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ELECTRICAL TRANSPORT PROPERTIES OF SOME MIXED TRANSITION METAL DICHALCOGENIDES $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ ($x = 0.3, 0.4, 0.85$ & 0.9)

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ABSTRACT

The mixed transition metal dichalcogenide single crystals, $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ series, taking $x = 0.3, 0.4, 0.85$ and 0.9 were successfully grown by direct vapor transport (DVT) techniques. The variation of resistivity with temperature was studied along perpendicular and parallel to c-axis using four probes and two probe techniques respectively. The Hall measurements at ambient temperature determined the conductivity type and carrier concentration of the as-grown crystals. The Seebeck coefficient variation with temperature was carried out. The obtained results are discussed in details.

Key words: $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ ($x = 0.3, 0.4, 0.85$ & 0.9), resistivity, Hall Effect, thermoelectric power.

INTRODUCTION

The transition metal dichalcogenides (TMDC's) has the general formula MX_2 , where 'M' is a transition metal ($M = \text{W}, \text{Mo}$) from IVB, VB and VIB group of the periodic table and 'X' is one of the chalcogens namely S, Se and Te. Its basic structure is loosely couple X-M-X sheets which makes the material extremely interesting, because within a layer, the bonds are strong while between the layer they are remarkably weak [1-3].

Significant optical to electrical/ chemical energy conversion efficiencies have been obtained in solid state photovoltaic and photoelectrochemical solar cells with TMDC semiconductor-electrolyte interface [4-6]. It is essential that semiconductor surface should be non-corroding and stable under conditions of illumination and environment of electrolyte. It is also important that the chosen semiconducting materials should have a band gap in the range of 1.1 to 2.1 eV to provide good optical matching for better solar energy conversion. Materials which possess both these properties are transition metal dichalcogenides (TMDCs), like MoS_2 , MoSe_2 , WS_2 , WSe_2 , etc. Lots of work has been carried out on these TMDCs [7-19]. Literature shows that very limited amount of work has been carried out on mixed transition metal dichalcogenides like $(\text{Mo}/\text{W})\text{Se}_2$, thus the authors thought of growing such mixed $(\text{Mo}/\text{W})\text{Se}_2$ in the form of single crystals and study the transport properties. In this research paper, growth of single crystals is reported and the electrical transport properties studies on these single crystals were studied and the results are discussed in details.

MATERIALS AND METHODS

Crystal Growth

Among TMDC's the compound with larger thermoelectric power, high electrical conductivity and low thermal conductivity posses excellent properties as a thermoelectric material. Number of investigators have carried out such measurements on tungsten diselenide and molybdenum diselenide single crystals, but limited literature exist on such measurements being carried out on $(\text{Mo}/\text{W})\text{Se}_2$ mixed single crystals. Therefore, authors have tried to see how these properties would be affected with increasing amount of molybdenum in WSe_2 and henceforth concentrated on four combination in $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ series, taking $x = 0.3, 0.4, 0.85$ and

0.9 . The initial materials taken for single crystals growth were in the powder form with following purities; Mo: 99.99%, W: 99.99% and Se: 99.99%. Firstly for compound preparation, stoichiometric amounts of the powder were introduced into a thoroughly cleaned quartz ampoule. The total charge taken in each case was about 10 gm. The ampoule was sealed at vacuum of 10^{-5} Torr. After vacuum sealing, the mixture was thoroughly mixed by vigorous physical shaking. Then the homogeneous mixture was evenly distributed along the length of the ampoule and was placed in the furnace. The temperature of the furnace was increased slowly to avoid any explosion, which might occur due to the strong exothermic reactions between the elements. In all the cases, the ampoule was then maintained at 1073 K for 40 hours to allow the complete reaction to occur which resulted in a polycrystalline form of $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ ($x = 0.3, 0.4, 0.85$ and 0.9) compounds. The obtained powder compound was then transferred to another growth ampoule and sealed at vacuum of 10^{-5} Torr. The complete powder compound was kept at one end of the ampoule known as charge end and the other end of the ampoule is the growth end where the crystal growth takes place. The details of the temperatures of the charge zone, growth zone, growth time, rate of heating, rate of cooling and size of the single crystals for each case is tabulated in Table - 1.

The grown single crystals of $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ ($x = 0.3, 0.4, 0.85$ and 0.9) series were compositionally and structurally characterized by EDAX and XRD techniques respectively. The compositional and structural results were in good agreement with the reported results [20].

