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GROWTH, STACKING FAULTS PROBABILITIES AND HIGH PRESSURE STUDIES ON MOLYBDENUM DISULPHIDE SINGLE CRYSTALS

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Abstract *—MoS_2 is a member of Transition Metal Dichalcogenides (TMDC) group. Single crystals of Molybdenum disulphide (MoS_2) have been grown by a chemical vapour transport technique using iodine as transporting agent. The composition of the grown crystals was confirmed on the basis of Energy Dispersive Analysis by X-ray (EDAX) and remaining structural characterization was accomplished by X-ray diffraction studies. Lattice parameters, volume and X-ray density have been determined for the grown crystals. The particle size for number of reflections has been calculated using Scherer's formula. The behavior of the Resistance under pressure is thoroughly studies using Bridgman anvils. The variation in the resistance with increase in pressure fir M_OS_2 has been carried out up to 6.5 GPa.*

Keywords*-Transition Metal Dichalcogenides, M_OS_2 Single crystals, EDAX, XRD and High pressure.*

I. INTRODUCTION

MoS_2 is a member of transition metal dichalcogenides group, which possesses layered structure. The TMDC group attracted many research workers on account of the interesting properties of the compounds of this family [1-4]. Moreover, TMDCs are found to decompose below their melting point and also are insoluble in water, hence the vapour pressure transport technique is used for growing single crystals of these compounds. Here, we have grown single crystals of MoS_2 using chemical vapour transport technique using iodine as transporting agent in order to get single crystals HIGH dimensions.

Nano size amorphous powder of molybdenum disulphide has been preparing al by γ-irradiation method in ambient pressure and at room temperature [5]. The structure of surface modified MOS_2 nanoparticles by dialkyldithiphosphate (DPP) molecules was investigated by XRD, XPS, TEM and HREM [6]. Single molecular layer M_OS_2 and WS_2 suspensions prepared by exfoliation provide excellent randomly oriented two dimensional systems for demonstrating the unique features of X-ray powder diffraction patterns of two dimensional materials and for structure identification using Bragg peak profile [7]. The crystal structure of synthesized M_OS_2 was observed by high resolution transmission electron microscopy to have a rhombohedral lattice of three sandwich S-Mo-S layer of ABC [8]. The crystal structure of MoS_2 is shown in figure 1.In the recent years there has been a considerable interest in the high pressure behavior of solids because of academic, technological and geophysical reasons. However, an application of high pressure, the atomic arrangement in solids changes and it resulting to changes in inter atomic distances and crystal structure. So, in this context it will be of great importance to study the physical behavior of lamellar solids like MoS_2 under extremely high pressure.

II. EXPERIMENT (GROWTH)

The growth process of the MoS_2 single crystals are as follows:

(a) Ampoule cleaning: The Best quality fused tube was washed initially using distilled water and heated with hydro fluoric acid (HF) which reacts with silica to create roughness. It was again washed distilled water and then with hot mixture of conc. HCl and conc HNO_3 taken in equal proportion. It was again washed with distilled water to make the inner surface free from any residue of chemicals. Such a cleaned ampoule was kept in a sico oven at 100° C and left over night to make it moisture free.

(b) Compound Preparation: - A 10 gm mixture of Mo (99.95%) and S (99.95% pure) was filled in the dried ampoule. Iodine of the quantity 2 mg / cc of the ampoule volume was sealed in thin capillaries and placed in the ampoule as transporting agent. Then the ampoule was sealed at the pressure of 10^{-5} torr. The sealed ampoule was placed in to the two zone furnace at a constant reaction temperature to obtain the charge of MoS_2.

The Growth parameters for MoS_2 single crystals are as shown in table 1. The chemical composition of the grown samples has been well confirmed by carrying out EDAX analysis as shown in table. 2. For X-ray diffraction work, several small

crystals were finely grinded with the help of an agate mortar and separated through 100 micron sieve to obtain of nearly equal size. The powder obtained during the growth process was used for the X-ray diffraction study. The X-ray diffractograms were obtained by Philips X-ray Diffractometer PW 1820 employing $CuK_{\propto}$ radiation. The X-ray diffraction pattern obtained for MoS_2 is shown in figure 2. Pattern consists of well-defined sharp diffraction Lines, indication good crystallinity of the specimen.

It is established fact that Pressure in an important physical variable in material science. The Development of high pressure techniques has led, in particular, during the last Five decades advances in fundamental scientific research and to industrial application. Electrical Measurements are among the most ancient methods used for characterization for the sample at high pressure. In Bridgman opposed-anvil system [9], the decrease in volume and therefore the maximum pressure, is limited by the flow of deformable gaskets in various geometries. The sample is Compressed between supported tungsten carbide (WC) anvils. It is contained by the phyrophyllite gasket [10.]. The design makes use of the massive support mechanism.

