Nanomechanical characterisation of single-crystal Bi$_2$Se$_3$ topological insulator

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Topological insulators (TIs) are recently discovered high-tech materials where their potential use in nanoelectronic devices such as spintronics and quantum computers, due to their unique electronic features, can be a solution to the emerging need for high-bit data processing. Yet their mechanical properties are not well understood for their use in practical applications. To understand the usefulness of Bi$_2$Se$_3$ in practical applications, a comprehensive research of indentation crack length (ICL) methods. The grown Bi$_2$Se$_3$ exhibits high carrier concentration ($>$ 1019 cm$^{-3}$) [13] of Bi$_2$Se$_3$ allows tuning the Fermi level from valance to conduction band, thus controlling the electron transport properties and conductivity. Despite many exceptional works concerning electronic [14] and optical [15] properties, to the best of our knowledge, comprehensive research of mechanical properties of Bi$_2$Se$_3$ has not been reported yet, except for Young's modulus and the hardness of the material investigated in few studies [16, 17]. To understand the usefulness of Bi$_2$Se$_3$ in practical applications, a deep understanding of its mechanical response under loading is crucial. A typical method to evaluate the mechanical properties of advanced technological materials is depth-sensing indentation (DSI). DSI is based on a novel principle in which the deformation is measured instantaneously with the change of the load applied to the surface of the material by the indenter. Traditional micro- and macro-indentation techniques provide a good idea of the response of the material in general, but experiments should continue at a lower scale for more accurate results required for precision applications. Nanoindentation measures the displacement of the material corresponding to the applied load with resolutions of the mN and nM levels. It is the most suitable tool for extracting mechanical properties in terms of elastic modulus and hardness and provides information of load-displacement data locally, therefore...

1 INTRODUCTION

In the last decades, with the high-speed data process demand resulting from the increase in data volume and spreading the use of information technologies to large masses, there is a need for new technology that can process large datasets much faster. The ongoing demand for such technology has required the use of new high-tech materials. Topological insulators (TIs) were recently defined as a new class of materials that exhibit conduction on the surface of the material while insulation occurs beneath the surface [1–3]. TIs allow symmetry-protected surface states to host Majorana particles via spin-locking in order to achieve superconductivity [4]. This unique phenomenon allows the material to contribute to the development of high-tech devices such as spintronics [5, 6] and quantum computers [7]. Bi-chalcogenides such as Bi$_2$Se$_3$ [8] and Bi$_2$Te$_3$ [9] exhibit strong TI property due to their topologically protected surface states. These compounds, which are also thermoelectric materials, are originally narrow-band semiconductor materials having linear dispersion near Fermi energy level, but the research has become extensive since their TI property was discovered [10, 11]. However, since Tellurium is rare, its cost is high and along with its toxicity, it made the use of Bi$_2$Se$_3$ more necessary [12]. In addition to the compatibility with other Bi chalcogenides, high carrier concentration ($>$ 1019 cm$^{-3}$) [13] of Bi$_2$Se$_3$ allows...
the test results are affected minimally from possible defects within the material [18]. The use of nano-indentation experiments is not only limited to measuring the hardness and elastic modulus, but it is also possible to evaluate fracture toughness (\(K_c\)). Fracture toughness is an essential material property as it indicates rapid crack propagation at a critical point where it can lead to subsequent failure of the material, hence being a major design criterion. However, conventional testing methods such as Chevron-notched beam (ISO 24370:2005) and the single-edge notched beam (ASTM STP1419) requires special geometry and complex notches, therefore they are time-consuming and costly. The indentation crack length (ICL) method allows evaluating the \(K_c\) by measuring the crack lengths emanated from corners of the imprint and is very useful for a variety of materials in small scales and thin films/coatings [19, 20]. Guo et al. [21] examined the existing ICL models of various materials such as silicon, soda-lime glass, silicon carbide, sapphire and fused quartz by using nanoindentation, and they revealed that these materials exhibit different crack development mechanisms at the same indentation depths. Bor et al. [22] studied the fracture toughness of supercrystalline materials by comparing indenter types and ICL models from literature and stated that Berkovich indenter presents the most accurate results, and distinct \(K_c\) values were obtained for different models that indicated that the determination of crack mechanism is crucial to obtain correct results. Moreover, Ayatollahi and Karimzadeh [23] reported that \(K_c\) is highly dependent on grain structure and crystal defects by investigating the mechanical properties of biomaterials that are another class of materials in which the samples are difficult to prepare according to standard methods. As can be seen from previous studies, the methodology for investigating fracture toughness can be summarised under three main criteria; material properties, crack mechanism and indenter geometry.

