Materials Letters 344 (2023) 134402

Contents lists available at ScienceDirect

Materials Letters

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

Friction and wear characteristics of bismuth selenide topological insulator

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ARTICLE INFO

Keywords:
Bi_2Se_3
Crystal growth
Superconductors
Scratch
Mechanical properties

ABSTRACT

Single crystal Bi_2Se_3 was grown using the novel Bridgeman-Stockbarger method. Surface characteristics in terms of wear rate (*W*), wear coefficient (*K*), coefficient of friction (*COF*), and strain hardening exponent (*n*) were investigated using nanoindentation and nano scratch techniques. Our results revealed that the remarkable wear properties of single-crystal Bi_2Se_3 are promising for nanoscale design of topological insulators.

1. Introduction

In recent years, the demand for high-speed data processing has emerged because of the spread of digitalization and the increase in data volume. Therefore, new materials are needed to transmit these data faster as datasets grow. Topological insulators (TIs) are a new group of materials that can transmit data faster thanks to their unique spin–orbit coupling system [1]. Bismuth selenide (Bi_2Se_3), a traditional semiconductor, has rapidly increased in popularity in recent years due to the discovery of TI properties that provide exceptional conductivity [2]. Its superior conduction properties make it an essential potential candidate for quantum computers [3] and optoelectronics [4]. However, the remarkable advances in understanding the electronic properties of Bi_2Se_3-based materials contrast with the understanding of the mechanical properties of Bi_2Se_3. An incomplete understanding of the material's mechanical properties may pose a problem for the design and limit its use in new high-tech applications. To date, the mechanical properties of Bi_2Se_3, such as hardness, Young's modulus, and fracture toughness [5], have been comprehensively examined. However, the tribological properties of the material are still lacking, which is of great importance because the superior electrical properties are strongly dependent on surface integrity and crystallographic quality [6].

In this study, the wear and friction properties of single-crystal Bi_2Se_3 in terms of wear volume, wear rate, and coefficient of friction were analyzed for the first time using the nanoscratch technique. In addition, the strain hardening exponent was evaluated with a novel method based on pile-up width using nanoindentation tests. This study aims to understand the wear behavior of single-crystal Bi_2Se_3 for design purposes, whose excellent electronic properties are still being investigated.

2. Experimental

Bulk Bi_2Se_3 single-crystal was grown using the Bridgeman-Stockbarger method. To start the crystal growth process, polycrystalline Bi and Se samples having a high purity ratio (99.999%) were placed into a conical-based quartz crucible and sealed under a vacuum. The crucible was heated at a constant temperature (1050 K) for 12 h before the melt was transferred to the cool zone (900 K). The transfer speed was set to 1 mm/h to obtain a good-quality crystal.

Nano scratch tests were performed in either constant or ramping load modes. In the case of continuous load mode, the samples were penetrated until the scratch load was achieved and then scratched along the scratch distance. In the ramping load mode, the load was continuously increased along the scratch distance until the scratch load was reached. Tests were conducted from 500 μN to 2500 μN with 500 μN intervals. A standard Berkovich tip with a tip radius of 82 nm and a half-angle $\theta = 65.27^\circ$ was used in all tests using the Hystrion Triboindenter (TI-950). Scratch speed and scratch distance were set to 0.2 μm/s and 10 μm for all tests. To evaluate the stress hardening exponent (*n*), five indentation loads from 5000 μN to 9000 μN with 1000 μN intervals were applied to the sample's surface. Each testing condition was repeated five times to ensure reliability.

