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Hardness and fracture toughness of moissanite

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Abstract

Disparities prevail among the reported hardness and fracture toughness values for hard and brittle materials. A better understanding of the physical nature of hardness and fracture toughness and a standardized technique for reliable measurements of these quantities is urgently needed. We strongly recommend the use of the measured hardness after the bend in the hardness versus load ($H-F_{\mathrm{Load}}$) curve, when the hardness approaches its asymptotic value. The present work reports a systematic study of hardness and fracture toughness on moissanite (single crystal hexagonal silicon carbide, 6H-SiC) samples. The measurements were performed over a broad load range from 0.49 to 294 N with the direct indentation method. Asymptotic values of Knoop hardness of $H_{\mathrm{K}}=19$ GPa and Vickers hardness of $H_{\mathrm{V}}=22$ GPa were reached at a high load between 50 N and 100 N. A consistent fracture toughness of $K_{\mathrm{IC}}=1.8$ MPa·m$^{1/2}$ was obtained across the entire load range. Our study presents experimental results for the hardness and fracture toughness of moissanite in the asymptotic-hardness region, and it raises concern regarding the application of moissanite single crystals as anvil material under shear/fconditions.
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Keywords: Silicon carbide; Hardness; Fracture toughness

1. Introduction

Thanks to the availability of large, gem quality single crystals with high hardness and relatively low cost, moissanite (6H-SiC) has become a popular material for anvils for high-pressure research in addition to the traditional diamond [1–6]. Moissanite's wide band gap (3.0 eV), X-ray transparency (energy > 25 keV), high thermal conductivity (500 W m^{-1} K^{-1} at 293 K), and high thermal stability (1400 K in the air) make it an ideal window for optical spectroscopy and X-ray diffraction. To date, the highest pressure of 58.7 GPa at room temperature and the highest temperature of 3700 K at 3 GPa have been achieved with a moissanite anvil cell [2,3]. Furthermore, large moissanite anvils with the capability of compressing cubic millimeter samples to a pressure higher than 30 GPa is expected to be able to extend the pressure–temperature range of other analysis techniques such as neutron diffraction (with the advantage of moissanite's small absorption cross-section), [5] inelastic X-ray spectroscopy, and ultrasonic interferometry to a new level [6].

The prospect of moissanite as the anvil material for the next-generation, large volume, high-pressure apparatus stimulated our interest to evaluate its fundamental mechanical properties, namely the indentation hardness and fracture toughness. There are extensive studies about the mechanical properties of sintered silicon carbide in polycrystalline form with the presence of sintering aids such as boron, carbon and boron carbide, [7–10] but only a few works have been published for single crystal silicon carbide and there is a discrepancy among them [11–13]. In the scope of this work, we report a systematic study of the indentation hardness and fracture toughness of moissanite under a wide range of load from 0.49 N to 294 N.

2. Experimental

The indentation method was adopted in the experiment due to its unique simplicity and economy [14,15]. Under this direct approach, we applied a load F_{Load} (in N) on a

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pointed indenter (Knoop or Vikers type) to produce a hardness indentation with a diagonal length 2a (unit in μm) on the mirror smooth surface of a moissanite single crystal. For Vickers hardness H_V (in GPa), the total length of the radial cracks extending from the indent 2c (unit in μm) was also measured to calculate the fracture toughness K_{IC} (unit in MPa·m$^{1/2}$) [16].

$$H_K = 3557.3 F_{Load}/a^2$$

$$H_V = 463.5 F_{Load}/a^2$$

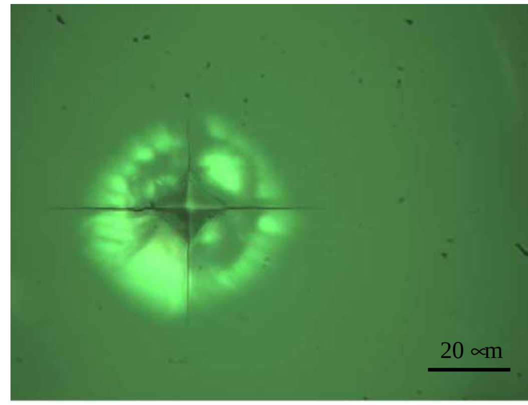
$$K_{IC} = 16(E/H_V)^{1/2}\left(F_{Load/c^{3/2}}\right)$$

H_K is the Knoop hardness; E is the Young's modulus, 447 GPa, which is calculated from the bulk (220 GPa) and shear (192 GPa) moduli determined for polycrystalline 6H-SiC [17].

