



Laser gas assisted texturing and formation of nitride and oxynitride compounds on alumina surface: Surface response to environmental dust



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ABSTRACT

Laser gas assisted texturing of alumina surface is carried out, and formation of nitride and oxynitride compounds in the surface vicinity is examined. The laser parameters are selected to create the surface topology consisting of micro/nano pillars with minimum defect sites including micro-cracks, voids and large size cavities. Morphological and hydrophobic characteristics of the textured surface are examined using the analytical tools. The characteristics of the environmental dust and its influence on the laser textured surface are studied while mimicking the local humid air ambient. Adhesion of the dry mud on the laser textured surface is assessed through the measurement of the tangential force, which is required to remove the dry mud from the surface. It is found that laser texturing gives rise to micro/nano pillars topology and the formation of AlN and AlON compounds in the surface vicinity. This, in turn, lowers the free energy of the textured surface and enhances the hydrophobicity of the surface. The liquid solution resulted from the dissolution of alkaline and alkaline earth metals of the dust particles in water condensate forms locally scattered liquid islands at the interface of mud and textured surface. The dried liquid solution at the interface increases the dry mud adhesion on the textured surface. Some dry mud residues remain on the textured surface after the dry mud is removed by a pressurized desalinated water jet.

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1. Introduction

Aluminum oxynitride (AlON) is one of the ceramics that has high hardness and superior optical characteristics such as high optical transmittance in the near-ultraviolet, visible, and mid-wave-infrared radiations [1]. It finds applications in various industries, particularly for those involved with blast-resistant transparent windows and infrared optics. Aluminum oxynitride has a cubic spinel structure, and it can be fabricated by the conventional methods incorporating ceramic powder processing technologies [2]. However, producing a thin layer of aluminum oxynitride coating on the ceramic surfaces via the method of conventional powder processing is challenging and may involve a high cost. One of the methods to produce aluminum oxynitride film on the ceramic surfaces is to incorporate laser gas assisted surface processing [3]. Laser surface treatment of alumina tiles with the presence of assisting gas, composing of a mixture of nitrogen and oxygen, in the treated region can produce a thin layer of aluminum nitride and aluminum oxynitride film at the surface while improving the tribological properties of the treated surface significantly [3]. Since the laser controlled melting/ablation is involved with high temperature processing, care must be taken to reduce the surface defects such as micro-cracks, deep cavities, and voids

[4]. Forming a thin film of coating on ceramic surface prior to laser treatment improves surface properties in terms of microhardness and fracture toughness significantly [5]. In addition, the selection of laser processing parameters becomes critical for securing the desired surface topology and microstructure [6]. Laser texturing can also be used to improve surface hydrophobic characteristic, which in turn lowers the friction coefficient and minimizes the adhesion of the particles on the surface [7]. Consequently, investigation of laser oxynitriding of alumina surface towards improving the surface hardness and tribological properties becomes essential.

Considerable research studies were carried out to examine laser treatment of alumina surfaces. A study on aluminum oxynitride thin films grown by ion-beam-assisted pulsed laser deposition was carried out by Zabinski et al. [8]. They showed that the films were nearly stoichiometric except for depositions in a vacuum and the nitrogen concentration could be controlled through selection of gas pressure and ion energy. In addition, the crystalline Al-O-N films showed improved hardness in comparison to amorphous films. Laser texturing of alumina surface for improved hydrophobicity was studied by Yilbas et al. [9]. The findings revealed that laser controlled ablation resulted in micro/nano texturing of the surface. Although the surface texture did not exactly follow the regular pattern, pillars and dimples like structures were formed,

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and the averaged surface roughness was within the sub-micro scale. In general, laser texturing improved the surface hydrophobicity; however, Wenzel and Cassie and Baxter states were present at the treated surface due to the variation in the surface texture. Tribology and superhydrophobicity of the laser-controlled-melted alumina surfaces with the presence of hard particles at the surface were examined by Yilbas et al. [10]. They demonstrated that laser treatment produced micropoles, nanopoles, and small size cavities at the surface, which enhanced surface hydrophobicity. The microhardness of the laser-treated surface increased almost 50% because of the dense layer formed on the surface, and the residual stress was in the order of 2 GPa, which was compressive. The scratch resistance and friction coefficient of the laser-treated surface were superior. Laser micro machining of epoxy/alumina nanoparticle composite generating the micro-patterns was investigated by Psarski et al. [11]. They showed that the textured surface had hierarchical topography, which consisted of hexagonally spaced microcavities and nanoparticle agglomerates. Laser short-pulse fabrication of a superhydrophobic Al_2O_3 surface was studied by Jagdheesh [12]. The findings revealed that the geometry of the laser-machined pattern played a major role in changing the wetting properties rather than the chemical changes induced on the surface. The micropillars exhibited a consistent superhydrophobic surface with a static contact angle in the order of $150^\circ \pm 3^\circ$.

