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Proceeding Paper

Fabrication of 2-D Nanosheets of $NbSe_2$ via Liquid Phase Exfoliation and Their Morphological, Structural, and Optical Characterization †

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Abstract: The present work is on the synthesis and investigation of the structural, optical, and optoelectrical properties of $NbSe_2$ as an efficient material for energy conversion applications. The liquid phase exfoliation method was employed for the synthesis of 2D nanosheets from the bulk $NbSe_2$ at different exfoliation levels. SEM was used to confirm the physical dimensions of the nanosheets, while XRD was used to verify the structural retention of hexagonal nanosheets. The results demonstrate that high-quality, single-crystalline $NbSe_2$ nanosheets with a size of ≈1 μm in the lateral dimension and ≈6–12 nm thick were obtained. The 2D nanosheets will be further explored for energy storage and conversion applications.

Keywords: 2D transition metal dichalcogenides; liquid exfoliation method

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1. Introduction

For decades, 2D graphene has played a vital role, but due to its zero band gap, its use is restricted in logic devices [1]. Recently, 2D Transition Metal Dichalcogenides (TMDs) have been thoroughly studied for their fascinating characteristics [2–5]. $NbSe_2$, MoS_2, and NbS_2 2D materials are the focus of recent research due to their unique physical and chemical properties compared to their bulk counterparts [6]. In the science of electrochemistry, TMDs provide a viable alternative to photovoltaics as counter electrodes in solar cells [7]. TMDs are semiconductors of the MX_2 type, where M is an atom of a transition metal and X is an atom of a chalcogen [8]. The TMD family includes superconductors [9] and semiconductors [10]. The unique properties of TMD 2D materials have been widely utilized for diverse applications, such as in catalysis, energy storage devices, flexible and transparent electrical and optoelectronic devices, and high-performance sensors [1,6–8].

Among all 2D materials, $NbSe_2$ is novel because of its unique combination of charge density wave transitions and superconductivity [11–13]. $NbSe_2$ is a 2D metal that naturally conducts electricity at a temperature of about 7.2 K [11]. $NbSe_2$ has a single-layer thickness of 0.6 nm; these layers are further stacked together by weak van der Waals forces and can be exfoliated down into thin 2D layers [12]. The novel $NbSe_2$ nanosheets have been less explored relative to other TMDs. The physical, chemical, and mechanical characteristics of $NbSe_2$ make it ideal for use in composites, electronics, photonics, and energy storage [13].

The actual implementation of this material frequently involves overcoming manufacturing, stability, and device integration difficulties. As the subject of 2D materials science develops, researchers continue to investigate and create new uses for $NbSe_2$ [14]. To the best of our knowledge, very few articles on $NbSe_2$ have been reported. This article can contribute to future technologies and applications, ranging from quantum computing to electronics.

2. Synthesis of 2D $NbSe_2$ Nanosheets

$NbSe_2$ 2D nanosheets were prepared via a liquid-phase exfoliation procedure in which 1.6 g of bulk $NbSe_2$ powder was dispersed in 8.0 mL of N-methyl-2-pyrrolidone (NMP) and stirred for 30 min. Later, the solution was probe-sonicated for 6 h with a power of 520 Watts (S 80%) and 390 Watts (S 60%). The exfoliated samples were centrifuged at RPM 500, filtered, and dried at 70 °C in an oven overnight.

3. Results and Discussion

3.1. X-Ray Diffraction (XRD)

The XRD patterns (Figure 1) match the standard JCPDS 65-7464 [15–17]. The (004) peak is significantly prominent in all patterns. However, the bulk sample exhibits the highest peak intensity due to the presence of multiple layers in this plane. As $NbSe_2$ is exfoliated by the probe sonicator, a significant number of layers are eliminated, as seen by the (004) plane's absence of XRD intensity in S 60% and S 80%. The detailed XRD signature from 29 to 56 2 theta values exhibits peaks at (101), (102), (104), (006), and (101), representing the perfect hexagonal crystal structure (inset of Figure 1). The (101) and (110) peaks are noticeable in the bulk sample, whereas the (104) peak is clearer in S 60% and S 80%. The blue shift is seen in the (104) and (006) planes at 2θ. The blue shift indicates a reduction in the number of layers, which creates stresses and defects in the $NbSe_2$ crystal lattice. The rearrangement of the Nb and Se planes in the lattice changed the d spacing in the respective planes. The other planes do not show any significant changes in their 2θ values.

