Structural analysis of $ZrS_{0.2}Se_{2.8}$and $ZrS_{2.8}Se_{0.2}$Single crystals

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Abstract

X-ray diffraction (XRD) and electron diffraction methods (EDAX) are powerful techniques for studying the atomic structure of materials. XRD is particularly useful for determining the crystal structure of single crystals and powders, while EDAX can provide information on the composition and crystallographic orientation of materials at the nanoscale.

X-ray topography is another XRD-based technique that allows for the visualization of crystal defects such as dislocations, stacking faults, and grain boundaries. By analyzing the diffraction pattern produced by a crystal, x-ray topography can provide information on the type and distribution of defects within the crystal lattice.

In addition to XRD and EDAX, there are other techniques that can be used to study the atomic structure of materials, such as neutron diffraction, transmission electron microscopy (TEM), and scanning electron microscopy (SEM). Each technique has its own advantages and limitations, and the choice of technique depends on the specific research question and the properties of the sample being studied.

Keywords— TEM, XRD, TMDC, CVT, EDAX, x-ray

I. INTRODUCTION

To prepare electron-straightforward films for TEM study, several procedures are commonly used, including physical vapour deposition (PVD), chemical vapour deposition (CVD), and sputtering [1]. PVD involves evaporating a material in a vacuum chamber to create a thin film on a substrate, while CVD utilises chemical reactions to deposit the material on the substrate. Sputtering, on the other hand, involves bombarding a material with high-energy particles to dislodge atoms, which then deposit onto the substrate.

In electronic and photonic material frameworks, the beneficial features of semiconductors arise from their unique properties, which are closely related to their atomic and electronic structures. By controlling the interaction between materials during preparation, scientists can design and engineer new semiconductor materials with improved performance for specific applications [5].

In crystal structure characterisation, the basic symmetry of the crystal lattice can be used to identify well-defined developments. The dimensions of the unit cell, which is the smallest repeating unit of a crystal lattice, can be measured using various techniques, including X-ray diffraction (XRD). For instance, single crystals of layer compounds produced by iodine vapour transport with compositions ZrSxSe3-x, where x ranges between 0 and 3, may be measured by XRD using CuK radiation [11].

II. EXPERIMENTS TECHNIQUE

A. E.M. WAVES INTERACTION WITH MATERIAL

Electromagnetic waves interact with matter in different ways depending on their energy and frequency. when electromagnetic waves interact with matter, they can be absorbed, transmitted, or scattered, leading to various physical and chemical effects in the material[7].

Microwaves, infrared, and ultraviolet (uv) radiation are examples of electromagnetic waves that interact with matter by exciting molecular vibrations and rotations. This can lead to heating and chemical reactions, as well as changes in the electronic structure of the material (Figure-2).

X-rays, on the other hand, have high energy and can penetrate deeply into matter, interacting with the electrons of atoms and causing ionization. This ionization process can lead to the creation of free radicals and other reactive species, which can be useful in a range of applications such as radiation therapy for cancer treatment [7][8].

In the ionization-ray photonic energy transfer method, X-rays transfer energy to electrons, resulting in ionization and the production of ions. The remaining energy of the X-ray photon is scattered in a process known as Compton scattering [9]. This process can be used to study the electronic structure of materials and is commonly used in X-ray diffraction experiments.

Overall, the interaction of electromagnetic waves with matter is a complex process that depends on the energy and frequency of the waves, as well as the physical and chemical properties of the material. By understanding these interactions, we can develop new materials and technologies with a wide range of applications.

B. ENERGY DISPERSIVE ANALYSIS OF X-RAYS (EDAX)

EDAX. is a powerful tool for elemental analysis and can provide qualitative as well as quantitative information on the composition of the sample being analyzed. The X-ray energy spectrum obtained from EDAX can be used to identify the elements present in the sample and

their relative abundances. The peaks in the energy spectrum correspond to the energy difference between the electron shells of the atoms, and their positions are unique to each element.

