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Relating residual stresses to machining and finishing in silicon carbide

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Abstract

Machining and surface finishing is essential in the processing of many ceramics, however, it can be detrimental to subsequent performance as it introduces residual stresses and structural defects. Using micro-Raman spectroscopy the residual stress and crystallinity of hot-pressed SiC tiles were examined after finishing with several different methods. 514 nm and 633 nm lasers in both conventional and confocal settings enabled stress as a function of depth to be assessed. Single crystal, electronics grade SiC was used for comparison. Compressive residual stresses were present at the surface and in the sub-surface region in all the samples, but the surface with lowest roughness and a mirror-like finish had the highest residual stress. Crystallinity in SiC is reduced by defects and stacking faults produced during machining and finishing. The finer surface finishes had higher residual stresses, but were actually found to have better crystallinity than the rough surfaces. The dependence of residual stress and crystallinity on surface roughness is attributed to a change in the mode of ceramic removal from energetic and brittle for rough finishes to ductile for the shallow cuts of smooth finishes. The dependence of stress and crystallinity on roughness means a balance must be struck between smoothness, crystallinity and stresses in choosing the best finish for a given application.
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1. Introduction

1.1. Silicon carbide

Silicon carbide is of great importance as a structural ceramic as it is amongst the hardest materials known [1]. As such, machining SiC parts into the required shape and form for a given application can be difficult and may yield unwanted defects. Whether the pieces are machined by grinding and polishing, laser-assisted techniques, or ultrasonic techniques [2], the potential for residual stresses to form at the surface is always present. It is important to know how much stress machining can impart to components, since this stress may be either detrimental, leading to early failure, or advantageous, by helping to toughen the component.

Silicon carbide's crystal structure consists of tetrahedra of either 4 Si atoms and 1C atom, or 4C atoms and one Si atom [3]. The tetrahedra can be stacked in either parallel or anti-parallel sequences, and this stacking allows for SiC to take on many different polytypes. The most common polytypes are a cubic structure known as 3C (or beta-SiC) and 2 hexagonal phases 4H and 6H, known as alpha-SiC. Each polytype has slightly different mechanical, optical and electronic properties that make them useful for specific applications for instance as armor ceramics, power electronic semiconductors or industrial abrasives [4]. As a result of their different structures each polytype has its own unique Raman spectrum. This makes Raman spectroscopy very useful for characterizing variations in polytype within a sample and, significantly for the current study, changes in the Raman peak position can be related to strains (and hence stresses) in the SiC [5,6].

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In single crystal SiC, stress concentrators take the form of micropipes [7,8] and dislocations [8]. For some applications what is more detrimental than the increased stress is the fact that these features often impinge on electronic properties and therefore performance when used as single crystal semiconductors. As electronic properties are less important for bulk polycrystalline samples used in structural applications this effect is not so significant, nonetheless when machining SiC it is important to limit the defects so they are not able to act as stress intensifiers. In bulk, polycrystalline samples, stress points can come from processing inclusions and thermal stresses [9,10] created during hot-pressing or heat treating. If the density of defects is large enough, different polytypes with mis-matching lattices are formed or stacking faults are present it can also cause a significant stress in the sample [11,12]. Machining can further augment these stresses by introducing more stacking faults and increasing the defect density.

1.2. Raman spectroscopy

Raman spectroscopy is a form of vibrational spectroscopy that relies on inelastic scattering of monochromatic light to provide vibrational, rotational and other low-frequency mode information about a material system [13]. Data obtained from Raman spectroscopy comes in the form of a vibrational spectra. For a given material system, this spectrum will be unique, and contain information related to bonding between atoms within that system. The theory and fundamentals of Raman have been discussed at great length elsewhere [13–15], as well as the ability to use Raman as a stress measuring tool [16–21]. In the case of silicon carbide the numerous polytypes that can form all have unique Raman spectra. This allows for easy characterization and distinction between polytypes, as seen in Fig. 1. The use of Raman to measure stress in silicon carbide has been examined quite thoroughly. However, these experiments have been done mostly on the cubic, 3C, polytype [12,22–28] because of its importance and wide usage in the power electronics field. Only a few papers [5,6,29] discuss the use of Raman for stress analysis in 6H–SiC. In the current study we have focused on the equations developed by Liu and Vohra [5,6] for analysis of data as these are widely accepted for use on 6H silicon carbide.

