is keenly analyzed.



Fig. 1. Experimental set up

2. EXPERIMENTAL SET UP

The experimental set up, which is shown in Fig. 1., consists of the following major components.

- Laser source: It is a KrF Excimer laser (Model Compex 201, Make : Lambda Physik, Germany) with characteristics wavelength of 248nm. The laser has a pulse width of 25ns, maximum energy upto 750mJ and maximum repetition rate upto 10Hz. The unfocussed beam is rectangular with cross-section of 30x15mm.
- Beam delivery system: It comprises of beam deflector and telescopic lens holder assembly. The deflector bends the laser beam by 90⁰ onto the work-table. The lens focuses the beam to requisite size
- Work-holding & positioning stage: Worktable with three axis controller is for alignment of work piece and laser beam.
- Masking arrangement: The masking system is required to selectively pass & block the laser beam. The system designed is such that its masking aperture can keep open only required part of ceramic. Special attachment made for mask holding purpose with precise vertical movement so that any fixed distance between workpiece and mask can be maintained throughout the set of experiments.

3. RESULTS

3.1 Experiments for the Effect of the Variation of Number of Pulses

A ceramic composite containing 54 wt% Al_2O_3 and 46 wt% TiC is used in this work. The material is isostatically hot-pressed during manufacturing to ensure low porosity. Before the laser treatment, the samples are carefully polished (Ra=0.068µm) and cleaned with acetone in an ultrasonic bath. The pulse repetition rate is 10Hz, pulse energy is 750mJ and laser fluence is 5J/cm² with number of pulses varied in the range of 400 to 10000.

To analyse the effect of the number of pulses on the output parameters like surface roughness and depth of cut, six different experiments are performed. The number of pulses is varied in increasing order as shown in Table 1. The laser beam profile is shaped using a rectangular mask and focused on the sample surface to produce laser-treated areas of about 0.15 cm². All experiments are carried out in air. Table 1 also shows the output results.

Expt. no	No. of pulses	Depth of spot (µm)	R _a (µm)	Depth /Pulse (µm)
1	400	44.87	0.969	0.1121
2	1000	106.00	1.180	0.1060
3	2000	143.60	1.610	0.0718
4	3500	163.18	1.520	0.0466
5	6000	226.72	2.160	0.0378
6	10000	347.65	2.870	0.0347

 Table 1. Experimental results for variation of the number of pulses

Figure 2 shows the SEM (Scanning Electron Microscope) micrographs of the surface morphologies of the laser-treated Al_2O_3 & TiC composite ceramic surfaces. The morphology mainly consists of a conical structure. The density and the height of the cones are dependent upon the number of pulses it is irradiated with.

To obtain roughness results, the mechanical surface roughness profiles are captured on a PC using Taylor Hobson's Talysurf instrument (Model: Surtronic 3+) for collection of R_a values. The surface roughness profiles are shown in Fig. 3.

3.2 Experiments for Surface Roughness and Depth of Machining Measurement

After the above set of six experiments, an L16 Taguchi orthogonal array (Chen et al., 1996) is followed. Experiments are performed by KrF Excimer Laser on the Al_2O_3 +TiC composite sintered ceramic keeping standoff distance constant at 247mm so that a spot area 0.15cm². is maintained throughout. The data obtained is tabulated as shown in Table 2.



400 Pulses

1000 Pulses

2000 Pulses



3500 Pulses

6000 Pulses

10,000 Pulses

Fig. 2. Surface morphologies of ceramic, laser treated at 750mJ, 10Hz, with a stand-off distance 247mm



6000 Pulses, R_a = 2.16 µm, R_{max} = 14.2 µm

10000 Pulses, R_a = 2.87 μ m, R_{max} = 23.9 μ m

Fig. 3. Surface roughness profiles of experiments shown in Table 1

E-mat in a	Delas	Ma of	Damati	Danth of	р
Expt no.	Puise	INO. 01	Kepeu-	Depth of	\mathbf{K}_{a}
	energy	pulses	tion rate	spot	(µm)
	(mJ)		(Hz)	(µm)	
1	300	400	2	16.3	0.63
2	300	1000	5	51.0	0.86
3	300	1800	8	71.4	1.04
4	300	3000	10	83.6	1.19
5	425	400	5	24.5	0.55
6	425	1000	2	81.6	0.57
7	425	1800	10	102.0	1.12
8	425	3000	8	122.3	1.81
9	550	400	8	30.6	0.72
10	550	1000	10	91.8	0.78
11	550	1800	2	109.3	1.20
12	550	3000	5	124.4	1.16
13	650	400	10	32.6	0.81
14	650	1000	8	97.9	1.27
15	650	1800	5	114.2	1.58
16	650	3000	2	130.5	1.93

Table 2. Experimental layout and output data



(Treated with 425 mJ, 3000 Pulses, 8 Hz) Fig. 4. Measurement of depth of spot by SEM

To study the surface characteristic of the machined surface, SEM micrographs of the surface morphologies of the laser-treated ceramic surfaces are taken. These micrographs are taken (Fig. 4) by tilting the ceramic insert surface by an angle of 40° . The purpose of these micrographs is to measure the depth of spot as obtained by experiments. The surface roughness of all the treated specimens is measured using Taylor Hobson Talysurf (Model: Surtronic 3+).

