Journal of the European Ceramic Society xxx (2017) xxx–xxx

Contents lists available at www.sciencedirect.com

Journal of the European Ceramic Society

journal homepage: www.elsevier.com/locate/jeurceramsoc

Feature article

Surface property modifications of silicon carbide ceramic following laser shock peening

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ARTICLE INFO

Article history:
Received 15 September 2016
Received in revised form 24 February 2017
Accepted 2 March 2017
Available online xxx

Keywords:
Laser shock peening
LSP
SiC
Ceramics
Hardness
K_{1c}
Residual stress
Microstructure
Surface finish
Composition and phase transformation

ABSTRACT

This paper if focused on Laser shock peening (LSP) of silicon carbide (SiC) advanced ceramic. A comprehensive study was undertaken using a pulsed Nd:YAG laser. Surface modifications were investigated, particularly: the roughness, hardness, fracture toughness, microstructure, phase transformation and residual stress induced before and after the LSP surface treatment. The findings showed increase in the surface roughness, changes to the surface morphology, improved hardness, and a reduction in the fracture lengths. The LSP surface treatment also improved the surface fracture toughness from an average of 2.32 MPa $m^{1/2}$ to an average of 3.29 MPa $m^{1/2}$. This was attributed to the surface integrity and the induced compressive residual stress as a maximum of −92 MPa was measured compared to an average of +101 MPa on the as-received SiC. A slight change in the surface chemistry was also observed from the XPS spectra, however, no real phase transformation was seen from the x-ray diffraction analysis. Laser energy density of around 1.057 J/cm^2, 8.5 mm spot size, 10 Hz pulse repetition rate (PRR) at 6ns pulse duration, and 1064 nm wavelength resulted to obtaining a crack-free surface treatment and demonstrated that the technique is also beneficial to enhance some of the properties to strengthen brittle ceramics such as SiC.
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1. Introduction

Laser shock peening (LSP) or laser shock processing has been an applied technique for many years to engineer surface properties of metallic materials [1–4]. This is so because the process offers many benefits such as, improvement in fatigue and hardness; reduced wear rates; increase in compressive stresses just to mention a few. Such benefits have been addressed extensively for metals and alloys over the last two decades [1–5]. With that said, research in LSP of ceramics is rare due to their physical properties inhibiting mechanical yielding and plastic deformation in the same way as it occurs with metals. This is particularly so when ceramics are introduced to intense shock pulse pressure during LSP surface treatment. Therefore, it is not common to obtain the same gains which are conventionally obtained by LSP of metals and alloys. Thus, it is extremely challenging and fruitful to investigate the effects of LSP upon ceramics such as SiC. A successful process to strengthen SiC for machine tool applications for instance could manifest faster spindle speeds, higher feeds rates, with less wear, and have longer operational life. The work in this paper would also enable one to understand the effects of the pulsed laser energy interaction with brittle ceramics such as SiC.

Over two decades of research has been conducted in the area of LSP of metals and alloys [6–11]. Published literature in this area has evolved from the use of micro-second, nano-second to even shorter pulses being applied in both picosecond and femtosecond range on metals and alloys [11–15]. In terms of ceramics, there is very little progress made to-date. Koichi et al. [16], and the preliminary work from the leading author of this study [17], as well as the work of Schnick [18], are the few investigations that exist in the field of LSP of ceramics such as Si_3N_4 and Al_2O_3. Koichi et al. used the Nd:YAG laser at 532 nm to laser peen a Si_3N_4 ceramic. Their results reported an increase in surface roughness as the laser irradiance increased. This was reported due to plastic straining as the surface layer of the Si_3N_4 was induced with compressive residual stress with increase in laser irradiance. However, other findings in their work showed reduction in strength, thus, showing inconsistency and contradiction with their results, as residual

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http://dx.doi.org/10.1016/j.jeurceramsoc.2017.03.005
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Please cite this article in press as: P. Shukla, et al., Surface property modifications of silicon carbide ceramic following laser shock peening, *J Eur Ceram Soc* (2017), http://dx.doi.org/10.1016/j.jeurceramsoc.2017.03.005

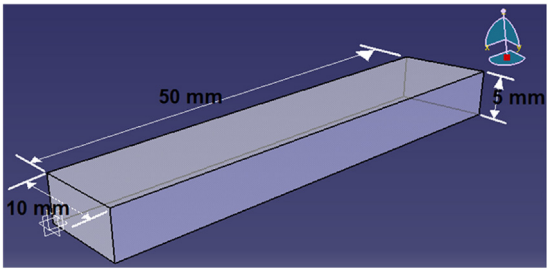

Fig. 1. A schematic of the SiC ceramic used for laser shock processing surface treatment.

compressive stress in general would indicate surface strengthening leading to a reduction in the flaw size unlike the findings reported by Koichi et al. Bending strength was also reported to be lower than the as-received surface, but showed some inconstancy between identical samples. Important details such as evidence of the laser shock peened microstructures were not illustrated in their report. Furthermore, Schnick et al. [18] investigated the laser shock processing of Al + SiC particulate composite coatings. The coating was produced using a spray of powders that ranged between 100 μm to 500 μm over 30 samples. The results showed a modified morphology of the sprayed coatings as the surfaces became smoother and the coatings were less porous. In addition, the process was made to improve wear resistance of high-velocity oxygen fuel sprayed Al + SiC particulate composite coatings.