In the present study, nanoindentation measurements were used for the evaluation of the mechanical properties of bulk, three-dimensional, single-crystal Bi\(_2\)Se\(_3\) TI. We report the growth of the single crystal by the Bridgeman–Stockbarger method. The hardness and the elastic modulus in nanoscale were extracted from load-displacement data. The fracture toughness of Bi\(_2\)Se\(_3\) was investigated for the first time. A comprehensive chronology review was made among the ICL models, and the Laugier’s equation was chosen to calculate the \(K_c\) of the material.

2 | EXPERIMENTAL

2.1 | Crystal growth

The two most commonly used methods for growing single-crystal Bi\(_2\)Se\(_3\) are Bridgeman–Stockbarger and Czochralski techniques [24]. While the Czochralski technique provides many advantages, such as producing large crystals in a relatively short time, the use of such a technique in this study should be approached with caution because the growth of the samples with the Czochralski technique can form precipitates, possibly consisting of oxide impurities, on the melt surface [24]. This would directly affect the elasticity, hardness and correlatively the fracture toughness measured by indentation techniques without further surface treatment.

For this reason, single-crystal Bi\(_2\)Se\(_3\) samples were grown by using Bridgeman–Stockbarger method in the present study. The polycrystalline sample synthesised from high purity (99.999%) starting elements was placed in a quartz ampoule with a conical base and sealed under vacuum (10−5 Pa). Before the growth process, the ampoule was held in the hot zone for 12 h for the homogenisation process. At the growth process, the ampoule was transferred from the hot zone (1050 K) to the cold zone (900 K) with the required rate of 1.0 mm/h in order to obtain good quality crystals. The growth specimen was half-pyramidally shaped, therefore it was fixed along-side its cleavage plain in order to obtain (0001) oriented surface. Note that, the authors avoided annealing or any temperature instability at this point of the process because depressions might occur on the surface of the specimen with a fluctuation of ambient temperature [25]. Therefore, Bi\(_2\)Se\(_3\) samples were cleaved at room temperature set by an air-conditioner (21.4°C) in order to avoid any contribution to surface irregularities that could be problematic while undergoing the nanoindentation process.

2.2 | X-ray diffraction (XRD) analysis

XRD analysis was applied to single-crystals Bi\(_2\)Se\(_3\) by using Malvern PANalytical Empyrean diffractometer with Cu-K\(_{\alpha}\) radiation. Continuous scanning with a wavelength of 1.5406 Å at 45 kV and 45 mA, the scan speed for the spectrum was 0.5 min\(^{-1}\) with 2\(^\circ\) range of 5—80 degrees at room temperature (T=21.4°C). Lattice constants of the specimen were evaluated by using HighScore Plus V4.9 software.

2.3 | Scanning electron microscopy (SEM)

Scattering electron microscopy visuals were screened by ThermoFisher QUANTA 450 Field Emission Gun in order to observe the crack formation after indentation and to make the required measurements for fracture toughness calculations.

2.4 | Nanoindentation

In order to obtain the load-displacement curve of the sample, a three-sided pyramidal Berkovich diamond indenter was used in the experiments. The radius of the indenter was 20 nm and the angle between the centreline and the three faces was 65.27 degrees. The cleavage process was carried out immediately before the nanoindentation tests in order to avoid any oxidation. Surfaces were cleaved carefully with a surgical blade and a holder perpendicular to (0001) axis. Experiments were done by
FIGURE 1  Types of crack morphologies developed by indentation (a) radial (Palmqvist) crack, (b) half-penny crack

using a Nano-micro-combi tester by Anton-Paar that was fully calibrated. The applied load started from 1 mN and performed up to 80 mN. Experiments between 1 and 20 mN were investigated in a narrow range in order to observe crack formation and development at ultra-low loads for \( K_c \) evaluations. The elastic modulus and the hardness measurements were made separately using continuous stiffness measurement (CSM) by applying a very low sinusoidal load (0.3 mN, 20 Hertz, 0.01 mN Amplitude) in order to prevent any damage and crack effect. Indentation depth was made to never exceed 10% of specimen thickness in order to prevent the effect of specimen holder [26]. Other parameters to be stated are approach speed and dwell time, 2000 nm/min and 10 s, respectively. In total, 21 indentations were applied on the cleaved surface (0001) of single-crystal Bi\(_2\)Se\(_3\) according to Oliver and Pharr’s classical method [27]. Each experiment was repeated five times to ensure that the results were accurate and in good agreement with each other.