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https://doi.org/10.1016/j.matlet.2023.134402
Received 30 January 2023; Received in revised form 23 March 2023; Accepted 17 April 2023
Available online 23 April 2023

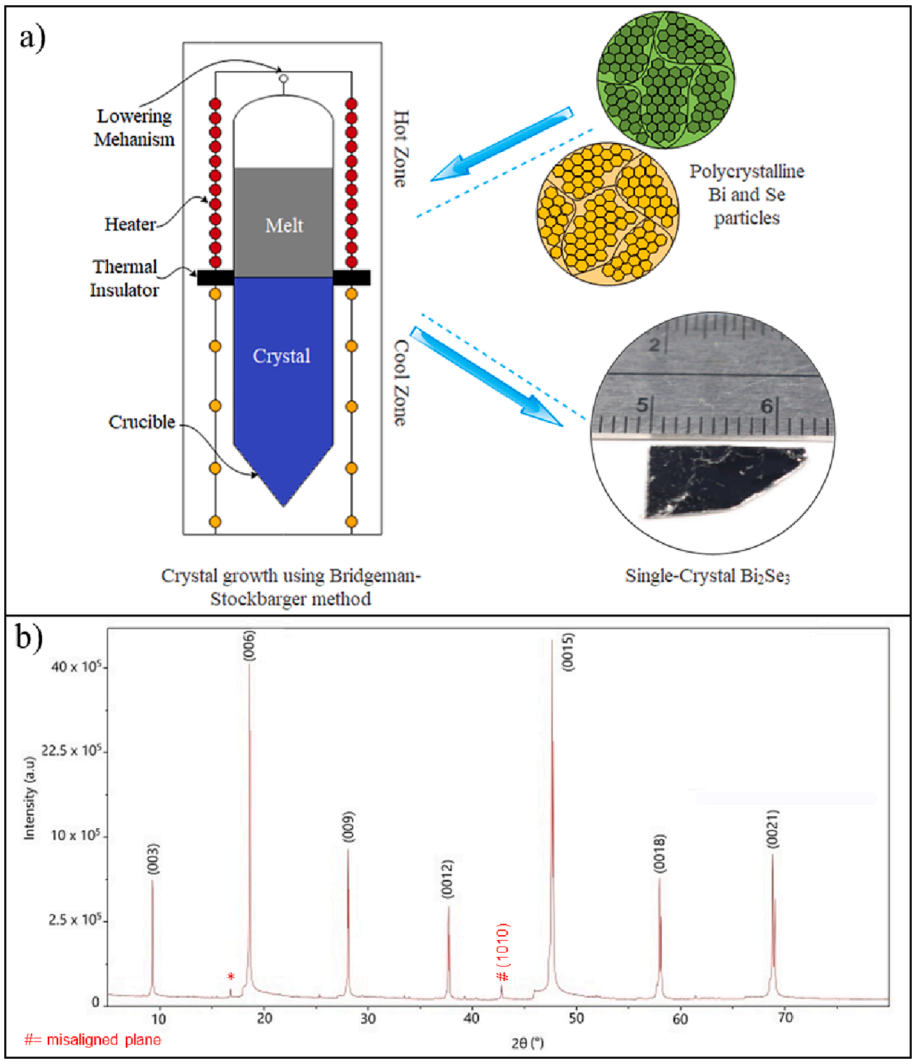

Fig. 1. (a) Schematic representation of single crystal growth of Bi_2Se_3 (b) XRD patterns of the grown sample.

3. Results & discussion

The crystal growth process and X-Ray diffraction (XRD) patterns are illustrated in Fig. 1(a) and 1(b), respectively. Highly indexed peaks with (00l) directions reflect the single crystalline structure of the sample. It is also observed that there are small peaks around 17° and 43.5°. The first of these peaks is unidentified, and the second is due to a misaligned Bi_2Se_3 plane. Fig. 2(a) shows the normal displacement and normal force versus lateral displacement at loads ramping from 0 to 1500 μN. The normal displacement measured during testing increased linearly with the increase in lateral displacement, eventually reaching 2607 nm, while the post-scratch depth of the compound slightly increased along with the scratch distance and was recorded as 166.04 nm at its maximum. In other words, the compound regained 93.63 ± 1.5% of its initial volume after being subjected to plastic deformation due to scratching. A linear increase in the normal force followed by a linear increase in instantaneous scratch depth suggests the tip encountered a slight resistance. A side view of the surface at ramp load increasing from 0 to 1500 μN is presented as an inset to Fig. 2(b), and plastic deformation is present with the onset of the normal force.

