

## Synthesis, Structure, Magnetic and Electric Transport Properties of $W_{0.5}Si_{0.5}Se_{1.95}$

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### Abstract

A new phase with the composition  $W_{0.5}Si_{0.5}Se_{1.95}$  has been synthesised by the standard ceramic method. X-ray diffraction studies show that the phase crystallizes in orthorhombic unit cell ( $a=16.973\text{\AA}$ ,  $b=12.981\text{\AA}$  and  $c=8.609\text{\AA}$ ). 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 semiconductor in nature in the temperature range 300K-500K and the conduction occurs via thermally activated mechanism. The thermal analysis suggests that upto 673 K there is almost complete mass stability of the phase, while above this temperature there is mass loss of 11.88%.

**Keywords:** Mixed binary dichalcogenides, XRD, electrical resistivity, TG-DTA.

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### Introduction:

Binary dichalcogenides of numerous elements with composition  $MX_2$  and their mixed analogues  $M_{1-x}M^{\square}X_2$  (M and  $M^{\square}$  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  $M_{0.5}M^{\square}_{0.5}X_2$  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  $W_{0.5}Si_{0.5}Se_{1.95}$  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. The phase has been analysed for thermogravimetric analysis (TGA) and differential thermogravimetric analysis (DTGA).

### Experiment

#### Synthesis

Aldrich make Tungsten (W) 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  $W_{0.5}Si_{0.5}Se_{1.95}$ , 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  $\sim 10^{-5}$  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 pulverised to fine powder for further investigations [5, 6, 7].

### Elemental Analysis

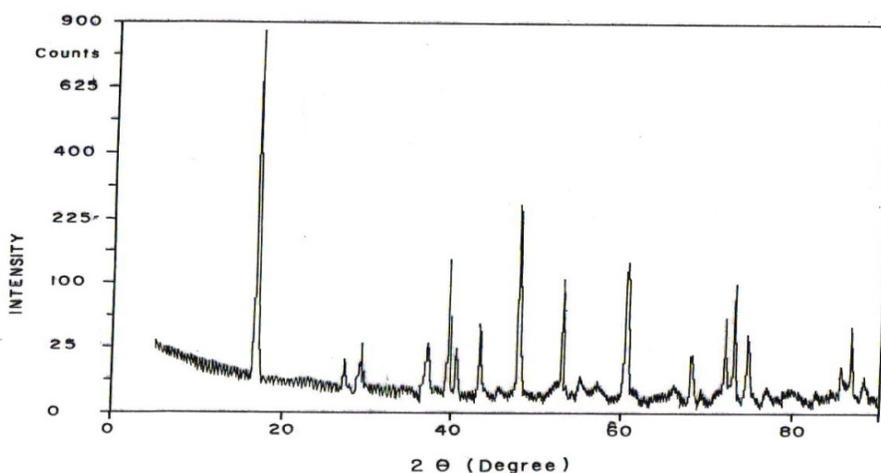
The phase was further analysed by atomic absorption spectrophotometry, which is one of the most prevalent methods for the trace element analysis [8, 9, 10]. The results of chemical elemental analysis [11, 12] and the atomic absorption spectrophotometry are in good agreement. The data are given in Table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

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

Phase	W	Si	Se
$W_{0.5}Si_{0.5}Se_{1.95}$	34.70 (34.83)	5.19 (5.32)	58.34 (59.84)

### 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  $2\theta$  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  $2\theta$  is drawn in the figure1.



**Figure 1:** X-ray Diffraction pattern of  $W_{0.5}Si_{0.5}Se_{1.95}$

### 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.

### Electrical Resistance study

Electrical Resistivity of thin pellet of the phase as a function of temperature in a continuous flow of nitrogen was recorded by four probe method in a four probe cell, using Keithley programmable constant current 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 ( $\rho$ ) as a function of temperature are given in Table 4, while the  $\log \rho$  versus  $1/T$  data are plotted in the figure 2.