2.5 Fracture toughness model selection

Many equations based on the ICL method were derived [28] since Palmqvist first enounced that fracture toughness can be calculated through indentation imprint [29]. The choice of the most appropriate ICL equation depends on the crack-path configuration under the indentation loads. If brittle materials are to be examined, usually two types of crack patterns are observable, namely, radial (Palmqvist) and half-penny cracks. Figure 1 shows the modality of both crack types. Most of the equations are in the following form as Sebastiani et al. stated [30]:

\[
K_i = \frac{P_{\text{max}}}{\epsilon^{3/2}} \prod \left( \frac{E}{H}, \nu, \psi, \frac{c}{a} \right)
\]  

where \( P \) is the load, \( H \) is the hardness, \( E \) is Young’s modulus, \( K_i \) is the fracture toughness, \( \nu \) is Poisson’s ratio, \( \epsilon \) is the indentation crack length and \( \psi \) is the angle between axis and surface. Considering the radial and lateral cracks that contribute substantially to the morphology of the crack system, in case of half-penny crack systems are present, Lawn et al. suggested the following equation [31]:

\[
\epsilon = \left\{ \phi_r (\cot \psi)^{2/3} \left( \frac{E}{H} \right)^{1/2} \right\}^{2/3} \frac{P}{c^{2/3}}.
\]  

They evaluated \( \phi_r = 0.032 \pm 0.002 \) and \( \psi = 74^\circ \) in their work, thereby the formula on the top where \( \phi_r \) is the independent empirical constant of the indenter-specimen system that can be rewritten as follows:

\[
K_i = 0.0139 \left( \frac{E}{H} \right)^{1/2} \frac{P}{c^{3/2}}.
\]  

The above equation obtained by considering the Vickers indentation tip is found to be valid in cases where \( (c/a \leq 2) \). This restriction may have undesired consequences, such as not getting compatible results while working with lower indentation loads. Subsequently, by working on a number of polycrystalline ceramics, Anstis et al. modified the formula with a new independent material constant \( (X = 0.016 \pm 0.04) \) for Vickers indenters, but the formula is also applicable for Berkovich indenter used experiments. Anstis et al. proposed the following equation [32]:

\[
K_i = X \left( \frac{E}{H} \right)^{1/2} \frac{P}{c^{3/2}}.
\]  

From their work, it is observed that \( \epsilon^{3/2} \) value is independent of \( P \) and highly dependent on the geometry of the indenter and material properties. Thus, changing the experimental constant for different indenter geometries allows the transition from Vickers to Berkovich indenter geometries to achieve more precise results at lower indentation loads. It is beneficial to emphasise that Anstis’ equation is based on the half-penny-shaped
crack configuration where the author investigated the variety of materials such as practical ceramics, embracing monocristalline, polycrystalline, and amorphous solids and covered the $K_c$ ranging between 0.74 and 12 MPa m$^{1/2}$. The author suggested that cracks must be well-developed and fulfil ($c \geq 2a$) criteria in order to keep the validity of the formula. However, Laugier proposed that toughness values obtained by Anstis’ is reliable to some extent but argued that ceramics generally develop in radial (Palmqvist) crack configuration. In this regard, Laugier proposed [33]:

$$K_c = k_P \left( \frac{a}{l} \right)^{1/2} \left( \frac{E}{H} \right)^{2/3} \left( \frac{P}{\sigma_a^{1/2}} \right)$$  \( (5) \)

where $a$ is the distance between the centre and the corner of the imprint, $l$ is the crack length and $k_P$ is the empirical constant for Vickers indenter and defined as 0.015 by Laugier. This empirical constant must be changed for nanoindentation studies where three-sided Berkovich indenter is preferred. In this regard, Dukino and Swain stated that the Berkovich indenter provides more consistent $K_c$ estimates than the Vickers indenter at minimum loads and reformulated the formula with a correction factor in order to make it effectively available to be employed for the Berkovich-used nanoindentation tests [34]. This was done through a small variable and the modified formula for evaluating fracture toughness through Berkovich indenter by Dukino and Swain that becomes as follows:

$$K_c = 1.073 \ k_P \left( \frac{a}{l} \right)^{1/2} \left( \frac{E}{H} \right)^{2/3} \left( \frac{P}{\sigma_a^{1/2}} \right)$$  \( (6) \)