The EDAX setup is commonly integrated with the SEM system and can also be used with scanning transmission electron microscopes. X-ray fluorescence spectrometers, on the other hand, use X-ray beam excitation to generate X-rays [3]. The X-rays produced by the sample are converted to voltage signals, which are then processed and analyzed using software to provide information on the elemental composition of the sample.

Liquid-cooled Si (Li) detectors are commonly used in EDAX setups at cryogenic temperatures. However, newer systems may also include silicon drift detectors (SDD) and Peltier cooling systems. The use of EDAX is particularly important for the analysis of materials with complex structures and compositions, such as semiconductors, oxides, metals, and organic materials [13,15].

Energy-dispersive X-ray analysis (EDAX) is a technique that is used to determine the elemental composition and atomic structure of a material by analyzing the X-rays that are produced when an electron beam or charged particle beam is directed at the material. When the beam interacts with the atoms in the material, it can cause inner-shell electrons to be excited, which can then produce X-rays as the electrons transition to lower energy states [16]. The energy and number of these X-rays can be measured using an EDAX detector, and the resulting data can be used to identify the elements present in the material and their relative concentrations.

In the case of ZrS2.8Se0.2 and ZrS0.2Se2.8 materials, EDAX was used to confirm the presence of zirconium, sulfur, and selenium in the samples and to determine their off-stoichiometric composition. The X-ray energy spectrum obtained from the EDAX analysis showed distinct peak positions that corresponded to the possible transitions in the principal energy level of the elements present in the sample. By analyzing the peak positions and intensities, it was possible to determine the relative concentrations of each element in the sample (Figure-2 &3).

Overall, EDAX is a useful tool for both qualitative and quantitative analysis of materials, and it can provide valuable information about their elemental composition and atomic structure [17].

Figure 1 Layout of EDAX with TEM for Bruker X Flash 630 EDS Detector

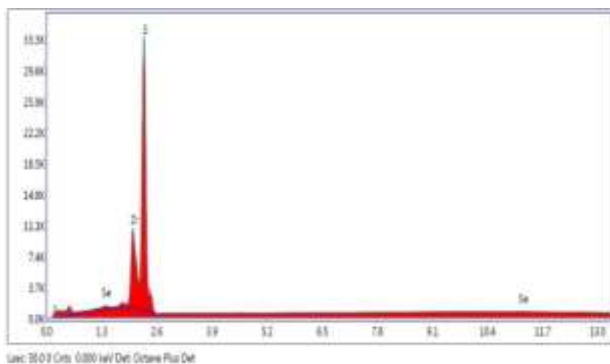

Figure 2The spectrum of energy dispersionfor $ZrS_{2.8}Se_{0.2}$

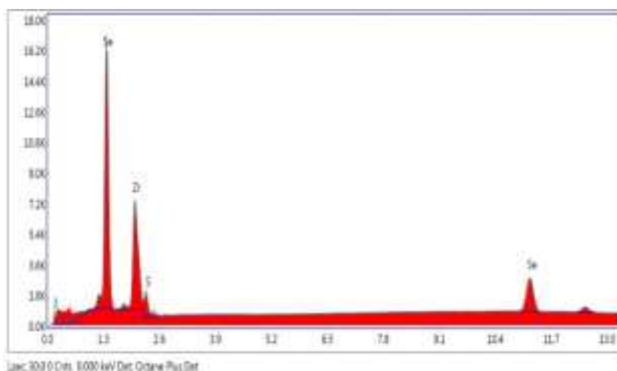

Figure 3 The spectrum of energy dispersion for$ZrS_{0.2}Se_{2.8}$

Element	Experimental Weight %	Weight (%)	Atomic %
ZrL	24.64	26.62	10.53
S K	72.35	69.56	87.98
SeK	3.01	3.82	1.48

Table 1.EDAX analysis of thechemical composition (in percent) for $ZrS_{2.8}Se_{0.2}$ single crystals.

