

Synthesis, Structure, Magnetic and Electric Transport Properties of $Zr_{0.5}Al_{0.5}Se_{1.95}$

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Abstract

A new phase with the composition $Zr_{0.5}Al_{0.5}Se_{1.95}$ has been synthesized by the standard ceramic method. X-ray diffraction studies show that the phase crystallizes with the hexagonal unit cell ($a=21.784\text{\AA}$, and $c=8.063\text{\AA}$, α and $\beta = 90^\circ$ and $\gamma=120^\circ$). The molar magnetic susceptibility measurements as a function of temperature suggest that the phase is diamagnetic and magnetic susceptibility is temperature independent.

Keywords: Mixed binary dichalcogenides, XRD, Molar Magnetic Susceptibility.

Introduction:

Binary dichalcogenides of numerous elements with composition MX_2 and their mixed analogues $M_{1-x}M'X_2$ (M and M' are different transition elements; X=S, Se or Te) are known in the literature [1, 2]. Many dichalcogenides with reduced content of X are also known [3, 4]. It has been reported that structure and physical properties substantially vary with change in composition [1, 2, 3, 4]. It was thought interesting to prepare mixed chalcogenides with composition $M_{0.5}M'_{0.5}X_2$ study of their crystal structure & follow their magnetic properties as function of temperature. In the present study, synthesis of a new phase with the composition $Zr_{0.5}Al_{0.5}Se_{1.95}$ has been reported. Its crystal structure has been determined from the powder X-ray diffraction data. Magnetic properties have been studied in the temperature range 80K-300K.

Experiment:

Synthesis

Aldrich makes Zirconium (Zr) Aluminum (Al) and Selenium (Se) elements (purity 99.9%) have been used for synthesis of the new phase. The constituent elements weighed corresponding to the stoichiometry $Zr_{0.5}Al_{0.5}Se_{1.95}$, were mixed and homogenized by grinding in cyclohexane. The dried and homogenized mixture, pressed into pellets in hydraulic press was placed in quartz tube and evacuated to $\sim 10^{-5}$ Torr, vacuum sealed and was heat-treated at 1048K for 72 hours. The mixture during the heat treatment was subjected to a number of intermediate grindings, pelletizing and sealing under same conditions for the completion of the reaction. The final product was pulverized to fine powder for further investigations [5, 6, 7].

Elemental Analysis

The phase was further analyzed by atomic absorption spectrophotometry, which is one of the most prevalent methods for the trace element analysis [8, 9, 10]. The results of chemical elemental analysis [11, 12] and the atomic absorption spectrophotometry are in good agreement. The data are given in Table 1.

Table 1: Analytical data of the phase ($Zr_{0.5}Al_{0.5}Se_{1.95}$).
The theoretical value is given parenthesis. Analysis (%)

Phase	Zr	Al	Se
$Zr_{0.5}Al_{0.5}Se_{1.95}$	20.91 (21.01)	6.09 (6.21)	70.94 (72.76)

X-ray Diffraction studies

Room temperature powder X-ray diffraction data of the product were recorded on a Stoe-powder diffraction system and a Philips diffractometer at a scanning speed of 1degree/minute in the 2θ range using $CuK\alpha$ and $FeK\alpha$ radiations [13, 14 and 15]. The X- ray diffraction data are given in the Table 2, while the X-ray pattern, intensity, versus 2θ is drawn in the figure1.