4. DISCUSSION

4.1 Results of Number of Pulses versus MRR/Pulse

Ablation Rates:

Figure 5 shows the variation of the removal depth per pulse with the number of pulses at constant laser

fluence. For all the values of number of pulses used in the experiments, there is a significant drop in removal rate with increasing the number of pulses. The material removed per pulse decreases from $0.1121\mu m$ to $0.0347\mu m$ by increasing number of pulses from 400 to 10,000 respectively.

Figure 6 shows the variation of the material removal depth with the number of pulses at constant laser fluence. The removal depth at constant laser fluence increases with the number of pulses from $44.87\mu m$ to $347.65\mu m$.

The behavior of the graphs shown in Fig. 5 and Fig. 6 is because of the formation of conical structures on the ceramic surface. Such conical structure formation is due to different ablation rates of the constituents (Al_2O_3 and TiC) of the composite ceramic. As the ceramic surface is irradiated with more number of laser pulses the height difference between the peak and valley becomes greater and eventually conical structures are formed. After exposing the surface with a substantial number of pulses the area is mostly covered by the cones, which are relatively difficult to ablate by excimer laser. Therefore the MRR per pulse goes down rapidly from the initial rate. Once conical structure formation starts it tends to become stable during further irradiation.



Fig. 5. The variation of material removal per pulse with number of pulses at constant laser fluence of 5J/cm²



Fig. 6. The variation of material removal depth with number of pulses at constant laser fluence of 5J/cm²



Fig. 7. The variation of surface roughness with number of pulses at constant fluence of $5J/cm^2$

Roughness:

The comparison of the surface roughness profile of the initial surface and a laser-irradiated surface demonstrates clearly the rapid degradation of surface finish with pulse energy of 750mJ. In fact for all variations, the value of roughness increases with increase in the number of pulses used. The rate of increase of roughness is rapid at the beginning; afterwards the rate of increase of roughness recedes for higher number of pulses as shown in Fig. 7.

Surface Morphology and Microstructure:

The SEM micrographs of Fig. 2 show the surface morphology of areas irradiated with 400 to 10000 pulses at 5J/cm². After irradiation with 400 laser pulses small conical features appear, uniformly dispersed on the flat surface. The area occupied by the cones and their average diameter increase with increasing number of pulses. The differential ablation rate of the components present in the ceramic composites by the initial pulses leads to such conical structure. The regions representing the conical structure have higher titanium, oxygen and carbon contents than the starting material. Conversely, the regions between cones present higher aluminum content. This has been established by EDS analysis by SEM. After around two thousand laser pulses the surface is completely covered by the cones and the mean diameter of these cones does not increase further and can be visualized by SEM micrographs. The growth of Excimer laser modified conical surface is definitely responsible for the variation of the removal depth per pulse observed in Fig. 6.

4.2 Results of Surface Roughness and Depth of Machining Measurement

4.2.1 Surface Roughness Analysis

The standard analysis approach is selected to determine the optimum condition. "The bigger is the

best" quality characteristic is selected. Analysis uses the experimental results shown in Table 2. Figure 8 shows the effect of each factor on surface roughness. Following statements can be made from main effects.

- 1) The surface roughness increases with increase in pulse energy.
- 2) As the number of pulses increases, the roughness value increment is very prominent in comparison with the other factors.
- Effect of repetition rate on average roughness is not so significant.



Fig 8. Effects of factors on surface roughness

Factors		DOF	Sum of	Vari-	F-	Pure	Percent
			Squrs.	ance	Ratio	Sum	
А	Fluence	3	0.564	0.188	4.096	0.426	16.02
В	No.of Pulses	3	1.703	0.567	12.371	1.565	58.83
С	Repetition Rate	3	0.118	0.039	0.862	0.000	0.00
Others/Errors		6	0.274	0.045			25.15
Total		15	2.661				100.00

Table 3. Anova surface roughness analysis

It can be concluded from ANOVA (Table 3) that the effect of number of pulses as compared to the remaining two input factors is more. It is 58.83 %.