RESULTS

Resistivity \perp c-axis

The resistivity variations with temperature in the range from ambient to 423 K were studied on the grown single crystals using conventional four probe method. This resistivity variation was perpendicular to c-axis (\perp c-axis). The resistivity at each temperature was evaluated by using the formula,

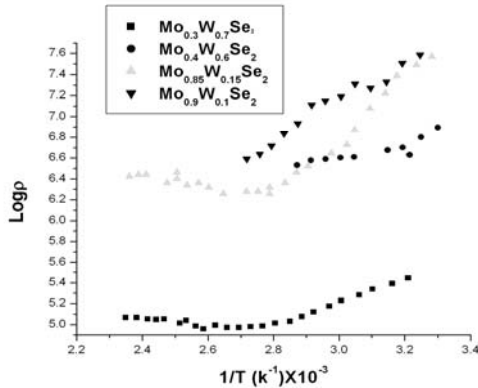
$$\rho = \frac{2\pi sR}{l} \quad (1)$$

where s is the distance between two probes, and 'R' is the resistance between two probes. The resistivity data obtained from the above equation are plotted as a function of inverse of temperature and are shown in Fig. 1 for $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ ($x = 0.3, 0.4, 0.85$ and 0.9) series.

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Table - 1. Growth parameter of $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ ($x = 0.3, 0.4, 0.85$ and 0.9) single crystals.

Crystals	Temperature (K)			Rate of heating (K s^{-1})	Rate of cooling (K s^{-1})	Time (hr.)	Crystal size (mm^3)
	Compound	Charge zone	Growth zone				
$\text{Mo}_{0.3}\text{W}_{0.7}\text{Se}_2$	1073	1423	1373	50	40	192	15 x 11 x 0.2
$\text{Mo}_{0.4}\text{W}_{0.6}\text{Se}_2$	1073	1373	1323	50	40	192	17 x 14x0.2
$\text{Mo}_{0.85}\text{W}_{0.15}\text{Se}_2$	1073	1363	1313	50	40	192	20x13x0.2
$\text{Mo}_{0.9}\text{W}_{0.1}\text{Se}_2$	1073	1296	1246	50	40	192	12x7x0.15

**Fig. 1** Logp vs. $1/T$ graph for \perp c-axis by four probe method

The activation energy is determined from the above plots [Fig. 1] using equation,

$$E_a = 2.303 \times k \times \text{slope (eV)} \quad (2)$$

where k is Boltzmann constant. For all the samples of $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ ($x = 0.3, 0.4, 0.85$ and 0.9), the \perp c-axis resistivity decreased with increase of temperature analogous to semi-conducting behavior. The activation energy values determined from the Fig. 1 is tabulated in Table 2. The values show that the activation energy increases with increase of molybdenum content.

Resistivity \parallel c-axis

High temperature resistivity parallel to c-axis measurements were performed in the temperature range ambient to 773 K for all the single crystal samples, $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ ($x = 0.3, 0.4, 0.85$ and 0.9), by two probe method. The resistivity at each temperature was evaluated by using the formula,

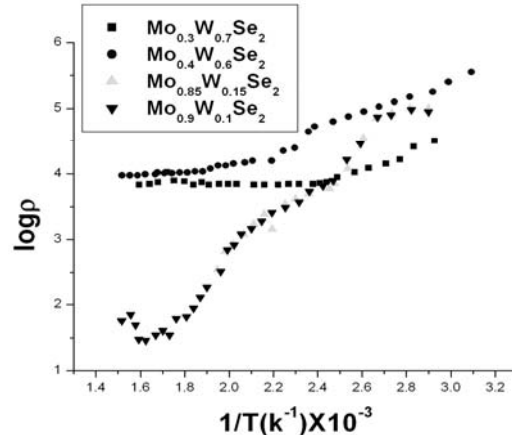
$$\rho = \frac{RA}{t} \quad (3)$$

where A is the area of the specimen, R is the resistance between two probes and t is thickness of the specimen. The resistivity data obtained from the above equation (3) as a function of inverse of temperature are plotted in Fig. 2 for $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ ($x = 0.3, 0.4, 0.85$ and 0.9).

The activation energy values were determined from the graph of Fig. 2 with the help of equation (2) and are tabulated in Table - 2.

