Synthesis, Structure, Magnetic and Electric Transport Properties of $Zr_{0.5}Si_{0.5}Te_{1.93}$

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Abstract

A new phase with the composition $Zr_{0.5}Si_{0.5}Te_{1.93}$ has been synthesised by the standard ceramic method. X-ray diffraction studies show that the phase crystallizes with the tetragonal unit cell (a=12.756Åand c=10.917Å).The molar magnetic susceptibility measurements as a function of temperature suggest that the phase is diamagnetic and magnetic susceptibility is temperature independent. The electrical resistivity measurements as a function of temperature suggest that the phase is semi-conductor in nature in the temperature range 300K-500K and the conduction occurs via thermally activated mechanism.

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

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 physical properties as function of temperature. In the present study, synthesis of a new phase with the composition $Zr_{0.5}Si_{0.5}Te_{1.93}$ has been reported. Its crystal structure has been determined from the powder X-ray diffraction data. Magnetic and electric transport properties have been studied in the temperature range 80K-300K and 300K-500K respectively.

Experiment

Synthesis

Aldrich make Zirconium (Zr) Silicon (Si) and Tellurium (Te) elements (purity 99.9%) have been used for synthesis of the new phase. The constituent elements weighed corresponding to the stoichiometry $Zr_{0.5}Si_{0.5}Te_{1.93}$, 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 ($Zr_{0.5}Si_{0.5}Te_{1.93}$).
The theoretical value is given parenthesis. Analysis (%)

Phase	Zr	Si	Te
$Zr_{0.5}Si_{0.5}Te_{1.93}$	14.42 (14.48)	4.39 (4.46)	78.21 (81.05)

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 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.

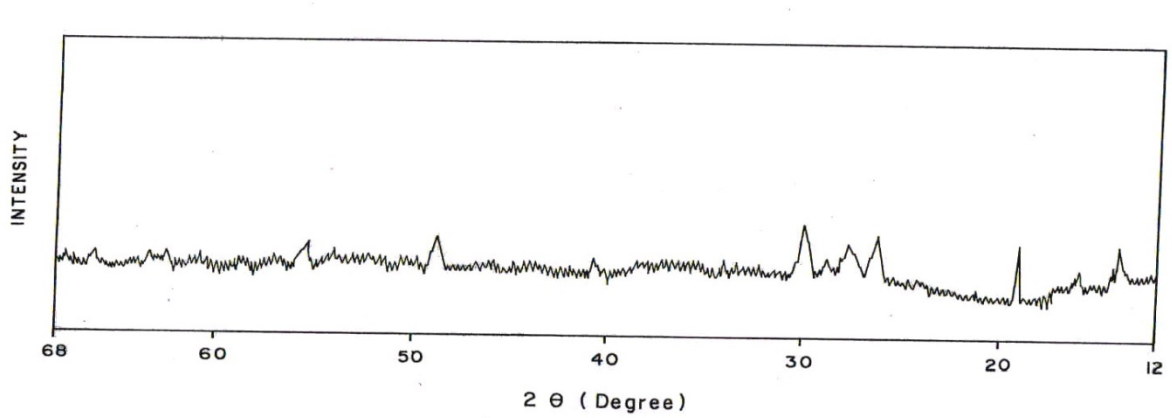

Figure 1:X-ray Diffraction pattern of $Zr_{0.5}Si_{0.5}Te_{1.93}$

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.

Electrical Resistance study

Electrical Resistivity of thin pellet of the phase as a function of temperature in a continues flow of nitrogen was recorded by four probe method in a four probe cell, using Keithley programmable constant current supply source model 224 and nanovoltmeter model 181 for the purpose of current source and voltage measurement respectively [18,19]. The bottom surface of the pellet was kept non-conducting. The data of specific resistance (ρ) as a function of temperature are given in Table 4, while the log ρ versus 1/T data are plotted in the figure 2.