The motivation of this paper is to examine the surface effects of LSP upon a brittle material such as SiC ceramic by particularly investigating modifications in the hardness, fracture morphology and the microstructure, plane strain fracture toughness (K_{Ic}) along with changes in the residual stress, phase transformations using X-Ray Diffraction (XRD) followed by examination of the surface chemistry with the use of X-ray Photoelectron Spectroscopy (XPS). To observe the effects of LSP on SiC, only the laser energy density was changed whilst keeping other parameters constant. This work is a first-step towards developing an LSP technique for SiC based ceramics for applications such as machine tools as well as other industrial ceramics in general. The effects of LSP were investigated to fill the gap in knowledge and will also demonstrate a first-step towards providing a strengthening technique which could improve the service life of industrial ceramics and create new avenue for their applicability, especially where ceramics could replace other materials but are currently restricted due to crack sensitivity and low fracture toughness.

2. Experimental procedures and analysis

2.1. Material characterization methods

2.1.1. Background of the SiC advanced ceramic

The SiC ceramic was mechanically and microstructurally characterized prior to all experimentation. The ceramic was cold pressed using isostatic pressing technique (CIP) by Shanghai Unite Technology, China. It was produced with dimensions of 50 × 10 × 5 mm^3 for the LSP experiments (see in Fig. 1). The CIP process was conducted at 455 bar pressure and was sintered at a temperature of 1200 °C for 5 h. The average grain size was 17.1 μm and ranged from 9 μm to 26 μm over a 100 μm^2 area of the polycrystalline SiC ceramic.

2.1.2. Surface finish, hardness testing and fracture toughness measurements (K_{Ic})

Surface finish was examined using a form Taylor Hobson, Talysurf Series 2, PGI plus – 8 nm; Leicester, U.K., for both the as-received and LSP samples. The average as-received surface finish (from 5 samples) was Ra 1.53 μm. Indentation tests were carried out with a Vickers macro/micro indentation method using 73.6N indentation load (VHTM 2000; Vickers Ltd. Engineering Group; Sheffield; England). Fracture toughness (K_{Ic}) was determined based on the Vickers indentation technique in relation to our previous work [19–22]. The surface hardness was measured to be 12.50 GPa and the K_{Ic} was determined as 2.31 MPa $m^{1/2}$.

2.1.3. Microstructural observations

Following the LSP surface treatment, a detailed observation of the microstructure of the laser shock peened zone was undertaken and all crack lengths found after Vickers hardness tests were observed using optical microscopy and with a scanning electron microscopy ((SEM) SUPRA 40, Zeiss SMT AG; Germany). Both the as-received and the laser shock peened samples were cross-sectioned using a diamond cutter and polished with 600 μm, 200 μm, 6 μm and 1 μm polishing cloth for approximately 8mins each so that the cross-sectional microstructure could be examined. The cross-sectional analysis required chemical/thermal etching to reveal the microstructure of the SiC ceramic. The etching process adopted the use of Murakami reagent at boiling temperatures (approximately 200 °C) for about 30 mins in a high temperature furnace.

2.1.4. Compositional analysis

Samples were analyzed for elemental change in the surface and near-surface regions using x-ray photoelectron spectroscopy (XPS) and scanning electron microscopy-energy dispersive x-ray (SEM-EDX) measurements were made using a silicon drift detector system (X-Act with INCA software, Leo 1455VP SEM, Oxford Instruments; U.K.). The XPS analyses were performed using a bespoke ultra-high vacuum chamber fitted with Specs GmbH Phoibos 150 analyzer, Focus 500 monochromater and F20 charge neutralizing gun. Spectra were acquired using the Al monochromatic source, 1486.6 eV x-ray energy with an analysis area approximately 2 mm diameter. The SiC ceramic was also analyzed for elemental change in the surface and near-surface regions using XPS and SEM-EDX prior to and after the LSP surface treatment.

2.1.5. Phase transformation and residual stress analysis

A detailed analysis of the phase evolution was carried out by XRD technique (Bruker D8 Discover, Germany) with Cu Kα radiation (wavelength ≈ 0.15418 nm) at a scanning speed of 0.02°/s. The x-ray source was operated at an accelerating voltage of 40 kV and current of 25 mA. The size-strain plot (SSP) method was used to calculate the crystallite size, strain, and stress on the as-received SiC surface and laser shock peened SiC based on previous methodology of Bindu and Thomas [23]. The SSP method has a significant advantage over other methods, so that less importance is given to the high angle peaks and it is assumed that the "strain profile" is characterized by a Gaussian function. The "crystallite size" is characterized by a Lorentzian function [23]. Hence, the SSP approximation is [23]:

$$(d_{hkl}B_{hkl}cos\theta_{hkl})^2 = \frac{1}{V_s}(d_{hkl}^2 B_{hkl}\cos\theta_{hkl}) + \left(\frac{\varepsilon}{2}\right)^2 \quad (1)$$

where d_{hkl} is the interplanar distance between (hkl) planes, β_{hkl} is the full width half maximum of the peak at a particular (hkl) which was obtained after subtracting from the instrumental broadening, V_s=(3/4)D_v (where D_v is the crystallite size), and ε is the apparent strain. The crystallite size and strain were calculated from the slope and intercept of the plot between $(d_{hkl}\beta_{hkl}\cos\theta_{hkl})^2$Vs $(d^2{}_{hkl}\beta_{hkl}\cos\theta_{hkl})$, where $(d_{hkl}\beta_{hkl}\cos\theta_{hkl})^2$ was plotted on the Y-axis and $(d^2{}_{hkl}\beta_{hkl}\cos\theta_{hkl})$ on the X-axis.

Residual stress on the laser shock peened and untreated zones were also measured using the XRD technique (d Vs $\sin^2\Psi$ technique) by application of a stress Goniometer attached to a Bragg Brentano Diffractometer (Bruker D8 Discover; Germany). The X-ray

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