In addition to that, Niihara stated that crack typologies developed in low-load indentations can be distinguished by using $c/a$ and $c/l$ ratios [35]. According to the work, when the crack-to-indent ratio decreases under a certain level ($l/a \leq 2.5$ or $c/a \leq 3.5$), the crack profile is defined as radial (Palmqvist) type, as the values of the ratio increase ($c/a \geq 3.5$) the crack profile defined as a half-penny type. Another criterion for defining the crack morphology through ICL was set by Schneider et al. [36] From their work, it is concluded that radial cracks develop at low-indentation loads where ($c/a \leq 2$), and this criterion is independent of the indenter type.

Consequently, a comparison of the different ICL equations was made, and for the fracture toughness analysis of the Bi$_2$Se$_3$ crystal, the Laugier’s model modified by Dukino and Swain was selected by using the Berkovich indenter tip.

3 | RESULTS

3.1 | XRD

The recorded XRD pattern shown in Figure 2 verified that Bi$_2$Se$_3$ crystal formed as single crystal having hexagonal rhombohedral lattice structure that belongs to R$ar{3}$m space group. The lattice dimensions of the single-crystal Bi$_2$Se$_3$ were determined as $a = 4.13$ Å, $b = 4.13$ Å and $c = 28.6$ Å by using HighScore Plus V4.9 software.

XRD patterns demonstrate that the diffraction peaks orderly positioned in (003 n) format as observed from major sharp (003), (006), (0015), (0018), (0021) peaks together with some minor (015), (110) peaks and this indicates that the single-crystal Bi$_2$Se$_3$ is mainly grown along the c-axis.

3.2 | Nanoindentation

A series of preliminary tests at various load levels were conducted to determine the most suitable model required to evaluate the fracture toughness. The cracks observed in all load ranges meet Niihara’s ($l/a \leq 2.5$ or $c/a \leq 3.5$) and Schneider’s ($c/a \leq 2$) criteria for radial cracks. Therefore, Laugier's
model was suitable and selected for this study. Twenty-one nano-indentation experiments were conducted with increasing loading with increasing intervals. Measurements were made at 1 mN intervals between 0 and 5 mN, 1–2 mN between 5 and 20 mN, 5 mN between 20 and 50 mN and 10 mN intervals between 50 and 80 mN. The reason for performing the experiments in this way is explained in Section 2. An SEM image of cracks emanated from the corner of the triangular imprint at 3 mN load applied by Berkovich indenter as shown in Figure 3. It can be seen from the figure that well-developed radial cracks appeared from the corner of the imprint even at very low loads.

The load-displacement curve reflects the material’s deformation at applied load during the indentation process quantitatively, and elastic recovery rates show the material’s ability to regain its form after plastic deformation. The overall load-displacement curve of the specimen under varying loads between 1 to 80 mN is shown in Figure 4(a). It would be appropriate to divide the load-displacement curves between 1–15 mN and 18–80 mN into two as shown in Figures 4(b) and (c), respectively, in order to demonstrate the behaviour of the material better at increasing loads and avoid confusion. Elastic recovery rates are also shown in Figure 5.

As can be clearly seen from Figures 4(a)–(c), discontinuities in the form of long plateaus occurred in the load-displacement curve. This unique phenomenon that occurs in small scale measurements is known as the ‘pop-in’ event. Pop-ins can be formed as a result of multiple issues such as the nucleation of dislocations, sudden phase transformation, or fracture with crack initiation and propagation. The number of the pop-ins tends to increase dependently with the increase of the applied load as a trigger indicating the start of plastic deformation due to growing nucleation activity. But in contrast, the numbers of ‘pop-in’ events decreased with increasing load in the present study.

