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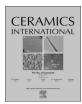
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## Effect of graphene film on laser textured alumina surface characteristics

B.S. Yilbas<sup>a,b,\*</sup>, A. Ibrahim<sup>b</sup>, H. Ali<sup>b</sup>, M. Khaled<sup>c</sup>, T. Laoui<sup>b</sup>

- <sup>a</sup> Center of Excellence in Renewable Energy, King Fahd University of Petroleum & Minerals, Dhahran, Saudi Arabia
- <sup>b</sup> ME Department, King Fahd University of Petroleum & Minerals, Dhahran, Saudi Arabia
- <sup>c</sup> CHEM Department, King Fahd University of Petroleum & Minerals, Dhahran, Saudi Arabia

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#### ABSTRACT

Laser gas assisted texturing of alumina surface is considered and the effects of graphene film on the properties of the textured surface are examined. Since laser texturing under the high pressure nitrogen gas jet environments results in formation of aluminum nitride compounds, free energy of the textured surface reduces considerably. The mismatch between the surface free energies of the graphene film and the laser textured surface makes it difficult to transfer the graphene film on the textured surface without rippling and edge defects. A graphene oxide film is formed at the textured surface prior to transferring of the graphene film. The characteristics of the laser textured and the graphene transferred surfaces are assessed using the analytical tools including electron and atomic force microscopes, Raman spectroscopy, X-ray diffraction, and UV visible absorbance spectroscopy. Surface hydrophobicity of the graphene transferred and laser textured surfaces is determined incorporating the water droplet contact angle measurement technique. Friction coefficient of the graphene transferred and laser textured surfaces are measured using the scratch tester. It is found that laser texturing results in hydrophobic characteristics because of the micro/nano size pillars formed at the surface and reduced surface energy due to aluminum nitride compounds. Transferring of the graphene film on to the laser textured surface reduces both the water droplet contact angle and the contact angle hysteresis. The presence of the graphene film reduces the friction coefficient and it does not alter notably the absorption characteristics of the laser textured surface.

#### 1. Introduction

Graphene has two-dimensional atomic structure with excellent properties such as high mechanical strength, superior electrical and thermal conductivities, and almost nearly optical transmittance. Graphene finds wide applications in electronic circuits [1], medicine [2], photovoltaics [3], and engineering fields [4]. Graphene film can improve surface characteristics, such as friction coefficient and thermal conductivity, of ceramics. On the other hand, the hydrophobic characteristics of the ceramics, such as alumina, surface can be improved through laser gas assisted texturing [5]; however, the textured surface has high friction coefficient and some surface defects such as micro-size cracks, which limit the practical applications of the treated surface in bearing applications. Although laser texturing of surfaces has several advantages over the conventional methods, such as local treatment, fast processing, precision of operation and low cost, high thermal stresses are developed in the surface region while causing formation of the micro/nano size cracks. The presence of micro-cracks has adverse effects on mechanical and optical properties because of the possible

delamination of the textured surface through the micro/nano cracks networks. However, graphene coating at the surface covers the crack sites and minimizes the mechanical defects at the surface. Consequently, investigation of the effects of graphene layer, which is formed on the top of the laser textured alumina surface, on the properties of the laser textured surfaces becomes essential.

Considerable research studies were carried out on laser texturing of ceramic surfaces. Laser-induced deposition of alumina ceramic coating on stainless steel was investigated by Adraider et al. [6]. They showed that laser irradiation of dry alumina films led to the deposition of crystalline alumina coating in  $\alpha\text{-Al}_2O_3$  form on substrate surface. In addition, the mechanical properties of the alumina-coated surface were significantly improved and reached the same level as pure  $\alpha\text{-alumina}$  ceramic. A pulsed laser deposition of alumina thin films was carried out by Boidin et al. [7]. They demonstrated that the use of argon gas during the deposition process resulted in grainy structure of the thin films and decrease of the refractive index of the alumina layers was observed within the 300–7500 nm spectral range when increasing argon pressure. Laser gas assisted nitriding and sol–gel coating of alumina

E-mail address: bsyilbas@kfupm.edu.sa (B.S. Yilbas).

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<sup>\*</sup> Corresponding author at: Mechanical Engineering Department and Center of Excellence in Renewable Energy, King Fahd University of Petroleum & Minerals, Dhahran, Saudi Arabia.