Here also for all the samples of $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ ($x = 0.3, 0.4, 0.85$ and 0.9), the \parallel c-axis resistivity decreases with increase of temperature confirming semi-conducting nature. The activation energy values determined from Fig. 2 are tabulated in Table 2. The activation energy values increases

with increase of molybdenum content, similar to the results obtained for \perp c-axis measurements.

**Fig. 2** Logp vs. $1/T$ graph for \parallel c-axis measured by two probe method**Table - 2** The activation energies calculated from \parallel and c-axis

Logp vs $1/T$.

Samples	Activation Energy (\parallel c-axis) E_a (eV)	Activation Energy (\perp c-axis) E_a (eV)
$\text{Mo}_{0.3}\text{W}_{0.7}\text{Se}_2$	0.21	0.09
$\text{Mo}_{0.4}\text{W}_{0.6}\text{Se}_2$	0.21	0.13
$\text{Mo}_{0.85}\text{W}_{0.15}\text{Se}_2$	0.54	0.17
$\text{Mo}_{0.9}\text{W}_{0.1}\text{Se}_2$	0.55	0.28

Here also for all the samples of $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ ($x = 0.3, 0.4, 0.85$ and 0.9), the \parallel c-axis resistivity decreases with increase of temperature confirming semi-conducting nature. The activation energy values determined from Fig. 2 are tabulated in Table 2. The activation energy values increases with increase of molybdenum content, similar to the results obtained for \perp c-axis measurements.

Hall Effect

The Hall measurements were carried out on $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ ($x = 0.3, 0.4, 0.85$ and 0.9) single crystals employing 11 KG electromagnet. The semiconductor type and carrier concentration were evaluated for all the crystals. The obtained values are tabulated in Table - 3.

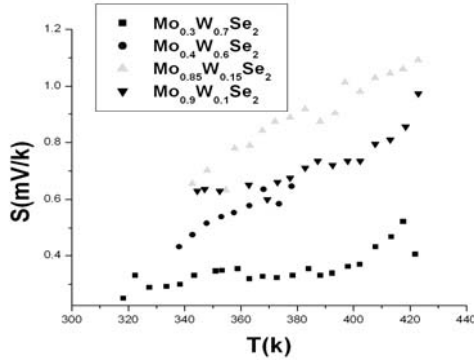
The Hall measurements showed that all the samples, $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ ($x = 0.3, 0.4, 0.85$ and 0.9) are p-type in nature and their carrier concentration comes out to be of the order of 10^{15} cm^{-3} .

Table - 3 Hall data of $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ ($x = 0.3, 0.4, 0.85$ and 0.9) single crystals.

Samples	Carrier Concentration p (cm^{-3})	Type
$\text{Mo}_{0.3}\text{W}_{0.7}\text{Se}_2$	1.52×10^{15}	P
$\text{Mo}_{0.4}\text{W}_{0.6}\text{Se}_2$	0.16×10^{15}	P
$\text{Mo}_{0.85}\text{W}_{0.15}\text{Se}_2$	0.22×10^{15}	P
$\text{Mo}_{0.9}\text{W}_{0.1}\text{Se}_2$	0.83×10^{15}	P

Thermoelectric Power

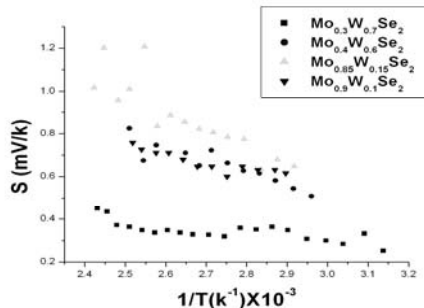
The thermoelectric power measurements have been carried out as a function of temperature for as grown $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ ($x = 0.3, 0.4, 0.85$ and 0.9) single crystals starting from ambient to 423 K with experimental set up TPSS-200, Scientific Solution, Mumbai. The variation of thermoelectric power 'S' for single crystals of $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ ($x = 0.3, 0.4, 0.85$ and 0.9) at different temperatures are shown in Fig. 3.