While a lot of pop-ins were observed between 1 and 15 mN (Figure 4(b)), it gradually decreased at loads higher than 18 mN (Figure 4(c)). This is because irregularities decrease in single crystals as one goes deeper than the sample surface with increasing loads. To be more specific, inevitable rapid oxidation after cleaving and surface roughness contributes to pop-in events to a certain nanometer level from the surface. The elastic modulus of single-crystal Bi$_2$Se$_3$ was measured by CSM and evaluated by Oliver and Pharr’s standard method [27] of the following formula:

$$E = \frac{E_i \left(1 - \nu^2\right)}{20 \sqrt{A_i E_i}} \left(1 - \nu_i^2\right)$$

where $E$ is the elastic modulus of the sample, $E_i$ is the elastic modulus of the indenter ($= 1141$ GPa for Berkovich), $A_i$ is the contact area between the sample and the indenter, $\nu$ is the Poisson ratio of the sample ($= 0.27$ for Bi$_2$Se$_3$), $\eta$ is a specific geometry dependent constant of the indenter ($= 1.034$ for Berkovich), $\nu_i$ is the Poisson ratio of the indenter ($= 0.07$ for Berkovich), and $S$ is the stiffness of the sample material. As shown in Figure 6(a), the elastic modulus of single-crystal Bi$_2$Se$_3$ decreases after the first increase and then stabilises as the indentation depth increases. The stable value evaluated to be approximately 6.018 GPa.

Nanohardness was calculated by the ratio of applied load ($P$) to contact area as shown in the equation below:

$$H = \frac{P}{A_i}$$

The relationship between nanohardness and indentation depth is shown in Figure 6(b), which indicates that nanohardness decrease and tend to stabilise ultimately as the indentation depth increase. The stable value is evaluated to be approximately 323 MPa. The measured value for nanohardness was slightly lower than that previously reported for the Bi$_2$Se$_3$ bulk sample [16]. This difference may be due to the fact that the tested materials were grown by different crystal growth methods. In particular, samples analysed by Gupta et al. were grown by the Vertical–Bridgeman method where they held the melt in the
hot zone for 6 h for the homogenisation process, while in the present work the ampoule was held the hot zone for 12 h in order to minimise the imperfections. However, the difference was low (≈26%) and the elastic modulus values obtained from the two studies were in good agreement.

In addition, comparison with the works of Lai et al. [17] and Yan et al. [38] may not be appropriate since they produced the Bi₂Se₃ sample as a polycrystalline thin film, whereas in the present work, we analysed high-quality single crystals grown by Bridgeman–Stockbarger method. A detailed comparison of the present work with works in literature is shown in Table 1.

For each imprint, c/a and l/a values were calculated, and Figures 7(a) and (b) show the distribution with increasing load. Critical values where (c/a ≤ 3.5) and (l/a ≤ 2.5) for Niihara and (c/a ≤ 2.5) for Schneider are shown with a dashed line. None of the values exceeds the critical values for c/a and l/a described by Niihara and Schneider. Therefore, the selection of the Laugier’s equation was right as the preliminary tests suggested.

It is important to remind that both criteria were based on ICL methods and selection of the Laugier’s methods validity by considering Niihara’s and Schneider’s criteria was only viable in mode I. As shown in Figure 7(b), although the c/a ratios were
very close to 2.5, once stabilisation was achieved, $c/a$ and $l/a$ values never exceeded the critical levels. Together, the present findings confirm that the calculated value of $K_c$ was obtained approximately 0.034 MPa m$^{-1/2}$. The calculated values of the $K_c$ were then compared with Bi$_2$Te$_3$ [39], which is another TI chalcogenide of Bi with the same quintuple-layer structure. The results showed that $K_c$ of Bi$_2$Se$_3$ was found to be 19% lower than $K_c$ of Bi$_2$Te$_3$ (0.042 ± 0.016 MPa m$^{-1/2}$). Other differences in mechanical behaviours between these Bi-chalcogenide TIs were also noteworthy. To explain, in Bi$_2$Te$_3$ single-crystal TI, cracks started to develop after 4 mN, while in the present work, cracks were observed even in the lowest applied load, which is 1 mN, and $c/a$ ratios of Bi$_2$Te$_3$ are much higher than the Bi$_2$Se$_3$. From Figure 8, one can see that the cracks were well-developed under 1 mN load and the $c/a$ ratio was around 2. These differences between two TIs with the same quintuple-layer structure.
TABLE 1  Summary of mechanical properties of Bi₂Se₃ found in the literature and the present study

| Sample preparation method | Young’s modulus (GPa) | Hardness (GPa) | Fracture toughness (MPa m¹/₂) | Author |
|---------------------------|-----------------------|----------------|-------------------------------|--------|
| Single-crystal growth by vertical Bridgman technique (VBT) | 6.361 | 0.437 | – | Gupta et al. [16] |
| Thin film synthesised by van der Waals epitaxy method | 20.67 | – | – | Yan et al. [38] |
| Thin-film deposition on Al substrate | 58.6 | 2.1 | – | Lai et al. [17] |
| Single-crystal growth by Bridgeman–Stockbarger method | 6.018 | 0.323 | 0.034 | Present study |