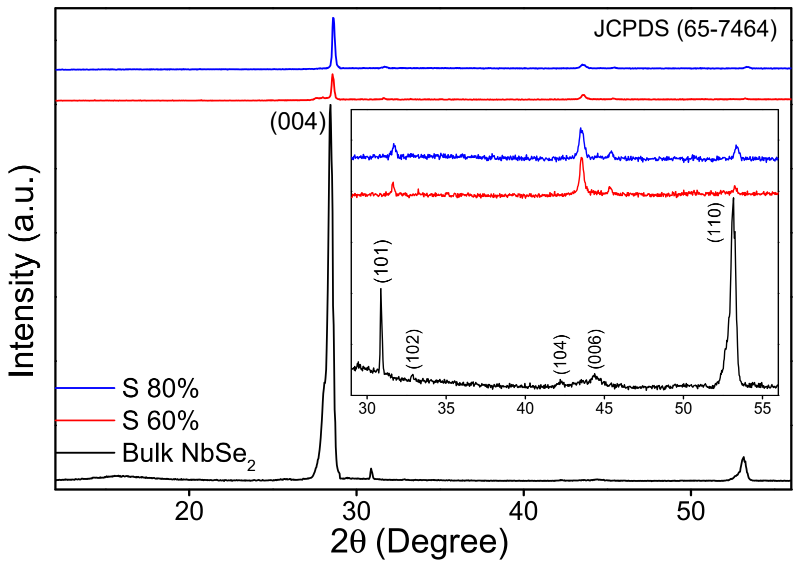

Figure 1. XRD of the $NbSe_2$ prepared via the liquid exfoliation method in comparison with the bulk (inset shows the peaks from 29 to 56 2 theta).

3.2. Scanning Electron Microscopy (SEM)

The SEM images of the $NbSe_2$ bulk (Figure 2a) show a wafer-like morphology with a thickness in the order of micrometres. A reduction in thickness can be observed in S 60% and S 80% (Figure 2b,c respectively). Despite this, S 80% exhibits less retention than S 60% in the prepared sheets' lateral dimensions. This reduction is due to the excess exfoliating power. S 60% shows much promise in maintaining the lateral dimensions of bulk samples while significantly reducing thickness.

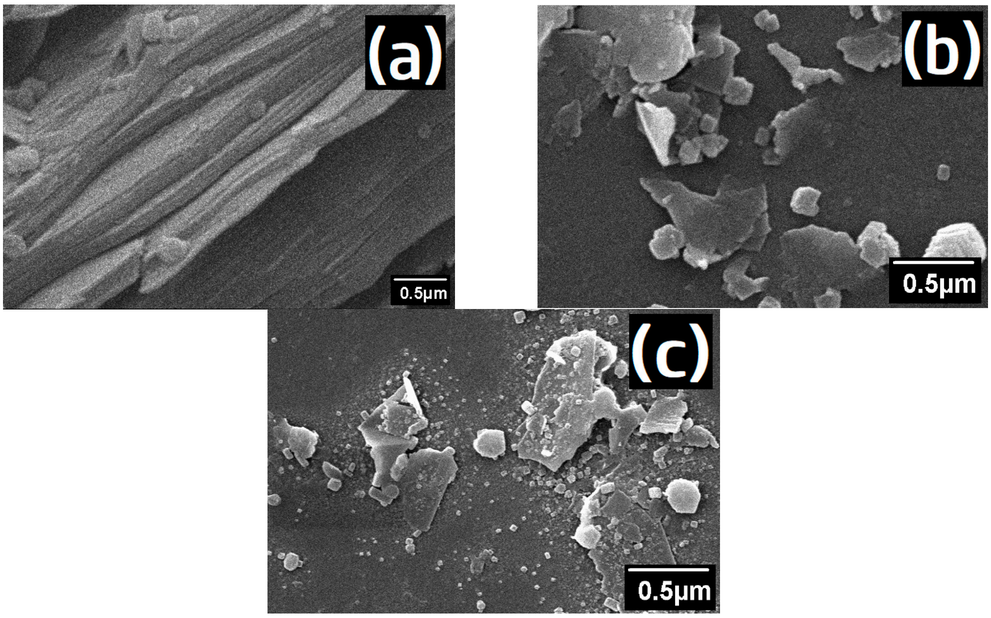

Figure 2. SEM images of the $NbSe_2$ prepared using the liquid exfoliation method compared to bulk. (**a**) Bulk; (**b**) prepared at 60%; and (**c**) prepared at 80%.

4. Conclusions

$NbSe_2$ 2D nanosheets were prepared using the liquid exfoliation method at different exfoliation powers, namely, 520 Watts (S 80%) and 390 Watts (S 60%). When compared to the bulk homologue, the XRD data show a hexagonal crystal structure with a noticeable reduction in thickness. The blue shift in the exfoliated sample in the (004) and (106) planes indicates the stresses and defects in the $NbSe_2$ nanosheets. The SEM images show a dramatic decrease in thickness from 80% to 60%. Furthermore, 60% power is relatively more effective in reducing the thickness of $NbSe_2$ bulk, with very little lateral dimension degradation.

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