Optimum Conditions and Performance:

Factors		Level description	Level	Contribution
А	Pulse enrgy (mJ)	650	4	0.321
В	No. of pulses	3000 4		0.446
С	Repetition rate (Hz)	8 3		0.133
Tota	al contribution f	0.9		
Cur	rent grand avera	1.076		
Exp	ected result at o	1.976		

Table 4. Optimum conditions and performance

At the optimum condition (Table 4) of 650mJ pulse energy, 3000 number of pulses and 8 repetition rate the expected surface roughness (R_a) is 1.976µm. Experiment 16 from Table 2, which has been performed with 650mJ pulse energy, 3000 number of pulses and 2 repetition rate, the R_a value obtained is 1.93µm. As the contribution of repetition rate is insignificant this can be treated as the confirmation experiment for the optimum condition.

4.2.2 Depth of Machining Analysis

The standard analysis approach is again selected to find out the optimum condition. "The bigger is the best" quality characteristic is chosen. Analysis uses the experimental results shown in Table 2.



Fig 9. Effects of factor on depth of machining

Figure 9 shows the contribution of each factor on depth of machining and following inference can be drawn.

- 1) As pulse energy increases the depth of machining increases.
- The effect of number of pulse has come out to be the most significant as compared to the other factors. The depth of machining increases with increase in number of pulses.
- 3) Effect of repetition rate on average depth of spot is not so prominent.

	Factors	DOF	Sum of	Vari-	F-Ratio	Pure	Per-
			Sqrs.	ance		Sum	cent
А	Fluence	3	3499.93	1166.64	21.46	3336.8	15.14
в	No.of Pulses	3	18098.89	6032.96	110.98	17935.8	81.39
С	Repetition Rate	3	112.24	37.41	0.69	0.0	0.00
Others/Errors		6	326.16	54.36			3.47
Total		15	22037.22				100.00

Table 5. Anova for depth of machining analysis

From ANOVA (Table 5) it can be concluded that the effect of the number of pulses as compared to the other two input factors is more. It is 81.388 %.

Optimum Conditions and Performance:

Factors		Level description	Level	Contribution
А	Pulse energy (mJ)	650	4	13.55
В	No.of pulses	3000	4	34.95
С	Repetition rate (Hz)	2	1	4.18
Tot	al contribution fro	52.67		
Cur	rent grand averag	80.25		
Exp	ected result at op	132.90		

Table 4. Optimum conditions and performance

At the optimum condition of 650mJ pulse energy, 3000 number of pulses and 2 repetition rate the expected depth of spot is 132.9 μ m. Experiment 16 from Table 2 which has been conducted with 650mJ pulse energy, 3000 number of pulses and 2 repetition rate, the depth of obtained spot is 130.5 μ m.

5. CONCLUSION

- a) The variation of roughness and material removal rate is due to the formation of a conical surface layer, with higher titanium, oxygen and carbon contents than the starting material. Conversely, the regions between cones have higher aluminum content.
- b) The Excimer laser surface treated Al₂O₃-SiC composite sintered ceramic has conical structures, which can provide the extra surface area for adhesive bonding and mechanical locking.
- c) The ablation rate of Al_2O_3 -TiC ceramic specimens irradiated in air with $5J/cm^2$ KrF Excimer laser depends strongly on the number of laser pulses. The ablation rate per pulse decreases from 0.1121µm to 0.0347µm for numbers of pulses increased from 400 to 10,000 respectively.
- d) The limiting value of roughness (R_a) increases from 0.969µm to 2.87µm when the number of pulses increases from 400 to 10,000.
- e) The material removal depth increases from 45μm to 348μm when the number of pulses increases from 400 to 10,000.
- f) The experimental results are very near to the predicted optimum condition values.
- g) The effect of repetition rate is much less than other two factors. So it can be kept at maximum level considering minimum operating cost.

6. REFERENCES

- Yilbas, B. S.; Davies, R. & Yilbas, Z. (1990). Study into the measurement and prediction of penetration time during the CO₂ laser cutting process. Proc. Inst. Mech. Eng. J. Eng. Manuf. B, Vol. 204, 105–113, ISSN 0954-4054
- Richerson, D. W. & Freitag, D. W. (2003). Ceramics Industry, Available from: http://www.ms.ornl.gov /programs/energyeff/cfcc/iof/chap2.pdf, Accessed : 2006-06-18.
- Ricciardi, G.; Cantello, M.; Mariotti, F.; Castelli, P. & Giacosa, P. (1998). Micromachining with excimer laser. Ann. CIRP, Vol. 47/1, 145-148, ISSN 0007-8506
- Tonshoff, H.K.; & Gonschior, M. (1993) High quality laser cutting of ceramics through adapted process techniques. *Proc. SPIE*, Vol. 2062, 125–135, ISBN: 0-8194-1327-5
- Chen, Y. H.; Tam, S. C.; Chen, W. L. & Zheng, H. Y. (1996). Application of Taguchi method in the optimization of laser micro-engraving of photo masks. Reprinted from *International Journal of Materials & Product Technology*, Vol. 11, Nos. 3/4, 333-344, ISSN 0268-1900