FIGURE 7  (a) ½/a, (b) ½/a, and (c) ½ values of single-crystal Bi₂Se₃ evaluated by using the Laugier’s method
The fracture toughness of single-crystal Bi$_2$Se$_3$ TI grown by using the Bridgeman–Stockbarger method was measured for the first time. The evaluated value of $K_c$ by using the Laugier’s model was 0.034 MPa m$^{1/2}$. The Young’s modulus and the hardness of the TI were obtained as 6.018 GPa and 323 MPa, respectively, and the elastic recovery was approximately 60%. These values, which are lower than other materials used in nanoelectromechanical systems such as Bi$_2$Te$_3$ [32], GaAs [33] and graphene [34], can make it difficult to use Bi$_2$Se$_3$ in practical applications. Further, processing of Bi$_2$Se$_3$ by known microfabrication methods will be significantly challenging. Therefore, improvement in the mechanical properties of Bi$_2$Se$_3$ without compromising its electrical properties is necessary in order to utilise the material in prospective high-tech applications.

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REFERENCES

1. Kane, C.L., Mele, E.J.: $Z_2$ topological topological order and the quantum spin hall effect. Phys. Rev. Lett. 95(146802), 3–6 (2005)
2. Fu, L., et al.: Topological insulators in three dimensions. Phys. Rev. Lett. 98(106803), 1–4 (2007)
3. Hasan, M.Z., Kane, C.L.: Colloquium: Topological insulators. Rev. Mod. Phys. 82(4), 3045–3067 (2010)
4. Zhang, H., et al.: Topological insulators in Bi$_2$Se$_3$, Bi$_2$Te$_3$ and Sb$_2$Te$_3$ with a single Dirac cone on the surface. Nat. Phys. 5(6), 438–442 (2009)
5. Mellnik, A.R., et al.: Spin-transfer torque generated by a topological insulator. Nature 511(7510), 449–451 (2014)
6. Wang, Y., et al.: Room temperature magnetization switching in topological insulator-ferromagnet heterostructures by spin-orbit torques. Nat. Commun. 8(1), 6–11 (2017)
7. Jauregui, L.A., et al.: Magnetic field-induced helical mode and topological transitions in a topological insulator nanoribbon. Nat. Nanotechnol. 11(4), 345–351 (2016)
8. Butch, N.P., et al.: Strong surface scattering in ultrahigh-mobility Bi$_2$Se$_3$ topological insulator crystals. Phys. Rev. B: Condens. Matter 81(24), 3–6 (2010)
9. Zhang, W., et al.: First-principles studies of the three-dimensional strong topological insulators Bi$_2$Te$_3$, Bi$_2$Se$_3$ and Sb$_2$Te$_3$. New J. Phys. 12 (2010)
10. Zhang, J., et al.: Raman spectroscopy of few-quintuple layer topological insulator Bi$_2$Se$_3$ nanoflakes. Nano Lett. 11(6), 2407–2414 (2011)
11. Zhang, G., et al.: Quintuple-layer epitaxy of high-quality Bi$_2$Se$_3$ thin films for topological insulator. Appl. Phys. Lett. 95, 053114 (2009)
12. Woodhouse, M., et al.: Supply-chain dynamics of tellurium, indium, manufacturing costs. IEEE J. Photovoltaics 3(2), 833–837 (2013)
13. Park, B.C., et al.: Terahertz single conductance quantum and topological phase transitions in topological insulator Bi$_2$Se$_3$ ultrathin films. Nat. Commun. 6, 6552 (2015)
14. Luo, X., et al.: First-principles investigations of the atomic, electronic, and thermoelectric properties of equilibrium and strained Bi$_2$Se$_3$ and Bi$_2$Te$_3$ including van der Waals interactions. Phys. Rev. B: Condens. Matter 86(18), 184111 (2012)
15. Sharma, A., et al.: High performance broadband photodetector using fabricated nanowires of bismuth selenide. Sci. Rep. 6(January), 1–8 (2016)
16. Gupta, S., et al.: Enhancement of thermoelectric figure of merit in Bi$_2$Se$_3$ crystals through a necking process. J. Appl. Crystallogr. 48, 533–541 (2015)
17. Lai, H., et al.: Nanoindentation of Bi$_2$Se$_3$ thin films. Micromachines 5(11), 17–20 (2018)
