Synthesis, Structure, Magnetic and Electric Transport Properties of $Zr_{0.5}Si_{0.5}Se_{1.90}$

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Abstract

A new phase with the composition $Zr_{0.5}Si_{0.5}Se_{1.90}$ has been synthesised by the standard ceramic method. X-ray diffraction studies show that the phase crystallizes with the orthorhombic unit cell (a=14.221Å, b=13.557 and c=6.537Å). 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 $Zr_{0.5}Si_{0.5}Se_{1.90}$ 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 make Zirconium (Zr) Silicon (Si) and Selenium (Se) elements (purity 99.9%) have been used for synthesis of the new phase. The constituent elements weighed corresponding to the stoichiometry $Zr_{0.5}Si_{0.5}Se_{1.90}$, were mixed and homogenised by grinding in cyclohexane. The dried and homogenised mixture, pressed into pellets in hydraulic press was placed in quartz tube and evacuated to ~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 pulverised to fine powder for further investigations [5, 6, 7].

Elemental Analysis

The phase was further analysed 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 ($Zr_{0.5}Si_{0.5}Se_{1.90}$).
The theoretical value is given parenthesis. Analysis (%)

Phase	Zr	Si	Se
$Zr_{0.5}Si_{0.5}Se_{1.90}$	20.84 (20.96)	6.35 (6.45)	68.95 (72.58)

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 1deg./minute in the 2θ range using CuKα and FeKα radiations [13, 14, 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.

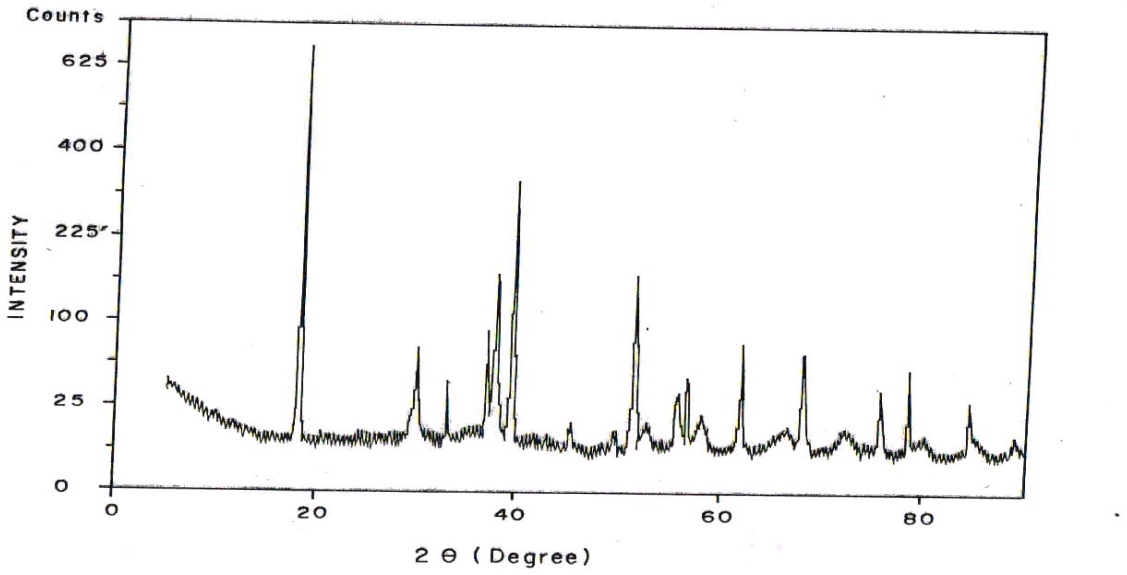

Figure 1: *X-ray Diffraction pattern of $Zr_{0.5}Si_{0.5}Se_{1.90}$*

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 polegaps 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.

