Fracture toughness of polycrystalline silicon carbide thin films

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(Received 13 August 2004; accepted 16 December 2004; published online 11 February 2005)

Thin film polycrystalline silicon carbide (poly-SiC) doubly clamped microtensile specimens were fabricated using standard micromachining processes, and precracked using microindentation. The poly-SiC had been deposited on Si wafers by atmospheric pressure chemical vapor deposition, a process which leads to residual tensile stresses in the poly-SiC thin films; we measured the residual stress adjacent each specimen via a micromachined strain gauge. The stress intensity factor, \( K_I \), at the crack tip in each specimen depends on the magnitude of these residual stresses and the precrack length. Upon release, those precracks whose stress intensity exceeded a critical value, \( K_{IC} \), propagated to failure, whereas no crack growth was observed in those precracks with \( K < K_{IC} \). The fracture toughness so determined was \( 2.8 \pm K_{IC} = 3.4 \) MPa m\(^{1/2} \). Our technique also allowed us to assess any susceptibility to moisture-assisted stress corrosion cracking, which proved to be essentially absent in poly-SiC. © 2005 American Institute of Physics. [DOI: 10.1063/1.1864246]
using the following fabrication sequence: (i) a 2.8 μm thick 3C poly-SiC film with an average grain size of 25 nm was deposited onto a 100 mm diameter (100) Si wafer via APCVD using SiH₄, C₃H₈, and H₂ at 1050 °C;¹⁸ (ii) a Ni masking layer was sputter deposited; (iii) standard photolithography was used to pattern the devices in photoresist; (iv) the Ni mask was wet-chemically etched, and the poly-SiC was reactively ion etched using CHF₃, O₂, and He; and (v) the Ni mask was removed by wet etching and the substrate masking layer was sputter deposited; and (vi) the substrate was indented using a Vickers microindenter with a 1 kg load.

As shown in Fig. 1, the radial crack that had formed at the corner of indents placed ~30 μm from the edge of the beam in the Si substrate propagated through the Si substrate into the overlying poly-SiC film, thus generating a precrack in the beam. The precrack length was measured using high-resolution scanning electron microscopy (SEM). Due to the stochastic nature of indent-induced cracks and the variation in the distance from the center of the indent to the edge of the beam, the precrack length varied significantly among different beams.

Before release, the 100 mm wafer was sectioned into 1 cm² dice, each containing 12 poly-SiC microtensile devices. Due to the vagaries of our CVD reactor, the residual stress of the poly-SiC film varied across the wafer, although room temperature stresses were consistently tensile due to the thermal expansion mismatch with the Si substrate. Therefore, the local stress in the poly-SiC device in each die was measured using microstrain gauges fabricated adjacent to the tensile beams.¹⁷ Figure 2 shows a micrograph of a poly-SiC strain gauge; upon release of the device, the sign and magnitude of the displacement of the verniers at the ends of the long suspended beams allows the residual stress to be determined with a 2 MPa uncertainty. Young’s modulus of the poly-SiC on each die was also determined using lateral resonant devices.¹⁹

The beams were released by etching away the underlying Si using a 1:2:2 aqueous solution of HF:HNO₃:CH₃COOH. Due to the large lateral dimensions of the anchor pads at both ends of the beam, these pads remained attached to the Si substrate after the beam was fully released. Upon release, the residual tensile stress in each doubly clamped poly-SiC microtensile specimen produced a well-defined stress intensity at the crack tip. The variation in crack lengths from specimen to specimen and the associated variation in the stress required to maintain the fixed displacement conditions at the ends of the specimens produced a range of K’s at the crack tips, which were calculated through finite element analysis.²⁰ The determination of residual stress and stress intensity both depend on Young’s modulus; the average value obtained by the lateral resonant devices, 393 GPa, is consistent with previous data on APCVD SiC films.⁶,⁸,₂¹,₂²

Seven dice were studied; the average residual stress of the poly-SiC on these dice varied from 149 to 214 MPa. The stress intensity factor for each die is shown as a function of precrack length in Fig. 3, which also displays the results of testing 73 specimens from these different dice. The fracture toughness, Kᵣ, was bounded by the range between the lowest K that resulted in crack propagation and the highest K that did not. There is some scatter in the results from devices with different residual stresses, but taken as a whole, the data indicate that 2.8 ≤ Kᵣ ≤ 3.4 MPa. The scatter in the data is attributed mainly to the uncertainty in the film stress measurements¹⁷ and the possibility that the crack front is inclined to the film normal, as the experiment is otherwise both rigorous and straightforward. (We note that poly-SiC satisfies the demands of linear elastic fracture mechanics, i.e., it is a perfectly brittle material at room temperature with atomically sharp crack tips.) For comparison, Kᵣ, for polysilicon determined using the same technique is 0.81 ± 0.05 MPa m¹/².¹⁷

Following the fracture toughness experiments, a total of 24 beams with precracks that had not propagated and were loaded by residual tensile stresses of 150, 159, or 214 MPa were placed in a chamber at 90% relative humidity. The residual tensile stresses induced a range of stress intensities from 1.94 to 3.30 MPa m¹/² at the crack tips, just at or below Kᵣ. Thus, if the moist environment induced any subcritical crack growth, the cracks would eventually reach a critical length and propagate to failure. The microspecimens were held in this humid ambient for 30 days, but no crack growth was observed. The absence of crack growth in these specimens indicates that silicon carbide is not susceptible to moisture-induced static stress-corrosion cracking. (Given the
uncertainty in crack length measurement following the 30 day exposure to a moist ambient, the maximum crack growth rate that could have occurred in the experiment was $3.9 \times 10^{-14} \text{ m/s}$. This is similar to the case of polysilicon, where conventional stress corrosion cracking is also absent.\(^\text{15}\)

In summary, poly-SiC single edge-cracked doubly clamped specimens were fabricated using processes and size scales relevant to MEMS devices. Atomically sharp pre-cracks were introduced via microindentation, and the stress intensity at each crack tip was determined from an independent measure of the residual stress. The fracture toughness of poly-SiC was $2.8 \leq K_{IC} \leq 3.4 \text{ MPa m}^{1/2}$. Twenty four pre-cracked specimens with stress intensities just at or below $K_{IC}$ displayed no stress corrosion-induced subcritical crack growth after 30 days at 90% relative humidity.

\(^{1}\)G. L. Harris, Properties of silicon carbide (The Institute of Electrical Engineers, London, 1995).


\(^{20}\)The finite element code FRANC2D was used to determine stress intensities. This code is available from the Cornell Fracture Group at http://www.cfg.cornell.edu/