surfaces and the response of the surface in harsh environments was investigated by Yilbas et al. [8]. The findings revealed that the laser treated and sol-gel coated alumina surfaces provided superior surface characteristics in the harsh environments because of the weak adhesion between the mud formed from the dust particles and the coating surface. The sol-gel coating did not alter the optical characteristics of the laser treated surface. The formation of dense  $\alpha$ -alumina layer on Ti-6Al-4V alloy was examined by Khanna et al. [9]. They indicated that the formation of a thin layer of dense alumina onto high toughness Ti-6Al-4V alloy increased the wear resistance of the surface. Laser treatment of alumina surface with presence of chemically distinct carbide particles on the surface was studied by Yilbas and Ali [10]. They showed that the presence of TiC and B<sub>4</sub>C hard particles caused formation of surface micro-crack in the vicinity of hard particles. This behavior was attributed to the differences between the thermal expansion coefficients of these particles. In addition, the laser treated surface was composed of a dense layer with fine sized grains and columnar structures formed below the dense layer. The formation of nitride compounds (AlN and AlON) contributed to volume shrinkage in the dense layer. A study on the laser surface modification of alumina was carried out by Moncayo et al. [11]. They indicated that, by controlling the laser energy density, more multi-faceted grains were formed with less porosity. Laser control melting of alumina surfaces with presence of B<sub>4</sub>C particles was investigated by Yilbas et al. [12]. The findings revealed that microhardness of the surface increased significantly after the laser treatment process, which was attributed to the high cooling rates and the formation of nitride species at the surface, and the residual stress formed at the surface was compressive. A high contrast laser marking of alumina was carried out by Penide et al. [13]. They indicated that the marking atmosphere was the key parameter, being the inert one the best choice to produce the darkest marks. A combined cold isostatic pressing of alumina and selective laser processing of parts was studied by Wang et al. [14]. They introduced the relative density distribution and its standard deviation to evaluate the homogeneity of the parts resulted. The nanotribological properties and oxidation resistance of laser deposited yttria stabilized zirconia and alumina thin films were examined by Nath et al. [15]. They demonstrated that trobological and mechanical properties of the surface were improvement significantly with increasing deposition temperature during the pulsed laser deposition process. Laser control melting of alumina surfaces and thermal stress analysis was studied by Yilbas et al. [16]. They demonstrated that high heating and cooling rates resulted in high von Mises stress levels in the surface region and the residual stress predicted agreed with the measurement results. The structural characteristics and mechanical properties of crystalline alumina coatings produced via sol-gel technology and fibre laser processing were examined by Adraider et al. [17]. They showed that the alumina xerogel films coated on the substrates were successfully converted into crystalline alumina ceramic coatings by the laser irradiation. Increase of laser specific energy led to the formation of initially  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> with increasing amounts of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>. In addition, the laser processing resulted in significant improvement in hardness and Young's modulus of the alumina-coated surface. The characterization study for microstructure in laser surface modified alumina ceramic was carried out by Harimkar and Dahotre [18]. They demonstrated that the fractal dimension of the surface microstructures could be effectively correlated with the surface features of laser surface modified alumina. Laser texturing of alumina surface for improved hydrophobicity was investigated by Yilbas et al. [19]. They indicated that the textured surface consisted of pillars and dimples like structures and the surface roughness is within the sub-micro scale. The formation of AlN compound contributed to hydrophobicity enhancement at the surface. In addition, Wenzel and Cassie and Baxter states were present at the treated surface due to the variation in the surface texture. Tribological behavior of hydrogenated diamond-like carbon on polished alumina substrate was studied by Hee et al. [20]. They showed that hydrogenated amorphous carbon had a hydrophilic characteristic for protein absorption, with low water contact angles of 69.5°. The wettability of graphene-based surfaces deposited on the silicon carbide surface was studied by Souza et al. [21]. They indicated that the water contact angle was dependent on the number of graphene monolayers on silicon carbide surface.

Graphene coating improves the surface characteristics such as corrosion resistance [22] and optical properties [23]. Electrochemical study of corrosion behavior of graphene coatings on copper and aluminum in a chloride solution was studied by Miškovic-Stankovic et al. [22]. They demonstrated that graphene coated copper using the chemical vapor deposition technique resulted in corrosion-inhibiting properties and graphene coated aluminum surfaces demonstrated improved corrosion resistance. A thermal spraying of graphene oxide as hole transport layer in polymer solar cells was carried out by Jeon et al. [23]. They indicated that high performance of polymer solar cells was fabricated via graphene oxide spraying at high temperatures. This in turn improved electrical property of graphene oxide hole transport layer, which resulted from moderate in situ reduction of graphene oxide during spray process on the hot substrate. The behavior of water droplets on curved graphitic-like surfaces was investigated by Alarcon et al. [24]. They showed that in the case of the convex graphitic-like surfaces, the curvature did not affect the local hydrophobicity. Investigation of increasing hydrophobicity of poly(propylene) fibers via coating of reduced graphene oxide was carried out by Ramasundaram et al. [25]. The findings revealed that the water contact angle increased from 108° to 125° after the first reduced graphene oxide coating, and it was saturated at about 135°. In addition, using kaolin as model hydrophilic particles, the depth filters with reduced graphene oxide coated via poly(propylene) fibers showed a superior performance in terms of water flux and trans-filter pressure in comparison with those with the pristine and hydrophilic poly(propylene) fibers prepared by coating functionalized graphene oxide. Although laser surface texturing of alumina surfaces was carried out previously [5,26], graphene coating on the texture surface was left for the future study. In the present study laser gas assisted texturing of alumina surface is carried out and the transfer of graphene film onto the textured surface is investigated. Since surface energy of the laser textured alumina becomes low because of the formation AlN compounds [26], the graphene oxide layer is initially deposited as a buffer layer, on the laser textured alumina surface and, then, the graphene film is transferred on to the surface of the graphene oxide layer at the textured surface. Morphological and structural changes in the laser treated surface are examined using electron scanning and atomic force microscopes, X-ray diffractogram and Raman spectroscopy. The hydrophobicity of the resulting surfaces is assessed incorporating the sessile water droplet contact angle method. The scratch resistance of the graphene transferred and the laser textured surfaces is measured using the micro/nano tribometer.

#### 2. Experimental

The  $\rm CO_2$  laser (LC-ALPHAIII) delivering nominal output power of 2 kW was used and the laser beam was focused to 200 µm spot at irradiated surface. High pressure nitrogen assisting gas jet co-axially with the laser beam was used during the laser treatment process. The repetition rate the laser pulses was set at 1500 Hz, which resulted in 75% overlapping ratio of the irradiated spots at the workpiece surface. The initial laser texturing tests were conducted and laser parameters resulting in micro/nano pillars at the surface were selected. The laser parameters selected gave rise to the minimum surface defects sites including large size cavities and crack-networks. It was observed from the initial tests that increasing laser power at the workpiece surface by 10% and keeping the laser scanning speed same as selected resulted in large cavity formation at the surface. However, when laser scanning speed was reduced by 10% and keeping the laser output power same as

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