Results 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 crystallises in the orthorhombic unit cell with **a=14.221Å, b=13.557 and c=6.537Å**. In order to determine the crystal structure, the theoretical X-ray diffraction data were generated by Treor and Lazy- Pulverix analysis. The $\mathbf{d_{cal}}$ 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 $Zr_{0.5}Si_{0.5}Se_{1.90}$

h	k	l	d_{obs} (Å)	d_{cal}(Å)	I_{obs}
1	2	0	6.123	6.124	100.0
4	1	0	3.431	3.441	18.6
2	4	0	3.060	3.062	12.1
0	4	1	3.000	3.011	21.6
1	2	2	2.880	2.885	46.5
3	3	2	2.308	2.313	0.7
2	4	2	2.234	2.235	18.9
0	0	3	2.181	2.180	1.3
5	4	1	2.066	2.068	3.8
3	6	0	2.041	2.041	5.2
2	2	3	1.993	1.993	2.0
4	5	2	1.801	1.801	1.7
8	1	0	1.762	1.764	1.8
3	6	2	1.731	1.731	8.8
2	5	3	1.651	1.653	0.9
6	0	3	1.604	1.605	0.7
7	5	1	1.577	1.579	4.6
4	5	3	1.532	1.533	6.3
2	3	4	1.502	1.503	0.9
1	7	3	1.440	1.441	3.0
4	8	2	1.385	1.386	1.3

a=14.221Å b=13.557 c=6.537Å

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 $Zr_{0.5}Si_{0.5}Se_{1.90}$ has been synthesised by the standard ceramic method. On the basis of Lazy-Pulverix analysis of the X-ray diffraction data it is concluded that the phase crystallises in the orthorhombic 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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References:

[1] Folmer J.C.W, Jellinek F. and Calis G.H.M. (1988).The Electronic Structure of Pyrites, Particularly CuS_2 and $Fe_{1-X}Cu_xSe_2$: An XPS And Mössbauer Study**,** J. Solid State Chem., 72**,** 137-144.

[2] Li Jing, Guo H.Y., Proserpio D.M. and Sironi A. (1995).Exploring Tellurides: Synthesis and Characterization of New Binary, Ternary, and Quaternary Compounds**,** J. Solid State Chem., 117**,** 247-255.

[3] Fischer P. Plambeck, Abriel W. and Urland W. (1989). Preparation and Crystal Structure of $RESe_{1.9}$ (RE = Ce, Pr), J. Solid State Chem., 78, 164.

[4] Pardo M.P., Gardette M.F., Dung N. and Flahaut J. (1991). Insertion d'oxygène dans les composés de type $Cu_{0.5}RTe_2$ et $Cu_{0.5}RTe_{1.75}$ (R = La et Nd)**,** J. Solid State Chem., 94,121-129.

[5] Huan G. and Greenblatt M. (1987*).* New $A_xNb_6 Se_8$ (A= Na, K, Rb, Cu, Ag, Zn, Cd, Pb) phases with the $Nb_3 Te_4$ structure, Mater. Res. Bull., 22, 505.

[6] Huan G. and Greenblatt M, (1991). Inorganic Syntheses, Non molecular Solids**,** Mater. Res. Bull., 22, 943.

[7] Limatta E.W. and lbers J.A. (1987*)***.**Synthesis, Structure, and Physical Properties of the New Layered Ternary Chalcogenide $NbNiTe_5$**,** J. Solid State Chem.,71, 384.

[8] Elwell W.T. and Gidley J.A.F., Atomic Absorption Spectrophotometry, Pergamon, 102. (1961).

[9] Robinson J.B., Atomic Absorption Spectroscopy, Marcell Dekker, New York. (1966).

[10] Khopkar S.M., Basic Concept of Analytical Chemistry, Wiley Eastern Limited. (1985).

[11] Taguchi H., Nagao M. and Shimada M. **(**1988*).* Metal-Insulator Transition in the System $(Nd_{1-x}Ca_x)$ $MnO_{2.99}$ $(0.5\leq x\leq 0.9)$**,** J. Solid State Chem., 76**,** 284-289.

[12] Jeffery G.H., Vogel's Text Book of Quantitative Chemical Analysis (ELBS, Longman, Essex). (1989).

[13] Yvon K., Jeitschko W. and Perthe E., Lazy Pulverix, Laboratory De Crystallographic Aux Rayons-X, Universities De Genève, Genève, Switzerland. (1977).

[14] Werner P. E., Treor - 4 Deptt. of Structural Chemistry, Arrhenius Laboratory University of Stockholm, Sweden. (1984).

[15] Noel H., Potel M., Troc R. and Shlyk. L. **(**1996). Crystal Structure and Physical Properties of β $USe_2$and USe_{2-x} Te_x (x= 0.24 and 0.72)**,** J. Solid State Chem., 126, 22-26.

[16] Selwood P.W., Magnetochemistry, (Interscience, New York), (1943).

[17] Goodenough J.B. (1963).Magnetism and the Chemical Bond (Interscience, New York).