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Laser treatment of alumina surface with chemically distinct carbide particles

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ABSTRACT

Laser treatment of pre-prepared alumina tile surface with a carbon film containing a mixture of 3 wt% TiC and 3 wt% B_4C hard particles was conducted. Morphological and metallurgical changes at the laser treated surface were examined using optical and electron scanning microscopes, energy dispersive spectroscopy, and X-ray diffraction. Microhardness and fracture toughness of the treated surface were measured together with indentation tests. Residual stress generated at the surface region was determined from the X-ray diffraction data. It was found that TiC and B_4C hard particles cause micro-crack formation in the vicinity of hard particles on the surface. This behavior is attributed to the differences between the thermal expansion coefficients of these particles. The laser treated surface is composed of a dense layer with fine sized grains and columnar structures formed below the dense layer. The presence of hard particles enhances the microhardness and lowers the fracture toughness of the surface. The formation of nitride compounds (AlN and AlON) contributes to volume shrinkage in the dense layer. Residual stress formed in the surface region is compressive.

1. Introduction

Alumina tiles are widely used in industry due to their superior properties such as corrosion and wear resistances at high temperatures. Alumina tiles are normally produced from powders by hot sintering; and some micro-voids are usually formed in the tile bulk because of high melting temperature of alumina. Micro-voids cause structural defects and limit the practical applications of sintered tiles in industry. Thermal integration of powders under controlled melting eliminates the structural defect sites and improves the surface properties such as hardness. One of the methods to achieve thermal integration at the surface is to melt the surface using a high energy laser beam. Laser surface treatment has several advantages over the conventional melting techniques, which include short processing time, local treatment, precision of operation, and low cost. Although laser controlled melting at the surface provides thermal integration, it generates high stress levels in the treated region, which in turn modify the fracture toughness of the treated surface. Deposition of hard particles such as TiC and B_4C on the laser treated surface improves surface properties of the treated layer. On the other hand, the mismatch of thermal expansion coefficients of alumina and hard particles contributes to the micro-stress levels in the laser treated region. Consequently, investigation of laser controlled melting of the alumina surface in the presence of hard particles is essential.

A considerable number of research studies have been conducted to examine laser treatment of alumina surfaces. Laser treatment of alumina ceramics in vacuum was studied by Yu et al. [1]. They showed that there were continuous line-like holes on the surface of alumina ceramic due to high rate of heating during laser processing, which in turn modified the flashover voltage characteristics at the surface. Influence of laser treatment on the dielectric strength of α-alumina was investigated by Decup et al. [2]. They demonstrated that that the dielectric strength of α-alumina was not affected by the laser induced surface modifications despite the presence of fine sized cracks on the treated surface. A characterization study of laser surface modified alumina coatings was carried out by Krishnan et al. [3]. They found that laser treatment triggered the transformation of α-alumina layer over the as-sprayed γ-alumina coating and the depth of the laser treated surface increased as a function of laser power. Crystallographic correlation for faceted morphology in a laser surface engineered alumina ceramic was examined by Harimkar et al. [4]. Their findings revealed that faceting of surface grains was closely linked with the evolution of a texture parallel to the surface and normal to the direction of laser treatment trace. Excimer laser surface treatment of plasma sprayed alumina–titania coatings was investigated by Ibrahim et al. [5]. They showed that the laser treatment played a major role in modifying the surface morphology of the coating, which depends on the pulse repetition rate.

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Surface modification of ceramics by laser treatment by Cappelli et al. [6] showed that laser treatment modified the surface roughness and altered the microstructure at the surface significantly. The effects of laser treatment on thermally sprayed alumina coatings were investigated by Costil et al. [7]. They demonstrated that a combination of chemical and mechanical interactions induced by a cone-like structure occurred when the surface was treated by a laser beam prior to thermal-spraying. Multilevel residual stress evaluation in a laser surface modified alumina ceramics was conducted by Samant and Dahotre [8]. They found that under the selected set of laser processing parameters, the ceramic was devoid of any major cracks, making it ideal for several applications. Laser surface modification of aluminum surfaces was investigated by Barnier et al. [9]. They found that laser treatment increased the aluminum oxide layer thickness at the surface. Surface analysis of nanocomposite ceramic coatings including alumina was carried out by Portinha et al. [10]. They demonstrated that after thermal cycling in air, the coatings had a few structure densifications and the roughness increased slightly. Growth behavior of copper/aluminum intermetallic compounds in hot-dip aluminized copper was investigated by Yu et al. [11]. They showed that the interfacial bonding was in a good state and plating an auxiliary potassium fluoride aqueous solution could significantly improve the substrate wettability. Effect of laser treatment on the initial oxidation behavior of Al-coated NiCrAlY bond-coat was examined by Zhu et al. [12]. The formation of θ-alumina, chromium oxide, and nickel–chromium spinel oxides was observed during initial oxidation on the surface of laser pre-oxidized bond-coat.