Fig. 2(b) illustrates the COF versus normal load. COF tends to stabilize after the initial rapid decline as the normal load increases. The rapid initial decrease could be attributed to adhesive forces that significantly increase the frictional forces between the tip and the specimen. COF values are even higher than one in this test stage because normal force is too low to produce plastic deformation. In the steady-state phase, some fluctuations and spikes are visible. This is due to the stick-and-slip motion within the specimen. In the stick phase, the movement of the indenter tip is constrained by surrounding Bi_2Se_3 piles. Therefore, the lateral force increases due to the resistance of piles. As the normal force increases to the critical point, the lateral force increases too and starts to push the piles in the scratch direction, and the slip phase takes place. Lateral force drops sharply in the slip phase causing spikes in the COF-Normal load graph, which can be seen in Fig. 2(b).

Fig. 3(a), 3(b), and 3(c) represent the three-dimensional and side views of the scratch trace of single crystal Bi_2Se_3 under 1500 μN constant load test, respectively. From Fig. 3(a), it was observed that a V-shaped groove was formed on the sample surface due to the pyramidal shape of the Berkovich tip, and accumulation of pile-ups is visible on the surfaces on both sides of the groove. The large pile-ups are due to the material pile movement towards the surface due to the initial immersion of the penetrating tip and plowing. Irregular stripes consisting of slip lines appear on the groove walls due to the stick and slip motion of the scratching process. Cross-slip events on groove walls where dislocations move from one slip line to another, suggesting that the dislocations are strong enough to overcome plane obstacles by changing the slip lines. Fig. 3(b) and 3(c) present the side views of the scratch trace in the x-y and y-z directions, respectively. As can be seen, the residual scratch depth is recorded as 198.05 nm along the scratch length.

Pile-ups around the scratch tracks indicate the material's ductility,

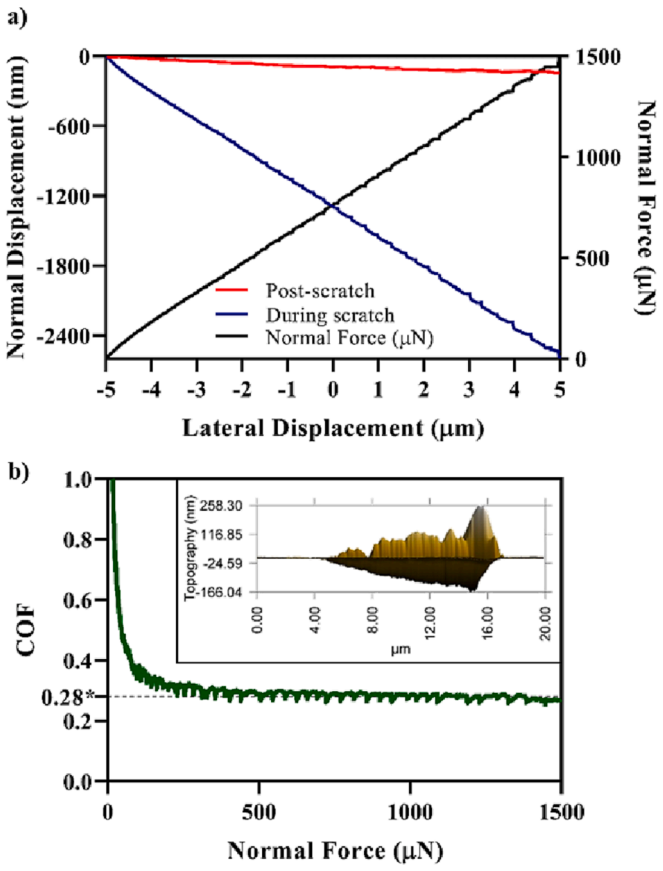

Fig. 2. (a) Surface profiles of single-crystal bismuth selenide (b) COF as a function of normal load.