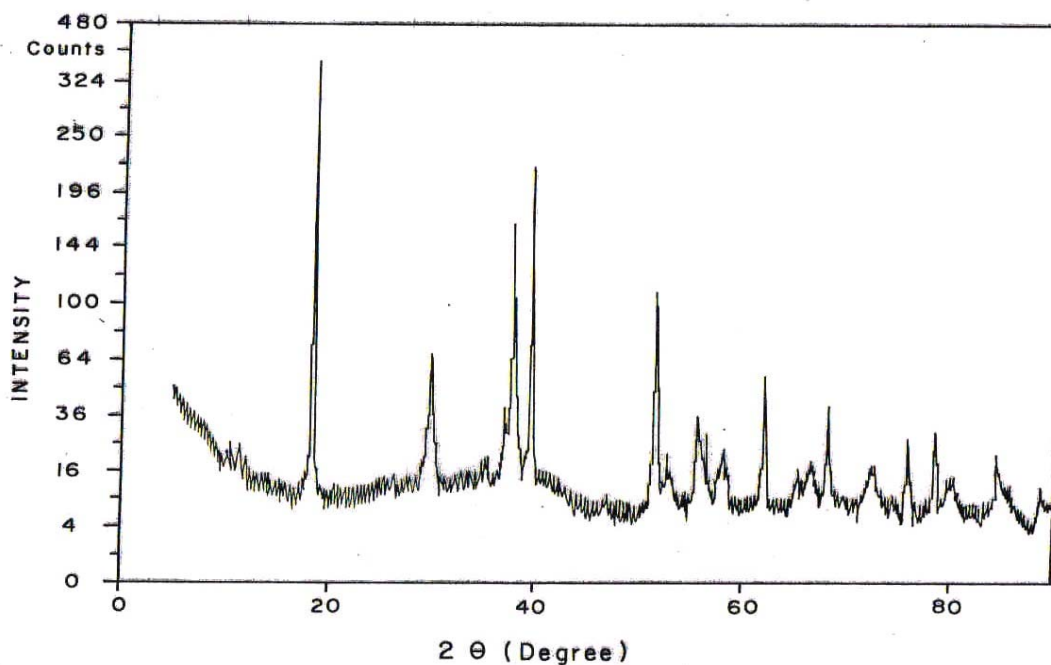


Figure 1: X-ray Diffraction pattern of $Zr_{0.5}Al_{0.5}Se_{1.95}$

Magnetic Susceptibility Measurement

Magnetic susceptibility of the powdered phase was recorded in a Faraday balance provided with Polytronic Faraday-type electromagnet and a Mettler microbalance. Specially fabricated Dewar flask of the size which could be adjusted within pole gaps of electromagnet was used for keeping liquid nitrogen,

which surrounded the phase crucible [16, 17]. The phase was held hanging in the inner tube of the Dewar flask with a fine thread. Magnetic susceptibility in the temperature range 77K-300K could be measured by this arrangement.

Result and Discussion

Crystal Structure

The unit cell parameters of the phase were calculated from X-ray diffraction data (Table 2). The indexing of the data shows that it crystallizes in the orthorhombic unit cell with $a = 21.784 \text{ \AA}$, $c = 8.063 \text{ \AA}$, $\alpha = \beta = 90^\circ$, $\gamma = 120^\circ$. In order to determine the crystal structure, the theoretical X-ray diffraction data were generated by Treor and Lazy- Pulverix analysis. The d_{cal} values computed from data are in good agreement with the experimental interplanar distances. The data along with the assigned $h k l$ values are given in the Table 2.

Table 2: Powder X-ray Diffraction Data of $\text{Zr}_{0.5}\text{Al}_{0.5}\text{Se}_{1.95}$

H	K	L	$d_{\text{obs}} (\text{\AA})$	$d_{\text{cal}} (\text{\AA})$	I_{obs}
2	0	1	6.129	6.133	100.0
3	1	1	4.403	4.392	1.4
5	0	0	3.776	3.776	15.7
4	0	2	3.062	3.066	10.3
4	1	2	2.881	2.882	58.5
4	0	3	2.333	2.337	29.8
5	5	0	2.180	2.180	3.3
5	4	2	2.069	2.073	7.4
6	0	3	2.043	2.044	4.1
8	2	1	1.994	1.996	3.5
5	1	4	1.732	1.733	8.7
12	1	0	1.505	1.506	2.1
8	2	4	1.440	1.441	4.1

$$a = 21.784 \text{ \AA}$$

$$c = 8.063 \text{ \AA}$$

$$\alpha = \beta = 90^\circ$$

$$\gamma = 120^\circ$$

Magnetic susceptibility studies

The molar magnetic susceptibility measurements as a function of temperature suggest that the phase is diamagnetic and magnetic susceptibility is temperature independent.

Conclusion:

A new phase with the composition $Zr_{0.5}Al_{0.5}Se_{1.95}$ has been synthesized by the standard ceramic method. On the basis of Lazy-Pulverix analysis of the X-ray diffraction data it is concluded that the phase crystallizes in the hexagonal unit cell. The molar magnetic susceptibility (χ_m) measurements as a function of temperature suggest that the phase is diamagnetic and magnetic susceptibility is temperature independent.

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