Laser treatment of alumina surfaces had been investigated previously [13–15]; however, the main focus was either forming nitriding/carbonitriding compounds at the surface [13] or injecting only one type of hard particles during the laser treatment process [14,15]. However, laser induced injection of multi-component hard particles and their effects on the surface characteristics, including hardness, residual stress, and fracture toughness, were left for the future study. Therefore, in the present study, laser surface treatment of pre-prepared alumina surface with two different types of particles is conducted and the resulting surface characteristics, including microhardness, fracture toughness, residual stress levels, morphology, and metallurgical changes, are examined using standard analytical tools. In the surface pre-preparation cycle, a carbon film, containing a uniformly distributed mixture of 3% TiC and 3% B_4C particles, is formed at the alumina tile surface through a multi-step treatment process.

2. Experimental

A CO_2 laser (LC-ALPHAIII) delivering a nominal output power of 2 kW was used to irradiate the workpiece surface. The delivery of the laser output power was in the form of repetition pulse and the maximum frequency of the laser pulse repetition was 1500 Hz. The proper combination of the laser beam scanning speed and the pulse repetition rate provides controlled melting of the surface. A carbon film, uniformly hosting the hard particles, was formed prior to the laser scanning. This enhanced the absorption of the incident beam at the workpiece surface. It should be noted that high reflection of the incident beam from the untreated surface resulted in poor melting characteristics in the irradiated region. This was because of the wavelength of the laser beam, which was 10.6 μm. In addition, the operational cost of the CO_2 laser was lower than those of the other high power laser sources. Therefore, CO_2 laser was selected to irradiate the pre-prepared workpiece surfaces. The nominal focal length of the focusing lens was 127 mm. The laser beam diameter focused at the workpiece surface was ~0.25 mm. Nitrogen assisting gas emerging from the conical nozzle co-axially with the laser beam was used. Laser treatments were repeated several times by using different laser parameters. During the treatment process, laser parameters resulting in the minimum surface defects, such as very small cavities with no cracks or crack networks, were selected. Laser treatment conditions are given in Table 1.

Table 1
Laser heating conditions used in the experiment.

Scanning speed (cm/s)	Peak power (W)	Frequency (Hz)	Nozzle gap (mm)	Nozzle diameter (mm)	Focus setting (mm)	N_2 pressure (kPa)
10	2000	1500	1.5	1.5	127	600

Alumina (Al_2O_3) tiles (Ceram Tec-ETEC, 2010) of 3 mm thickness were used as workpieces. Powders with about 350 nm particle size were mixed in the ratio of 3 wt% of TiC and 3 wt% of B_4C. Homogeneous mixing was ensured prior to the mixing with dissolved phenolic resin. The phenolic resin was dissolved into water prior to mixing with the hard particles and the hard particles were mixed with the water dissolved phenolic resin. Later, the mixture was applied uniformly on the workpiece surface. In order to form a 40 μm thick carbon film at the alumina tile surface, workpieces with the phenolic resin hosting the mixed hard particles at the surface were placed in a control chamber, which was set at a pressure of 8 bar and 175 °C, for 2 h. The use of high pressure ensured evaporation of water and drying of the phenolic–particle mixture at the workpiece surface. The workpieces were, then, heated to 400 °C in an argon environment for 6 h to ensure the conversion of the phenolic resin into carbon. The pre-prepared sample surfaces were scanned by a laser beam with the parameters given in Table 1.

Material characterization of the laser treated surfaces was conducted using an optical microscope, SEM, EDS, and XRD. In this case, a Jeol 6460 Scanning Electron microscope was used for SEM examinations. Copper tapes were used to ensure contact between the workpiece surface and the sample holder during the SEM micro-graphing. A Bruker D8 Advanced XRD equipment having CuKα radiation was employed for diffraction analysis. Typical settings of XRD were 40 kV and 30 mA and scanning angle (2θ) ranged from 20° to 80°. Surface roughness measurement of the laser-melted surfaces was performed using an Agilent 5100 AFM/SPM Microscope in contact mode. The tip was made of silicon nitride probes (r=20–60 nm) with a manufacturer specified force constant, k, of 0.12 N/m.

A microphotonics digital microhardness tester (MP-100TC) was used to obtain microhardness at the surface. The standard test method for Vickers indentation hardness of advanced ceramics (ASTM C1327-99) was adopted. Microhardness was measured at the workpiece surface after the laser treatment process. The measurements were repeated five times at each location for the consistency of results.

The XRD technique was used to obtain residual stresses in the surface region of the laser treated layer. The XRD technique provides data in the surface region of the specimens due to the low penetration depth of Cu-Kα radiation into the treated layer, i.e. the penetration depth is in the order of 5 μm. Measurements relied on the stresses in fine grained polycrystalline structure and the position of the diffraction peaks exhibited a shift as the specimen was rotated by an angle ψ. The magnitude of the shift can be related to the magnitude of the residual stress. The relationship between the peak shift and the residual stress (σ) is given as [16]

$$\sigma = \frac{E}{(1+\upsilon)\sin^2\psi}\frac{(d_n - d_0)}{d_0} \qquad (1)$$