thus its formability. To examine these pile-ups, strain hardening was measured. To assess the strain hardening of single-crystal Bi_2Se_3, a novel method based on the ratio of pile heights proposed by Das et al. [7] is used. According to the method, the strain hardening exponent, *n,* is calculated using the known radius of a cono-spherical tip and the radius of pile-up around the imprint using the following equation;

$$c^2 = \frac{a^2}{a^{*2}} = \frac{5(2-n)}{2(4+n)} \tag{4}$$

where c is a constant, a is the actual contact (pile-up) radius, and a^* is the ideal contact (without pile-up) radius. According to the strain hardening coefficient classification, the value of n varies between 0 and 1, values close to 1 indicate that the material has predominantly elastic behavior, and values close to 0 indicate that the material has more plastic behavior. Using the above equation, $n = 0.61 \pm 0.07$ was found for single-crystal Bi_2Se_3, reflecting the elastoplastic behavior.

Fig. 3(d) and 3(e) show that prominent pile-ups are generated around the imprint due to 8000 μN normal load. The normal displacement of the imprint is measured as 127.96 nm, whereas the instantaneous scratch depth is recorded as 670.38 nm for the same experiment. We can confirm that the material regains most of its initial volume after deformation. The generation of large pile-ups and the high recovery rate of initial volume indicates that the single-crystal Bi_2Se_3 possesses both ductile and elastic properties to a large extent.

Wear rate (W) is a good indicator of the materials' scratch resistance and defined as volume loss (V) per distance (L). V can be calculated as follows;

$$V = \int_{-l/2}^{l/2} A(x)dx \tag{1}$$

here A is the cross-sectional area of the trace. Archard's equation [8] for dry wear can be employed as follows;

$$V = \frac{KF_n}{H}L \tag{2}$$

Here K represents the wear coefficient which indicates the probability of material removal, F_n is the normal force applied on the surface, and H is the hardness. Therefore, W can be expressed as;

$$W = \frac{KF_n}{H} \tag{3}$$

In Fig. 4(a), the removed volume is plotted against the scratch length at loads varying from 500 μN to 2500 μN. It can be seen from the figure that the removed volume increased linearly as the scratch length increased for all loadings. To find the wear rates, the slope of the *V-L* curves from Fig. 4(a) was taken, and results are presented against the ratio of applied normal force versus hardness in Fig. 4(b). K is found to be 0.0623 by the slope of the curve in Fig. 4(b) according to Eq. (3).

4. Conclusions

In summary, Bi_2Se_3 topological insulator grown with the Bridgeman-Stockbarger technique recovers most of its original volume once