**Fig. 3** Plot of Seebeck coefficient(S) vs. temperature (T).

It has been seen that the thermoelectric power 'S' increases with increase in temperature for all samples. To study the temperature dependence of the thermoelectric power of a p-type semiconductor the below expression [21] can be used,

$$S = \frac{k}{e} \left[A + \frac{E_F}{kT} \right] \quad (4)$$

where k is the Boltzmann constant, e is the electronic charge, A is the constant determined by the dominant scattering process and E_F is the separation of the Fermi level from the top of the valence band. For a small temperature range, E_F is fairly constant and hence from equation (4) if thermoelectric power (TEP) is plotted against the reciprocal of temperature, a straight line is expected from where E_F and A can be determined, from the slope and intercept respectively. Fig. 4 shows the variation of TEP with an inverse of temperature for $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ ($x = 0.3, 0.4, 0.85$ and 0.9) single crystals.

**Fig. 4** Plot of Seebeck coefficient (S) vs. reciprocal of temperature ($1/T$).

The values of E_F and A from the slope and intercept, which were determined are listed in Table 4 for each samples

respectively. The effective density of states N_A for the as-grown single crystals were determined using relation,

$$S = \frac{k}{e} \left[A + \ln \frac{N_A}{p} \right] \quad (5)$$

where N_A is the effective density of states and is given by,

$$N_A = 2 \left[\frac{2\pi m_h^* kT}{h^3} \right] \quad (6)$$

where m_h^* is the effective mass of holes. Using the values of carrier concentration obtained from Hall effect measurements, the effective density of states N_A for the samples of $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ ($x = 0.3, 0.4, 0.85$ and 0.9) were calculated with the help of the formula,

$$p = N_A \exp \left(\frac{-E_F}{kT} \right) \quad (7)$$

Here E_F is the Fermi energy, k is the Boltzmann constant and T is the room temperature. The values of effective density of states N_A thus obtained are listed in Table 4 for the samples $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ ($x = 0.3, 0.4, 0.85$ and 0.9). Using these values of effective density of states N_A in equation (6), the effective mass of holes for $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ ($x = 0.3, 0.4, 0.85$ and 0.9) single crystals has been calculated.

Table - 4 Values of constant A , scattering parameter s , Fermi energy E_F , effective mass m_h^* and effective density of states N_A of $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ ($x = 0.3, 0.4, 0.85$ and 0.9) single crystals.

Sample	A	Fermi energy E_F (eV)	$s = (5/2) - A$	N_A (cm^{-3})	m_h^* $\times 10^{-32}$ (kg)
$\text{Mo}_{0.3}\text{W}_{0.7}\text{Se}_2$	0.48	0.32	2.02	1.52×10^{15}	0.01
$\text{Mo}_{0.4}\text{W}_{0.6}\text{Se}_2$	0.88	0.72	1.62	0.16×10^{18}	0.25
$\text{Mo}_{0.85}\text{W}_{0.15}\text{Se}_2$	0.024	0.80	2.47	0.22×10^{15}	0.31
$\text{Mo}_{0.9}\text{W}_{0.1}\text{Se}_2$	0.013	1.21	2.48	0.83×10^{15}	0.76

The calculated values of the scattering parameter ' s ' lies between 1 and 3 for $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ ($x = 0.3, 0.4, 0.85$ and 0.9) single crystals.

CONCLUSIONS

1. Large size single crystals of $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ ($x = 0.3, 0.4, 0.85$ and 0.9) were successfully grown by direct vapor transport technique.
2. The \perp c-axis resistivity measurements showed that the resistivity decreases with increase of temperature analogous to semi-conducting behavior. The activation energy values determined from the \perp c-axis resistivity vs. inverse of temperature showed that the activation energy values increases with increase of molybdenum content.
3. Similar results were obtained for the \parallel c-axis resistivity measurements. It also showed the resistivity decreased with increase of temperature. The activation energy values determined from the \parallel c-axis resistivity vs. inverse of temperature showed that the activation energy values increases with increase of molybdenum.

4. The Hall measurements showed that all the samples, $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ ($x = 0.3, 0.4, 0.85$ and 0.9) are p-type in nature and their carrier concentration comes out to be of the order of 10^{15} cm^{-3} .
5. For all samples, $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ ($x = 0.3, 0.4, 0.85$ and 0.9) the thermoelectric power 'S' increase with increase in temperature. The calculated values of scattering parameter 's' lies between 1 and 3 for $\text{Mo}_x\text{W}_{1-x}\text{Se}_2$ ($x = 0.3, 0.4, 0.85$ and 0.9) single crystals.

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NOTE: This information may be kindly circulated among your colleagues.