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Surface analysis of alumina ceramic exposed to shock waves produced by plasma expander

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Abstract. Material surface treatment by plasma expander is relatively recent. Plasma expander is based on the inverse pinch effect. The shock waves produced by plasma expander may also promote modifications in ceramic materials exposed to the expander. These modifications are mainly made by ablation phenomenon. This work was intended to verify the shock wave effects on the ionic ceramic samples with high dielectric constant. The alumina ceramic samples were formed by both uniaxial and isostatic pressing methods and sintered at 1650 ºC. They were also produced with addition 0.15 wt% of MgO in order to obtain a high densification. The ceramic samples were divided in groups and exposed to 700, 1000 and 1440 pulses during 20 min. These pulses were generated by nitrogen plasma expander at 13.0 Pa and 6 kV. After plasma exposure, there was an increase in roughness parameter values of Al_2O_3 ceramic surface. The treatment by plasma expander did not modify the hydrophilic characteristic of the alumina ceramic samples. The results of hardness test presented no significant changes on hardness mean values.

1. Introduction

This paper aimed to analyze the changes produced by shock waves with nitrogen ions generated by a plasma expander on dense alumina ceramics. The alumina ceramic (Al_2O_3) is an ionic material with a hexagonal crystalline structure, which has high mechanical strength and presents high corrosion and wear resistance. One of the features of high hardness and high elastic modulus of alumina ceramics is due to the ionic bond stability. These ceramics are used in several industry fields. New applications for these materials have emerged in biomaterial industry due to ceramic surface changes such as wettability and morphology [1-5]. Such changes can occur when these materials are exposed to plasma like pinch discharge [6]. Plasma expander is an innovative method for material treatment, based on inverse pinch effect [7-11]. Plasma expander has been used to treat polymer surfaces deposited by plasma [8-10] and in silicon carbide (covalent ceramic) [11].

2. Materials and methods

2.1. Ceramic

The alumina (CT-3000 SG), 0.15 wt% of MgO and 0.12 wt% of CMC were used to produce the ceramics. They were formed by uniaxial pressing (50 MPa) in a stainless steel mold (15 x 15 x 4 mm), followed by isostatic pressing (300 MPa) and sintered at 1650 ºC, with a heating rate of 5 ºC/min. The samples were polished by using diamond paste (granulometry 6 µm, 3 µm and 1 µm).

2.2. Plasma expander

The reactor (Figure 1a and 1b) consists of a glass tube (diameter: 28 cm) covered with two circular brass plates (diameter: 30 cm). In the center, there is a central hole with a conductor bar (diameter: 0.6 cm) isolated by the glass tube, which crosses the bottom plate. The expander has a capacitor (8.5 µF, 20 kV) which is charged at 20 kV by a 20 mA dc power supply. The expansion velocity of the shock wave is measured with a collimated optical fiber coupled to a spectrometer. In the visible spectrum there is a maximum value (approximately 1.3 x 10^6 cm/s) which decreases to a minimum (0.9 x 10^6 cm/s) at the edge of the conductive plates.

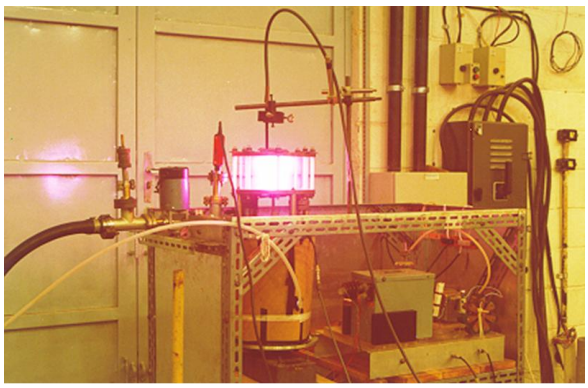

Figure 1a. Plasma expander system.

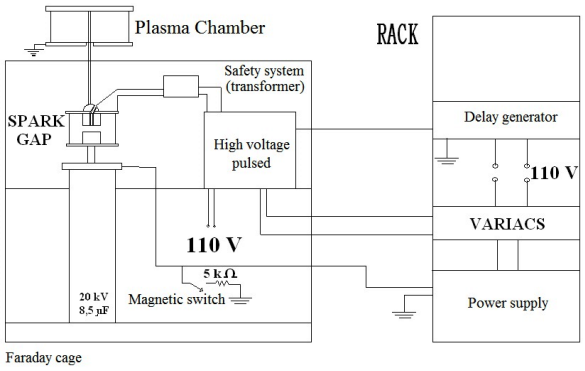

Figure 1b. Plasma expander diagram.

The ceramics were divided in three groups and exposed to 700, 1000 and 1440 pulses during 20 min. These pulses were generated by nitrogen plasma expander at 13.0 Pa and 6 kV. The samples were placed in the reactor (3.0 cm from the center of the conductor). According to [8-10], it was estimated an approximate value of 12 eV for the ion collision energy on the polished surfaces.

2.3. Characterization

All measurements for ceramic characterization were performed before and after the plasma exposure. The apparent porosity was determined according to ASTM C20-00 [12]. In order to determine roughness, it was used a profilometer Mitutoyo - Surftest 301, by choosing Ra (average roughness) and Rt (peak-to-valley roughness) parameters. For Vickers hardness measurements, it was used OttoWolpert-Werke, an equipment with 30 kgf load. The wettability of the sample surface was investigated by goniometer Rame-Hart 300-F1.

The light microscopy was used to monitor the change on the polished surfaces. The "ImageJ", public domain software, developed at the National Institute of Mental Health [13] was used to perform quantitative analyses of the images. The images used for porous and matrix fraction measurements were obtained from polished surfaces by using a Nikon microscope model Epiphot 200. SEM images were captured by Zeiss EVO LS-15 microscope.