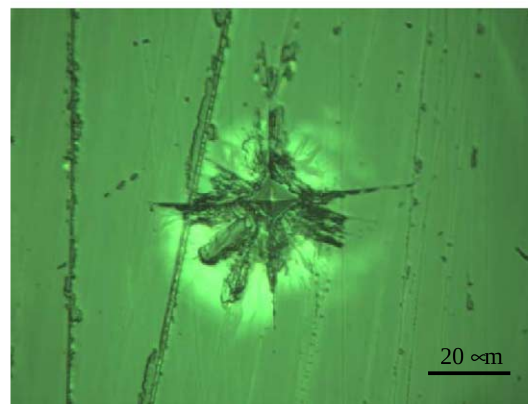
Moissanite crystals were grown by Charles and Colvard Ltd. [18]. The crystal was polished to a 16-mm height cone shape which has a flat culet with a diameter of 5 mm. A Buehler microhardness MMT-3 tester (load from 0.245 N to 19.6 N) and a macrohardness 5114 tester (load from 9.8 N to 490 N) were used for the indentation measurements. As a precaution to minimize slow crack growth due to moisture in the air, a drop of immersion oil was placed on the pre-selected contact site prior to indentation, and the indentation axis was always maintained parallel to the c-axis crystallographic direction of the moissante single crystal. The indent diagonal and the crack lengths were immediately measured with appropriate magnification (100×, 150×, 200×, 600×, or 1500×) after the indentation. The loading time was 10 s and at least five indentations were made for each load to provide satisfactory statistics. All measurements follow the procedures specified in ASTM E384-99e1 [19]. Extreme care was taken during these microscopic measurements to reduce the experimental uncertainty. Crack type estimation was done by polishing the indented samples with 1 μm diamond paste, and images were recorded with the CCD camera attached to the Buehler MMT-3 tester.

3. Results and discussion

Indentation marks right after loading (F_{Load}=4.9 N, Vickers indenter) and polishing are shown in Fig. 1(a and b), respectively. Well-developed radial-median (halfpenny shaped) cracks, which remain attached to the indent, are persistently observed throughout the polishing process for indentation marks made at loads higher than or equal to 1.96 N. But due to the combination of the rather tiny indent and surface roughness produced by polishing, it is difficult to establish the crack type of indentation marks made at loads lower than 1.96 N. It has been concluded that many materials can show both radial-median and Palmqvist cracks (which detach from the indent after polishing due to their shallow penetration in the cross-section direction). Commonly Palmqvist cracks appear at low loads and evolve into radial-median cracks at higher loads [14,20].



(a)



(b)

Fig. 1. Optical images of indentation mark made on moissanite crystal surface after loading. F_{Load}=4.9 N: (a) right after loading, (b) after polishing with 1 μm diamond paste.

The Vickers hardness versus load curve of the Buehler certified standard block was measured prior to that of moissanite. A nearly flat curve was obtained across the load range, shown as the line with squares at the bottom of Fig. 2. This guarantees that possible instrument and measurement factors affecting precision and bias in microindentation hardness test are minimized in our measurement. The indentation size effect, [10,13] under which hardness decreases with increasing indentation load, is observed with both Knoop and Vickers hardness of moissanite. An asymptotic hardness is reached at a load between 50 N and 100 N; solid up-triangle plot and open down-triangle plot in Fig. 2 stand for Vickers and Knoop hardness, respectively.

The Vickers hardness at low loads ($F_{Load} \leq 9.8$ N) falls in the commonly listed range of 28±3 GPa in the literature [21], and it decreases to an asymptotic value of H_V=22 GPa at high loads ($F_{Load} \geq 98$ N). The variation of hardness at small loads could be caused by the change of the tip geometry of the diamond indenter. However, no difference can be observed between the indentation marks on the