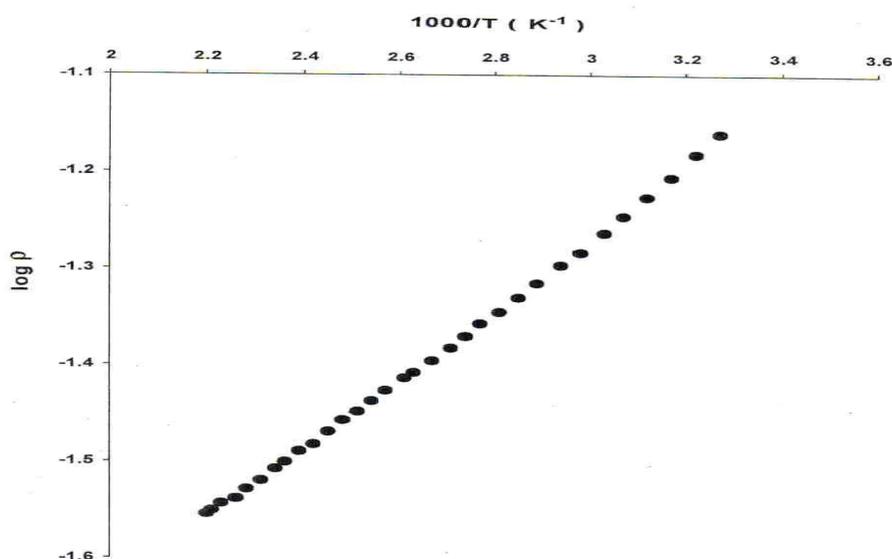


Figure 2: Log  $\rho$  versus  $1/T$  plot of  $W_{0.5}Si_{0.5}Se_{1.95}$

### Thermal Analysis

The phase has been thermally analyzed for thermogravimetric (TG) and differential thermal analysis (DTA) by Rigaku thermalAnalysis System 8150, provided with a microprocessor, in the temperature range 300K-873K at the heating rate of 10 deg./min in continuous flow of nitrogen [20, 21]. The TG and DTA plot is given in figure 3.

## 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 orthorhombic unit cell ( $a=16.973\text{\AA}$ ,  $b=12.981$  and  $c=8.609\text{\AA}$ ). In order to determine the crystal structure, the theoretical X-ray diffraction data were generated by Treor and Lazy-Pulverix analysis. The  $d_{\text{cal}}$  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.