On the other hand, recent changes in climate have the detrimental effects on environment plants, urban life, and industries. Depending on the geographic region, climate change gives rise to frequent dust storms and causes dust settlements on outdoor surfaces, which have adverse effects on the tribological properties. To restore the surfaces to the original state, the removal of the dust particles from the surfaces requires extra efforts, in terms of energy and cost. In some cases, adhesion of the dust particles at the surface causes permanent damages due to the chemical effects [13]. This occurs in the humid air ambient; in which case, water condensate on the dust particles, which results in dissolution of some alkaline and alkaline earth metal compounds in the particles while forming a chemically active solution. The mud solution flows towards the solid surface under the gravity and forms the liquid layer between the surface and the dust particles. Upon drying of the liquid solution, a solid layer is formed at the interface between the dust particles and the surface while enhancing the adhesion of the dust particles on the surface. This requires further efforts to remove the dust particles from the surface. Considerable research studies were carried out to examine the settlement of the dust particles and their effects on the surface characteristics. The environmental dust adhesion on the textured alumina surface was investigated by Yilbas et al. [14]. They demonstrated that the mud solution modified the texture characteristics of the laser treated surface once it dried; in which case, the surface hydrophobicity reduced significantly. The mud adhesion on the laser textured silicon surface was examined by Yilbas et al. [15]. The findings revealed that formation of nitride species contributed to microhardness increase and enhancement of surface hydrophobicity due to achieving low surface energy via nitride species. The mud residues did not influence the fracture toughness and microhardness of the textured surface; however, the mud residues reduced the surface hydrophobicity significantly. A study on laser texturing and sol-gel coating of alumina surfaces were carried out by Yilbas et al. [16]. The findings revealed that the laser treated and sol-gel coated alumina surfaces provided superior surface characteristics in the harsh environments because of weak adhesion between the mud formed from the dust particles and the coating surface. It was associated with the small texture height of the sol-gel coating, which lowered the area of the interfacial contact between the mud and the coated surface, and relatively lower surface energy of the sol-gel coating as compared to that of the laser treated surface.

Although dust accumulation and its effect on the laser textured surfaces was investigated previously [9,10,14,15], the influence of surface chemistry of laser textured surfaces is left for the future study. In addition, laser gas assisted processing modifies the chemical composition of the laser textured surface via formation of nitride and oxide com-

pounds. When nitrogen assisting gas with high pressure is used, nitride compounds are formed in the vicinity of the laser treated layer [3]. However, the multi-compounded chemical structures can be formed, such as nitrides, oxides, and oxynitrides when a mixture of assisting gas at high pressure is used, such as a mixture of oxygen and nitrogen. It alters the hydrophobic state of the laser textured surface and influences the adhesion of the dust particles and dry mud on the surface. Consequently, the present study examines the dust characteristics and their effects of on the laser textured alumina surfaces. High power laser with repetitive pulse mode is used to irradiate the surface towards achieving micro/nano size pillars at the surface. The mixture of nitrogen and oxygen at high pressure is used as an assisting gas during the laser texturing process. The characteristics of the laser textured surface are examined using the analytical tools. The surface characteristics examined include microhardness, surface energy, topology, elemental compositions and compound formation, and hydrophobicity. Analytical tools are used during the characterization study, which comprises of optical, electron scanning, and atomic force microscopes, X-ray diffraction, energy dispersive spectroscopy, and micro-tribometer.

2. Experimental

Alumina (Al_2O_3) tiles (Ceram Tec-ETEC, 2010) with 4 mm thickness were used as workpieces. The CO_2 laser (LC-ALPHAIID) was used to irradiate the alumina tile surfaces. The nominal output power of the laser was 2 kW and the irradiated spot diameter at the workpiece surface was about 200 μm . The mixture of nitrogen and oxygen at high pressure (600 kPa) was used as an assisting gas during the texturing. The partial pressure of nitrogen was $\frac{P_{N_2}}{P_T} = \frac{2}{3}$, where P_{N_2} is the nitrogen pressure in the mixture and P_T is the total pressure of mixture and the partial pressure of oxygen was $\frac{P_{O_2}}{P_T} = \frac{1}{3}$, where P_{O_2} is the oxygen pressure in the mixture. The laser pulsing frequency was set at 1500 Hz, which in turn gave rise to 70% overlapping ratio for the irradiated spots at the surface. The initial tests were conducted to select the laser texturing parameters in such a way that the laser parameters resulting in surface defects, such as micro-cracks, voids, and large size cavities, were avoided. The results of the initial tests revealed that reducing laser power by 10% while keeping laser scanning speed same gave rise to small size texture height without micro/nano pillars at the surface. On the other hand, reducing laser scanning speed by 10% while keeping the laser output power same resulted in cracks and cavities at the surface. Consequently, through controlling the laser power settings, beam intensity distribution, pulse repetition rate, spot size, and the scanning speed, crack free surface texture could be realized. Laser surface texturing conditions are given in Table 1.

Material characterization of the laser textured surfaces was carried out using Jeol 6460 electron microscope and atomic-force/scanning-force microscopes (AFM/SPM), by Agilent, in contact mode. The atomic force microscope (AFM) tip was made of silicon nitride probes ($r = 20 - 60 \text{ nm}$) with a manufacturer specified force constant, k , of 0.12 N/m. Bruker D8 Advanced having $\text{CuK}\alpha$ radiation was used for XRD analysis. A typical setting of XRD was 40 kV and 30 mA and scanning angle (2θ) was ranged $20^\circ - 80^\circ$.

A linear micro-tribometer (MCTX-S/N: 01–0,4300) was used to determine the tangential force and the friction coefficient of the dry mud formed laser textured surface. The microhardness of the surfaces prior to laser treatment, after laser treatment, and after dry mud removal was measured. The equipment was set at the contact load of 0.03 N and end load of 5 N. The scanning speed was 5 mm/min and loading rate was 1 N/s.

The wetting experiment was performed using Kyowa (model - DM 501) contact angle goniometer. A static sessile drop method was considered for the contact angle measurement. Droplet volume was controlled with an automatic dispensing system having a volume step resolution of 0.1 μl . Still images were captured, and contact angle measurements

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