3. Results and discussion

Table 1 shows the hardness and porosity results of each ceramic sample group. After the plasma exposure, there were no significant changes in bulk density and apparent porosity and the average values remained nearly the same. Probably, when nitrogen ions collided with the ceramics there wasn't enough energy to go into the material or to remove a large amount of material of the surface.

Table 1 also shows that Vickers hardness mean values decreased very little and standard deviation (dispersion) increased for all sets. These results confirm that nitrogen ions did not cause alterations in the alumina sample bulk.

Table 1. Bulk density, apparent porosity and hardness.

Group	Bulk density [g/cm^3]	Apparent porosity [%]	Vickers Hardness [kgf/mm^2]
		Before plasma expander	
1	3.83 ± 0.01	0.85 ± 0.27	1560 ± 80
2	3.81 ± 0.01	0.84 ± 0.23	1555 ± 80
3	3.81 ± 0.01	0.95 ± 0.23	1557 ± 80
Pulse		**After plasma expander**	
700	3.82 ± 0.02	0.83 ± 0.18	1528 ± 140
1000	3.81 ± 0.01	0.88 ± 0.15	1497 ± 160
1440	3.80 ± 0.01	1.12 ± 0.26	1541 ± 160

Figures 2a and 2b show micrographs obtained from the sample surface (group-1), before and after the plasma exposure, respectively. It can be seen an increase of sample pore number. Furthermore, there pores present a non uniform distribution and irregular morphology after shock wave exposure.

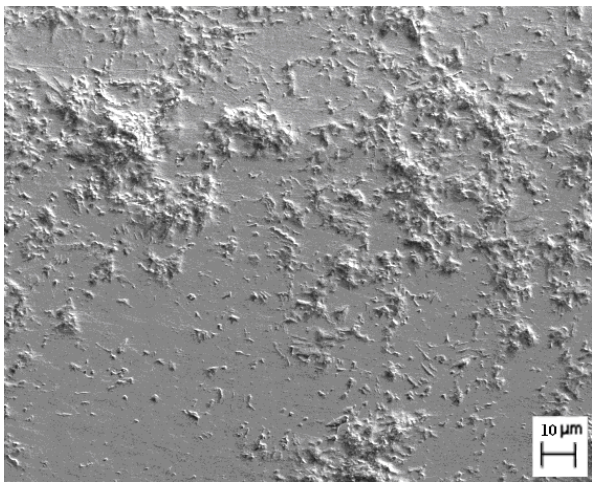

Figure 2a. Surface samples before plasma.

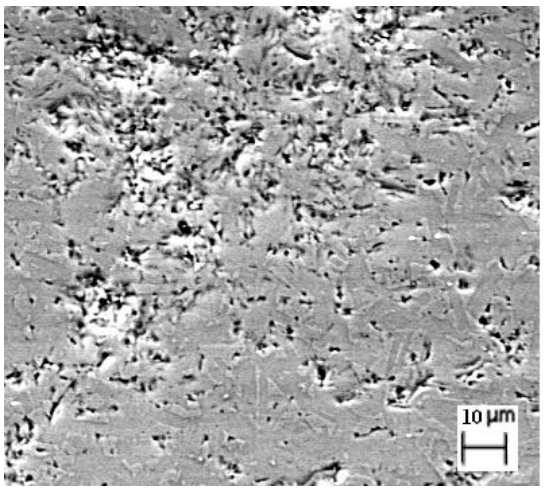

Figure 2b. Surface samples after plasma.

Table 2 shows roughness, wettability and porous fraction values. In this table, it can be observed that there were significant changes especially in roughness and wettability promoted by the shock waves. Despite the higher roughness values (described by Ra and Rt parameters), the average contact angle remained below 90°, i.e. the ceramic surface maintained the hydrophilic characteristic. The increase of wettability occurred due to the ablation process, which was generated by nitrogen ions of shock waves.

Table 2. Roughness, contact angle and porous fraction measurements.

Group	Ra (μm)	Rt (μm)	Contact angle (°)	Porous fraction (%)	Matrix fraction(%)
		Before plasma expander			
1	0.25 ± 0.06	2.53 ± 0.24	70.2 ± 1.0	21.7 ± 1.5	78.3 ± 1.5
2	0.43 ± 0.10	3.80 ± 1.19	71.3 ± 1.0	25.9 ± 3.2	74.0 ± 3.2
3	0.22 ± 0.06	2.63 ± 0.80	77.4 ± 1.0	20.7 ± 1.6	79.3 ± 1.6
Pulse		**After plasma expander**			
700	0.94 ± 0.14	8.53 ± 1.90	75.5 ± 1.0	24.4 ± 1.5	75.6 ± 1.5
1000	0.79 ± 0.23	5.99 ± 2.22	81.4 ± 1.0	28.7 ± 1.5	71.3 ± 1.5
1440	0.92 ± 0.12	6.34 ± 0.94	86.1 ± 1.0	23.6 ± 0.9	76.4 ± 0.9

4. Conclusions

The porosity measurement, which is based on Archimedes' Principle, and hardness results showed that the sample bulk remained practically unchanged. The plasma expander proved to be a novel surface treatment for promotes changes on Al_2O_3 ceramic surface.

The roughness, contact angle measurements and SEM image analysis indicate that there were changes in the sample surfaces due to momentum transfer from nitrogen ions to ceramic surface. The standard deviations determined after hardness and roughness tests, indicate that the nitrogen ions do not reach the entire ceramic surface with the same intensity.

SEM image analysis and roughness parameters indicate that the sputtering process by nitrogen ions occurs on the entire ceramic surface. However, the increase of the number of plasma pulses did not make the sputtering process more effective, probably due to the surface electric charging.

Acknowledgments

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