18. Broitman, E.: Indentation hardness measurements at macro-, micro-, and nanoscale: A critical overview. Tribol. Lett. 65(1), 1–18 (2017)
19. Karimzadeh, A., et al.: Assessment of nano-indentation method in mechanical characterization of heterogeneous nanocomposite materials using experimental and computational approaches. Sci. Rep. 9(1), 1–14 (2019)
20. Sergejev, F., Antonov, M.: Comparative study on indentation fracture toughness measurements of cemented carbides. Est. J. Eng. 12(4), 388–398 (2006)
21. Guo, Y., et al.: A detailed analysis of the determination of fracture toughness by nanoindentation induced radial cracks. J. Eur. Ceram. Soc. 40(2), 276–289 (2020)
22. Bor, B., et al.: Nanoindentation-based study of the mechanical behavior of bulk supercrystalline ceramic-organic nanocomposites. J. Eur. Ceram. Soc. 39(10), 3247–3256 (2019)
23. Ayatollahi, M.R., Karimzadeh, A.: Nano-indentation measurement of fracture toughness of dental enamel. Int. J. Fract. 183(1), 113–118 (2013)
24. Tonn, J., et al.: Czochralski growth of lead iodide single crystals through a necking process. J. Cryst. Growth 318(1), 276–289 (2010)
25. Cavallin, A., et al.: Preparation and characterization of Bi$_2$Se$_3$(0001) and of epitaxial FeSe nanocrystals on Bi$_2$Se$_3$(0001). Surf. Sci. 646, 72–82 (2016)
26. Karimzadeh, A., Ayatollahi, M.R.: Investigation of mechanical and tribological properties of bone cement by nano-indentation and nano-scratch experiments. Polym. Test. 31(6), 828–833 (2012)
27. Oliver, W.C., Pharr, G.M.: Measurement of hardness and elastic modulus by instrumented indentation: Advances in understanding and refinements to methodology. J. Mater. Res. 19(1), 3–20 (2004)
28. Miyazaki, H., Yoshizawa, Y.: A Reinvestigation of the validity of the indentation fracture (IF) method as applied to ceramics. J. Eur. Ceram. Soc. 37(15), 4437–4441 (2017)
29. Palmqvist, S.: Method att bestamma segheten hos sproda material, sarskilt hardmetaller. Jernkontorets Ann. 141, 303–307 (1957)
30. Sebastiani, M., et al.: Measurement of fracture toughness by nanoindentation methods: Recent advances and future challenges. Curr. Opin. Solid State Mater. Sci. 19, 324–333 (2015)
31. Lawn, B.R., Fuller, E.R.: Equilibrium penny-like cracks in indentation fracture. J. Mater. Sci. 10(12), 2016–2024 (1975)
32. Anstis, G.R., et al.: A critical evaluation of indentation techniques for measuring fracture toughness: I, direct crack measurements. J. Am. Ceram. Soc. 64(9), 533–538 (1981)
33. Laugier, M.T.: Palmqvist indentation toughness in WC-Co composites. J. Mater. Sci. Lett. 6(8), 897–900 (1987)
34. Dukino, R.D., Swain, M.V.: Comparative measurement of indentation fracture toughness with berkovich and vickers indenters. J. Am. Ceram. Soc. 75(12), 3299–3304 (1992)
35. Niihara, K., et al.: Evaluation of KIc of brittle solids by the indentation method with low crack-to-indent ratios. J. Mater. Sci. Lett. 1(1), 13–16 (1982)
36. Schneider, G.A., Fett, T., Computation of the stress intensity factor and COD for submicron sized indentation cracks. J. Ceram. Soc. Japan 114(1335), 1044–1048 (2006)
37. Koc, H., Ozsik, H., Deligöz, E.: Mechanical, electronic, and optical properties of Bi2S3 and Bi2Se3 compounds: First principle investigations. J. Mol. Model. 20, 2180 (2014)
38. Yan, H., et al.: Elastic behavior of Bi2Se3 2D nanosheets grown by van der Waals epitaxy. Appl. Phys. Lett. 109(3), (2016)
39. Lamuta, C., et al.: Indentation fracture toughness of single-crystal Bi2Te3 topological insulators. Nano Res. 1, 1–11 (2016)

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