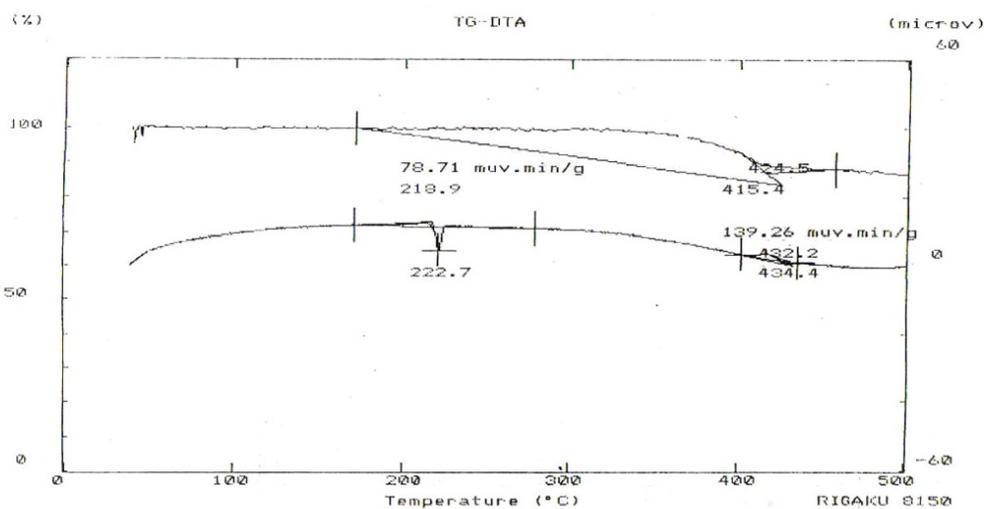


Figure 3: TG-DTA Curves of  $W_{0.5}Si_{0.5}Se_{1.95}$

Table 2: Powder X-ray Diffraction Data of  $W_{0.5}Si_{0.5}Se_{1.95}$

<b>h</b>	<b>k</b>	<b>l</b>	<b>d<sub>obs</sub> (Å)</b>	<b>d<sub>cal</sub>(Å)</b>	<b>I<sub>obs</sub></b>
1	1	1	6.608	6.613	100.0
0	2	0	6.494	6.496	7.7
0	1	2	4.087	4.089	1.4
3	2	1	3.820	3.824	2.6
4	2	1	3.278	3.285	0.3
0	4	1	3.040	3.039	2.9
0	0	3	2.869	2.872	17.1
0	1	3	2.799	2.804	2.5
0	2	3	2.625	2.626	5.3
0	3	3	2.392	2.393	29.1
2	5	1	2.387	2.387	26.5
2	0	4	2.087	2.087	0.6
8	2	0	2.017	2.018	0.3
0	3	4	1.927	1.928	17.1
3	2	4	1.921	1.923	11.5
7	2	3	1.780	1.782	0.3
9	3	0	1.729	1.730	1.7
1	5	4	1.648	1.650	6.3
5	7	0	1.627	1.628	11.7
7	1	4	1.597	1.598	4.6
0	7	3	1.557	1.558	0.4
0	1	6	1.426	1.427	1.3
0	8	3	1.412	1.413	5.3
3	5	5	1.391	1.392	0.4

a = 16.973Å

b = 12.981Å

c = 8.609Å

**Table 3:** Specific resistance ( $\log \rho$ ) of  $W_{0.5}Si_{0.5}Se_{1.95}$  as function of temperature (K).

Temperature (K)	Specific resistance $\rho$ (ohm cm)
454	0.027
452	0.028
447	0.028
442	0.028
437	0.029
432	0.030
427	0.031
422	0.031
417	0.032
412	0.032
407	0.033
403	0.034
398	0.035
393	0.036
388	0.037
383	0.038
379	0.039
374	0.040
369	0.041
364	0.042
360	0.043
355	0.045
350	0.046
345	0.048
340	0.050
335	0.052
330	0.054
325	0.056
320	0.059
315	0.062
310	0.065
305	0.069

#### ***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 electrical resistivity of the phase ( $W_{0.5}Si_{0.5}Se_{1.95}$ ), as function of temperature, is plotted in figure 2. The negative temperature co-efficient of resistivity and the values of the specific resistance (Table.3) suggest that the phase is semi-conductor in nature and the electrical conduction occurs via thermal activated mechanism.

**Table4:** Magnetic and Electric Transport Parameters of ( $W_{0.5}Si_{0.5}Se_{1.95}$ ) phase.

Phase	$\mu_{\text{eff}}$ (B.M)	$\mu_{\text{theo}}$ (B.M)	$E_a$ (eV)
$W_{0.5}Si_{0.5}Se_{1.95}$	Diamagnetic	-	0.07

**Thermal Analysis:**

The thermogravimetric analysis (TGA) of  $W_{0.5}Si_{0.5}Se_{1.95}$  fig. 3 suggests that there is almost complete mass stability upto 673 K and beyond this temperature there is mass loss of 11.88%. in the region where mass of the phase remains is almost constant, there is very sharp endothermic peak between 491.9 K and 497 K with peak temperature at 495.7 K.

**Conclusion**

A new phase with the composition  $W_{0.5}Si_{0.5}Se_{1.95}$  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 orthorhombic 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. The thermal analysis suggests that upto 673 K there is almost complete mass stability of the phase, while beyond this temperature there is mass loss of 11.88 %.

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