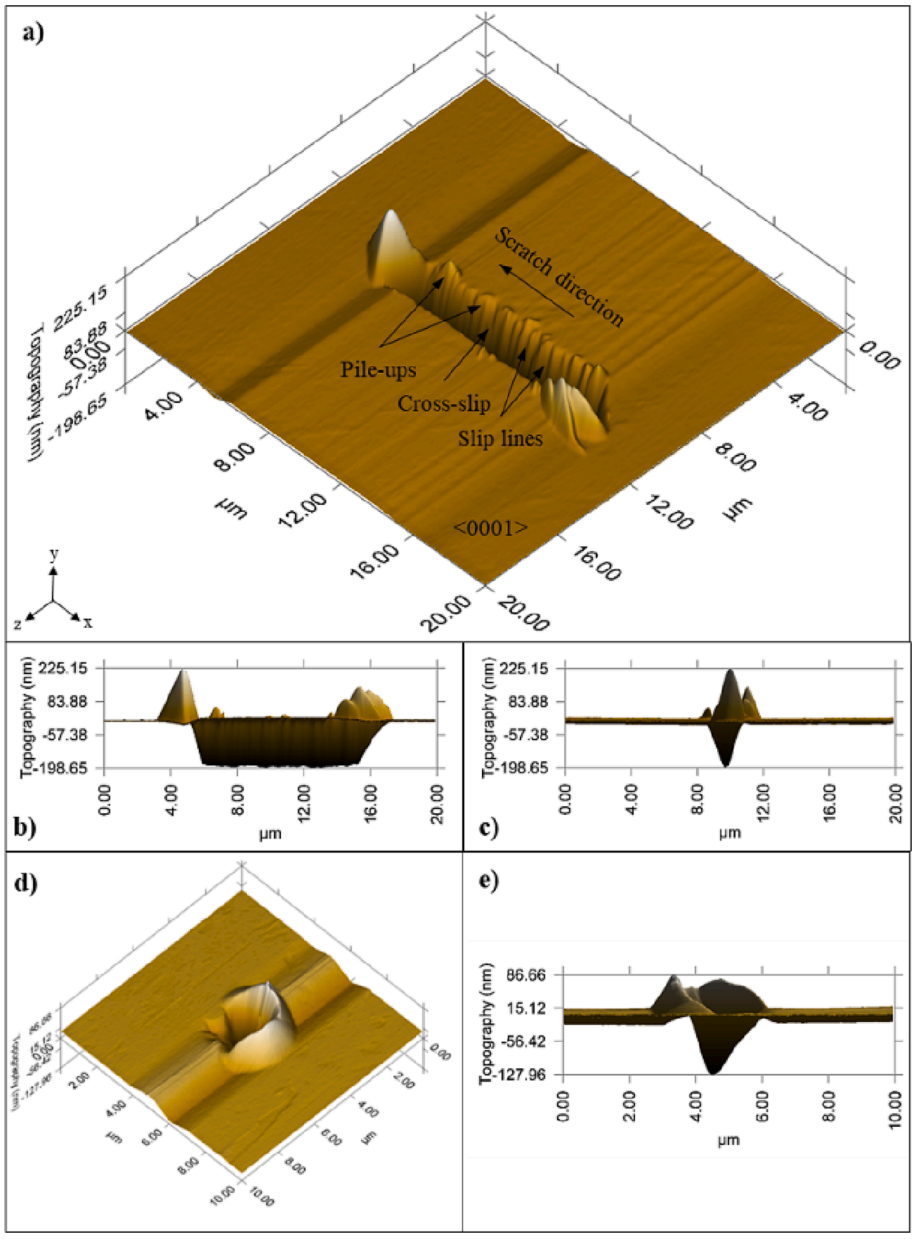

Fig. 3. SPM images of the (a-c) post-scratch surface at 150 μN and (d-e) indentation imprint at 8000 μN.

subjected to abrasion, as suggested by the volume recovery rate of 93.63%. COF is found to be 0.28, which is very close to that of GaN (≈0.27) [9]. *K* is found as 0.0623. *n* is found as 0.61 ± 0.07, slightly higher than polycrystalline Cu (0.456) [10]. Based on the results obtained in this work, we can conclude that single-crystal Bi_2Se_3 has competitive friction and wear properties in addition to its superior electronic properties, when compared to conventional flexible electronics. Our results revealed that these superior wear resistance properties of single-crystal Bi_2Se_3 can promote its use in future electronic systems.

CRediT authorship contribution statement

Utku Uzun: Conceptualization, Methodology, Writing – original draft, Investigation, Visualization, Methodology, Validation. **Caterina Lamuta:** Writing – review & editing, Resources, Supervision. **Mehmet Yetmez:** Resources, Supervision.

Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Utku Uzun reports financial support was provided by The Scientific and Technological Research Council of Turkey.

Data availability

Data will be made available on request.