The micro-Raman system used in the study was an inVia™ from Renishaw Inc. (New Mills, Wotton-under-Edge Gloucestershire, GL12 8JR, UK) with 633 nm and 514 nm lasers. The instrument's resolution in terms of deconvoluting overlapping peaks is on the order of 5 wavenumbers with a precision of 0.05 cm^{-1}, and an accuracy of 0.25 cm^{-1}. This means that peaks closer than 5 cm^{-1} could not be deconvoluted; however for the peaks of interest in silicon carbide this is not an issue. The penetration of the laser into the sample and hence the depth from which information is obtained is wavelength dependent and a function of the material being investigated. Penetration depth, D_p, can be estimated from the absorption coefficient, α [19]:

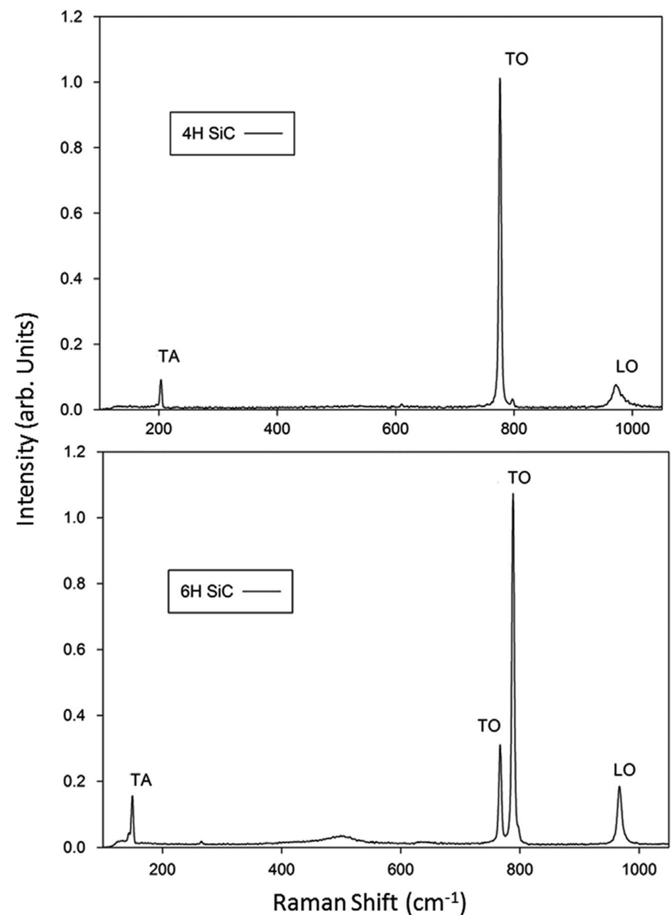

Fig. 1. Sample spectra for 4H-, and 6H–SiC. The variation in both position of optical phonons and unique acoustic modes allow for characterizing different polytypes. Peaks are designated by their propagation direction, with TO, TA and LO being transverse optical, transverse acoustical and longitudinal optical, respectively.

$$D_p = \frac{1}{\alpha(\lambda)}$$

where α depends on the wavelength, λ. In the case of SiC the 514 nm wavelength has a higher absorption coefficient than the 633 nm wavelength, which allows for analysis at different depths [30,31].

Penetration depth can be further tuned by using confocal Raman spectroscopy [32-37]. This involves the beam being focused with a numerical aperture, and then shone onto the sample. A confocal aperture, which can be a physical pinhole or a "virtual" pinhole, acts like a slit to block the path of some of the reflected beam. This allows only photons from a narrow focal plane to be focused on the detector. For non-confocal Raman spectroscopy the beam interacts with the sample at a range of depths and all of these are detected by the Charge Coupled Device (CCD) camera, but with the confocal feature light from only a narrow range of depths below the material's surface is sampled; the confocal aperture excludes light from higher and lower depths. This allows each laser available to the Raman operator to be used for two depths settings: a normal depth setting that goes farther into the sample and a confocal

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