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# Surface roughening of ground fused silica processed by atmospheric inductively coupled plasma



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#### ARTICLE INFO

ABSTRACT

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*Keywords:* Atmospheric inductively coupled plasma Ground fused silica Surface roughening Subsurface damage (SSD) is a defect that is inevitably induced during mechanical processes, such as grinding and polishing. This defect dramatically reduces the mechanical strength and the laser damage thresholds of optical elements. Compared with traditional mechanical machining, atmospheric pressure plasma processing (APPP) is a relatively novel technology that induces almost no SSD during the processing of silica-based optical materials. In this paper, a form of APPP, inductively coupled plasma (ICP), is used to process fused silica substrates with fluorocarbon precursor under atmospheric pressure. The surface morphology evolution of ICP-processed substrates was observed and characterized by confocal laser scanning microscope (CLSM), field emission scanning electron microscope (SEM), and atomic force microscopy (AFM). The results show that the roughness evolves with the etching depth, and the roughness evolution is a single-peaked curve. This curve results from the opening and the coalescing of surface cracks and fractures. The coalescence procedure of these microstructures was simulated with two common etched pits on a polished fused silica surface. Understanding the roughness evolution of plasma-processed surface might be helpful in optimizing the optical fabrication chain that contains APPP.

#### 1. Introduction

Fused silica has been widely used in high-energy laser systems, such as the Laser Megajoule<sup>[1]</sup> and the National Ignition Facility<sup>[2]</sup>. This transparent material shows excellent chemical and mechanical properties, making it an ideal material for the high-energy laser systems, military devices, and deep-sea technology devices. However, traditional mechanical processing methods used to fabricate fused silica inevitably lead to subsurface damage (SSD) [3,4]. Pores, absorbing impurities, particularly cracks and fractures contained in a subsurface, severely reduce and weaken the laser damage threshold (LDT) of the fused silica substrates [5–8]. Meanwhile, the mechanical strength of components is limited by its surface processing properties and the near-surface flaws, rather than by defect-free bulk material [9]. Due to the limitation of fused silica's LDT and its strength, performance and lifetime of the high-energy systems can be degraded [10]. Hence, material processing methods and techniques that reduce or do not induce SSD have attracted large numbers of scientific research, such as atmospheric plasma processing [11–13] and magnetorheological finishing (MRF)[14]. Atmospheric plasma processing as a kind of gas jet processing

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technique, its tool is less viscosity and more flexible than the tool of MRF. This makes the tool adapt better to the local surface shape in machining freeform or steep concave workpiece than the method using ferro fluid abrasives. Because there is no mechanical contact or physical loading on the substrate, almost no surface damage or SSD is induced to the substrate surface. The surfaces etched by plasma are supposed to be flat and uniform, yet plasma processing always makes surface roughness develop. Recently, researchers have experimentally and theoretically reported on the mechanism and the reasons for roughness evolution on plasma-etched surfaces. Zhao et al. performed simulation and theoretical investigation on plasma-etched Si (100), which showed that surface roughness had a close relationship with redistribution of reactive radicals and shadowing effect [15,16]. Martin and Cunge reported that the intrinsic etching characteristics of plasma made the surfaces of silicon components smoother, rather than rougher. And the roughness resulted from nonvolatile species sputtered from the reactor walls and deposited onto the silicon surface [17]. The research of Kokkoris et al. showed that "hard" inhibitors and "soft" inhibitors were the main factors leading to the nanoroughness of substrate surfaces processed using plasma etching [18]. Accordingly, the fundamental mechanism for plasma etching is complicated and not fully understood; many researchers are exploring the factors that influence the evolution of plasma-processed surface roughness.

The studies on roughness evolution mentioned above were carried out under the conditions of low pressure or vacuum, where the material removal rate was relatively low. Studies on the roughness evolution with relatively high material removal rate under atmospheric pressure are rare. In this paper, we report the experimental results of fused silica surfaces roughness evolution using Ar/CF<sub>4</sub> plasma at atmospheric pressure. The morphology and the roughness evolution of fused silica substrates surface were characterized by confocal laser scanning microscope (CLSM), field emission scanning electron microscope (SEM), and atomic force microscopy (AFM). Understanding the formation and evolution of roughness during atmospheric plasma processing are of primary importance in processing silica-based material.

#### 2. APPP-ICP system and processing characteristics

The schematic diagram of the experimental system is shown in Fig. 1. The atmospheric pressure plasma processing-inductively coupled plasma (APPP-ICP) method utilizes atmospheric pressure plasma to excite reactive radicals. The plasma generated by radio-frequency power can be regarded as a chemical reactor; the reactant gas fed into the reactor is decomposed by the collision of plasma electrons into active species [11]. These reactive radicals then react with the surface atoms to accomplish the atomscale material removal process. The products are always volatile gases that are easy to vent and handle. Inductively coupled radiofrequency electric power at 27.12 MHz is supplied to the plasma via the load coil through an impedance matching box (PSG-III, Rishige, China). The schematic drawing of the APPP-ICP process with Ar/CF<sub>4</sub> mixtures is shown in Fig. 2. The torch consists of three coaxial quartz tubes, with two tangential flows of ultrapure argon (99.999%) plasma gases passing through the outer gap and inner gap. The reactant gas,  $CF_4$ , is introduced into the plasma via the inner tube at the same time. And the radio-frequency power is then applied to the load coil. Subsequently, the gases are ignited by free electrons, which produced by a high-voltage spark. Finally, the inductively coupled plasma-containing reactive radicals are formed at the open end of the torch. The reactive radicals, carried by plasma jet flow, diffuse to the substrate surface and react with the surface material. The balanced chemical reaction equation that used in this process can be described as follows:

 $SiO_2 + CF_4 \rightarrow SiF_4 \uparrow + CO_2 \uparrow$ 

The SSD of mechanically machined brittle substrate surface consists of a residually stressed surface layer caused by the permanent deformation of the surface and a cracked layer [19]. This cracked layer could be removed by plasma chemical processing and no sputter associated subsurface damage occurred on the surface [20]. And the SSD removal procedure had been theoretically reported



Fig. 1. Schematic diagram of the experimental system setup.



Fig. 2. Schematic drawing of the APPP–ICP process with Ar/CF<sub>4</sub> mixtures.



Fig. 3. Experimental material removal profile and its Gaussian fitting curve.

by Zarowin [21]. The stress in the residually stressed surface layer could be mitigated by plasma processing and an undamaged and contaminant-free surface could be left behind [22]. Thereby, the SSD can be removed or relieved, and a surface with less SSD can be obtained after the plasma processing. What's more, the static etched footprint of APPP–ICP is proved to be near Gaussian-shaped showed in Fig. 3 which complies with the deterministic optical surfaces machining idiosyncrasies reported by Jones [23], Xin [24], and Wenzel and Mcfalls [25]. Also, a high removal rate at about 30 mm<sup>3</sup>/min can be achieved using APPP–ICP. These characteristics make the APPP–ICP an ideal tool in figuring optical surface errors efficiently with better convergence properties and, more importantly, with less or without SSD at the meantime.

#### 3. Experiment details

#### 3.1. Sample preparation

Fused silica samples (JGS<sub>2</sub>) were obtained from China Building Materials Academy, with the diameter of 50 mm and the thickness of 5 mm. The samples were ground with YM015.2A (Nanjing Lisheng Optics Machinery Co., China) to remove about 300  $\mu$ m material. In order to eliminate the contaminate influences on plasma processing, the samples were fully cleaned and rinsed in

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