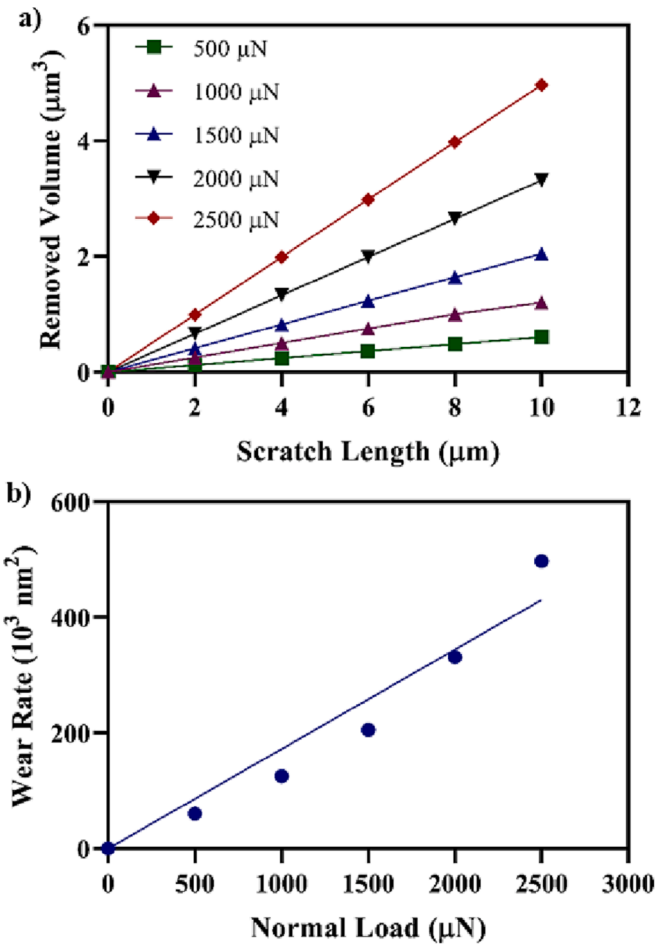

Fig. 4. (a). Scratch volume against scratch length for five different normal loads (b) wear rate against normal load for single-crystal Bi_2Se_3.

References

[1] C.L. Kane, E.J. Mele, Phys. Rev. Lett. 95 (146802) (2005) 3–6, https://doi.org/10.1103/PhysRevLett.95.146802.
[2] W. Zhou, H. Zhu, J.A. Yarmoff, Phys. Rev. B 94 (19) (2016) 1–10, https://doi.org/10.1103/PhysRevB.94.195408.
[3] L.A. Jauregui, M.T. Pettes, L.P. Rokhinson, L. Shi, Y.P. Chen, Nat. Nanotechnol. 11 (4) (2016) 345–351, https://doi.org/10.1038/nnano.2015.293.
[4] H. Xie, et al., Small 12 (30) (2016) 4136–4145, https://doi.org/10.1002/smll.201601050.
[5] U. Uzun, M. Yetmez, N. Akıncı, Micro Nano Lett. 16 (3) (2021) 203–212, https://doi.org/10.1049/mna2.12025.
[6] J.J. Cha, K.J. Koski, Y. Cui, Phys. Status Solidi – Rapid Res. Lett. 7 (1–2) (2013) 15–25, https://doi.org/10.1002/pssr.201206393.
[7] G. Das, S. Ghosh, S. Ghosh, R.N. Ghosh, Mater. Sci. Eng. A 408 (1–2) (2005) 158–164, https://doi.org/10.1016/j.msea.2005.07.026.
[8] J.F. Archard, J. Appl. Phys. 24 (8) (1953) 981–988, https://doi.org/10.1063/1.1721448.
[9] G. Zeng, C.K. Tan, N. Tansu, B.A. Krick, Appl. Phys. Lett. 109 (5) (2016) pp, https://doi.org/10.1063/1.4960375.
[10] B.M. Kim, C.J. Lee, J.M. Lee, J. Mech. Sci. Technol. 24 (1) (2010) 73–76, https://doi.org/10.1007/s12206-009-1115-8.