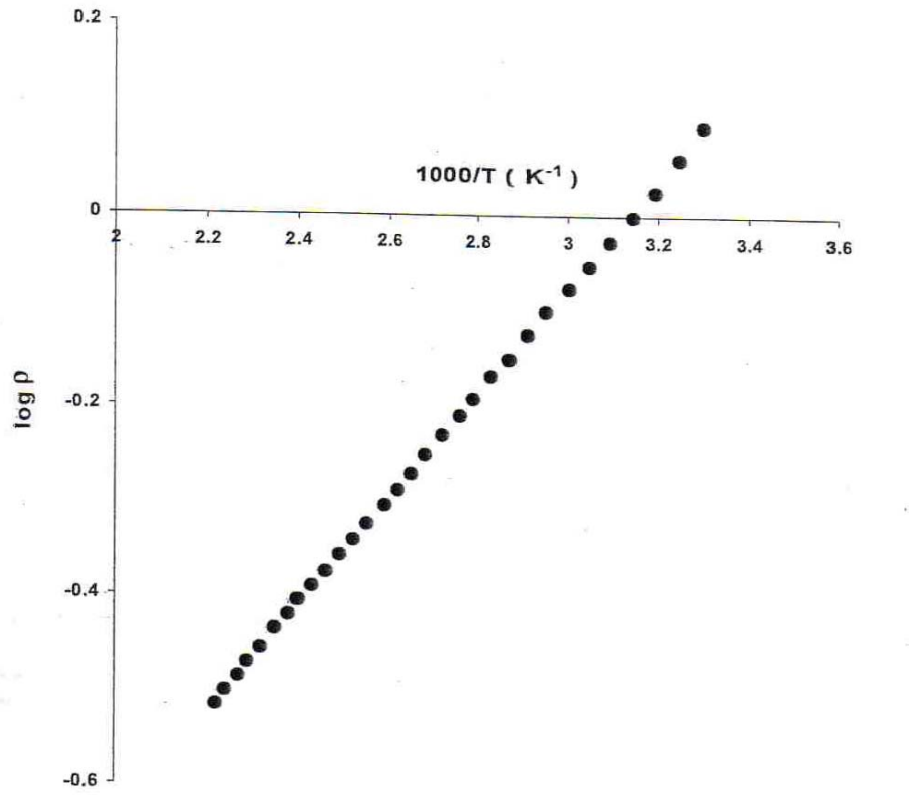

Figure 2: Log ρ versus1/T plot of $Zr_{0.5}Si_{0.5}Te_{1.93}$

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 tetragonal unit cell with **a=12.756Åand c=10.917Å**. 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.

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.

Electric Transport Properties

The log of specific resistance (log ρ) versus temperature (K) values (Table 3) are plotted in the figure 2. The negative temperature co-efficient of resistivity and the values of the specific resistance suggest that the phase is semi-conductor in nature and the linearity of the plot shows that the electrical conduction occurs via thermal activated mechanism.

Table 2: Powder X-ray Diffraction Data of $Zr_{0.5}Si_{0.5}Te_{1.93}$

h k l	d_{obs} (Å)	d_{cal}(Å)	I_{obs}

2	0	0	6.378	6.383	23
1	1	2	4.670	4.673	40
1	1	3	3.375	3.377	23
4	0	0	3.197	3.197	100
4	1	1	2.978	2.979	36
1	1	4	2.614	2.614	93
2	1	4	2.461	2.463	56
6	0	0	2.126	2.127	56
7	3	0	1.675	1.676	73
8	1	0	1.582	1.583	74
6	1	5	1.512	1.513	80
7	2	5	1.367	1.367	70

a= 12.756Å c= 10.917Å

Table 3: Specific resistance (log ρ) of $Zr_{0.5}Si_{0.5}Te_{1.93}$ as function of temperature (K).

Temperature (K)	Specific resistance ρ (ohm cm)
449	0.305
445	0.315
440	0.326
435	0.338
430	0.350
425	0.366
420	0.380
415	0.393
410	0.406
405	0.421
401	0.439
396	0.455
391	0.473
386	0.495
381	0.514
377	0.535
372	0.560
367	0.588
362	0.615
358	0.642
353	0.679
348	0.707
343	0.750
338	0.792
333	0.838
328	0.884
323	0.939

318	1.000
313	1.062
308	1.149
303	1.242

Table3: Magnetic and Electric Transport Parameters of ($Zr_{0.5}Si_{0.5}Te_{1.93}$) phase.

Phase	μ_{eff} (B.M)	μ_{theo}(B.M)	E_a(eV)
$Zr_{0.5}Si_{0.5}Te_{1.93}$	Diamagnetic	-	0.10

Conclusion

A new phase with the composition $Zr_{0.5}Si_{0.5}Te_{1.93}$ 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 tetragonal unit cell. The molar magnetic susceptibility measurements as a function of temperature suggest that the phase is diamagnetic and magnetic susceptibility is temperature independent. The study of electrical resistivity in the temperature range 300K-500K shows that the compound is an electrical semi-conductor and conduction occurs via thermal activated mechanism.

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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 $A_xNb_6Se_8$ (A= Na, K, Rb, Cu, Ag, Zn, Cd, Pb) phases with the Nb_3Te_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).
[18] Resistivity of Semiconductors by Four Probe Method at Different Temperature Scientific Equipment and Services Roorkee 247667 U.P).
[19] Wieder H.H and Elsevier Amsterdam., Laboratory Notes on Electrical and GalvanomagneticMeasurements.67, 53-55. (1979).