Element	Experimental Weight %	Weight (%)	Atomic %
ZrL	18.26	16.43	15.95
S K	1.06	1.16	2.62
SeK	80.68	82.41	81.42

Table 2. EDAX analysis of thechemical composition (in percent) for $ZrS_{0.2}Se_{2.8}$single crystals.

C. X-RAY DIFFRACTION

X-ray diffraction (XRD) is a powerful technique for identifying the crystallographic structure of materials. In XRD, a beam of x-rays is directed at a sample, and the resulting diffraction pattern is analyzed to determine the crystal structure. The diffraction pattern is created by the constructive and destructive interference of the scattered x-rays from the regular array of atoms within the crystal. The diffraction pattern is a unique fingerprint of the crystal structure and can be used to identify the phases present in a sample and to determine their relative proportions. In addition, XRD can provide valuable information on the unit cell dimensions, symmetry, and orientation of the crystal lattice. The technique is widely used in materials science, mineralogy, chemistry, and physics, and has applications in fields such as drug discovery, forensic science, and archaeology.

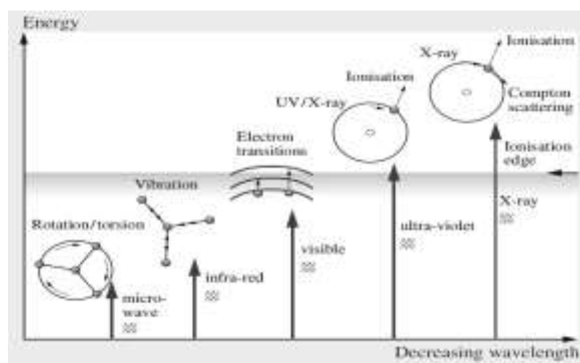

Figure 4(E.M. Waves Interaction with Material)

Figure 5(Schematic Diagram of Diffractometer)

A diffractometer (Figure-5) is an instrument used to measure the diffraction of X-rays or other electromagnetic radiation from a crystal. It works by directing a beam of X-rays at a crystal and measuring the intensity and angles of the diffracted X-rays. The diffracted X-rays are detected by an electronic detector, such as a scintillation counter or a charge-coupled device (CCD), and the resulting data is analyzed to determine the crystal structure. The diffractometer can be used to determine the crystallographic structure of materials, study the arrangement of atoms in crystals, and analyze the composition of materials[10,12].

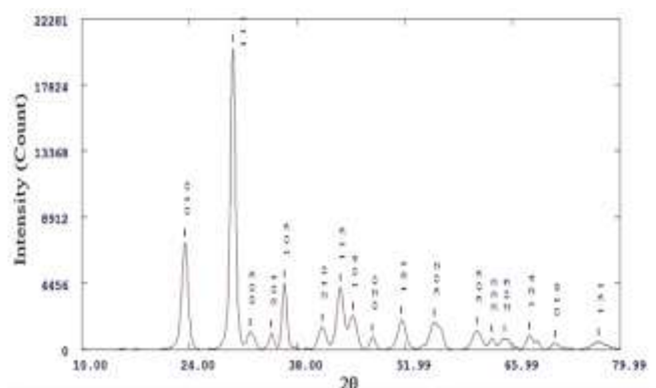

Figure 6(XRD Graph for $ZrS_{0.2}Se_{2.8}$)

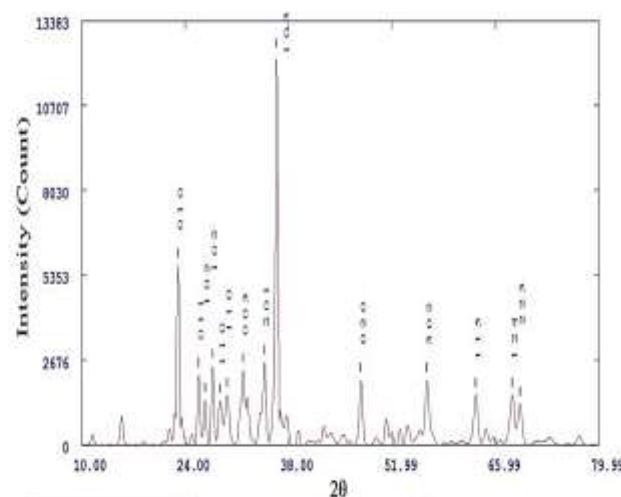

Figure 7(XRD Graph for $ZrS_{2.8}Se_{0.2}$)

XRD is a powerful technique for determining the crystal structure and properties of materials. The monochromatic copper K-alpha radiation with a wavelength of 1.542A0 is a common source used in XRD because it has a high intensity and is well-suited for diffraction studies[2]. The XRD diffractometer measures the reflected X-rays from a crystal plane and records the diffracted intensity using electronic counters. By analyzing the diffraction pattern and the intensity peaks, it is possible to determine the crystal structure, quality, and layer thickness of the sample [6]. The XRD technique is widely used in various fields, including materials science, chemistry, and geology, to understand the properties and behavior of different materials.

III. STRUCTURAL CHARACTERISATION

Crystal	$a(A^0)$	$b(A^0)$	$c(A^0)$	β	Grain size D (nm)	X-ray Density ρ (gm/cc)	Unit cell volume $(A^0)^3$	Composition(X)
$ZrS_{0.2}Se_{2.8}$	5.38	3.84	8.45	97.43	38.43	5.38	182.23	0.52
$ZrS_{2.8}Se_{0.2}$	5.32	3.43	8.43	97.26	24.61	4.24	165.36	1.48
Crystal	$a(A^0)$	$b(A^0)$	$c(A^0)$	β	Grain size D (nm)	X-ray Density ρ (gm/cc)	Unit cell volume $(A^0)^3$	Composition(X)
$ZrS_{0.2}Se_{2.8}$	5.38	3.84	8.45	97.43	38.43	5.38	182.23	0.52
$ZrS_{2.8}Se_{0.2}$	5.32	3.43	8.43	97.26	24.61	4.24	165.36	1.48

Table 3. Lattice parameters, unit cell volumes, X-ray densities For $ZrS_{0.2}Se_{2.8}$ and $ZrS_{2.8}Se_{0.2}$crystals.

At room temperature, $ZrS_{0.2}Se_{2.8}$ and $ZrS_{2.8}Se_{0.2}$ were passed through a 106 m mesh sieve to obtain powdered grown crystal. Powder for X-rays Diffractometer (Phillips, Model – X MPD) with 0.0030 accuracy and a Cu target X-Ray tube. The length of a unit cell is listed in Table 3, and the x-ray diffraction pattern is shown in Figures 6 and 7.

IV. CONCLUSIONS

The use of an X-ray powder diffractometer and energy-dispersive spectroscopy (EDAX) allowed for an effective study of the structural properties of single-crystalline ZrS0.2Se2.8 and ZrS2.8Se0.2. The results of the structural XRD pattern analyses showed that both samples have a monoclinic symmetry. The particle size obtained for ZrS0.2Se2.8 was 38.43 nm, while for

ZrS2.8Se0.2 it was 24.61 nm. EDAX analysis confirmed the presence of zirconium, sulfur, and selenium in the samples, with no contaminants detected.

The off-stoichiometric extent of constituent components was determined using EDAX, and the graph obtained showed that an off-stoichiometric structure exists in both ZrS0.2Se2.8 and ZrS2.8Se0.2 samples. The X-ray diffractograms showed excellent crystallinity, and the lattice parameters indicated that the volume of the unit cell decreased with an increasing proportion of sulfur.

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