III. RESULTS

The gray black single crystals of MoS_2 were obtained by chemical vapour transport (CVT) technique. The crystal structure of MoS_2 crystal is as shown in figure 1. Figure 3 shows the energy dispersive spectra of MoS_2 single crystals. The values of the lattice parameters a and c, the volume (V) and X-ray density (ρ) obtained from the analysis of the diffractograms are presented systematically in table 3. Values obtained by Traill, (1963) [11] have also been listed for comparison in table 3. The X-ray data for MoS_2 was used for the estimation of particle size using Scherer's formula [12].

$$t = \frac{K\lambda}{\beta \cos\theta}$$

Where t is the crystallite size as measured perpendicular to the reflecting plane,K the Scherer constant whose value is taken to be unity assuming the particles to be spherical, λ the wave length of X-ray radiation, β the half intensity which measured in radians and θ is the Bragg angle. The (hkl) values corresponding to prominent reflections, d- values, half width, peak intensities and particle size for MoS_2 single crystal are shown in table 4. The particle size calculated for reflections (104), (107) and (202) are given in table 5. Half widths of (hol) reflections with odd and even values of l can be used to make a realistic estimation of growth and deformation fault probabilities in MoS_2 by using the formulae

$$3\alpha + 3\beta = \frac{\beta_{2\theta}\pi^2 c^2}{360 \tan\theta\, l^2 d^2} \quad \text{For l = even}$$

$$3\alpha + \beta = \frac{\beta_{2\theta}\pi^2 c^2}{360 \tan\theta\, l^2 d^2} \quad \text{For l = odd}$$

Where $\beta_{2\theta}$ denotes the full width at half the maximum intensity, d is the hkl spacing , c is equal to $2_{d002,}$ αis the probability for deformation fault and β is the probability for growth fault. Values of α and β obtained from (1 0 4), (107) and (202) reflections for MoS_2 are presented in table 5. Using bridgman anvils we achieved pressure up to 6.5 GPa. As shown in figure 4, resistance decreased continuously as pressure increased. No phase transition is found in these crystals up to 6.5 GPa and with increasing pressure the samples are becoming more conducting in nature.

IV. CONCLUSIONS

1] The large single crystals size of MoS_2 have been grown successfully using a chemical vapor transport technique with I_2 as a transport material.
[2] The XRD analysis shows that MoS_2 possesses hexagonal crystals structure.
[3] The EDAX studies confirmed that MoS_2 single crystals are stoichiometrically perfect.
[4] Resistance decreases continuously as pressure increased. No phase transition is found up to 6.5 GPa and with increasing pressure the sample becoming more conducting in nature.

Charge Preparation		Growth Condition			Dimension of the ampule			Crystal Color
Charge Temperature K	Time hour	Hot Zone K	Cold Zone K	Time hour	Inner Diameter mm	Outer Diameter mm	Length mm	
973	36	1130	1073	336	22	25	250	Grey Black

Table1: Growth Condition for MoS_2 Single Crystals

Composition	Wt (%) of elements obtained from the EDAX	
	Mo	S
MoS_2	62.19	37.81
$MoSe_2$	38.00	62.00
Taken	37.80	62.20

Table 2: The EDAX data for MoS_2 Single Crystals.

Sample	a=b (A°)	c (A°)	Volume $(A°)^3$	X-ray Density (gm/cm^3)
MoS_2	3.11 ± 0.04057	18.89 ± 0.6456	158.22	5.0417
$MoSe_2$	3.157 ± 0.000983	12.926 ± 0.1958	111.565	7.545
Value obtained by Traill (1963) [11]	3.287	12.925	….	….

Table 3: Results obtained from X-ray diffractograms.

hkl	d- spacing	Perk width ($°2\theta$)	Relative Intensity (%)	Peak Intensity (Counts/ sec)	Particle Size
003	6.1336	0.36	100	22965.48	424.35
006	3.0674	0.36	8.83	2028.09	413.66
104	2.2723	0.30	0.37	85.37	546.29
009	2.0457	0.36	57.42	13189.68	396.12
107	1.8262	0.30	0.55	125.20	566.79
110	1.5763	0.36	0.12	27.26	373.02
113	1.5355	0.36	64.21	27.26	370.02
202	1.3389	0.36	0.19	43.75	523.28
211	1.034	0.48	1.13	259.26	213.99

Table 4: X-ray diffraction data for MoS_2 single crystals.

$3\alpha + 3\beta$ (even)	$3\alpha + \beta$ (odd)	α	β
0.8449	0.2955	0.01386	0.2747

Table 5: Stacking fault probabilities for MoS_2 single crystals.

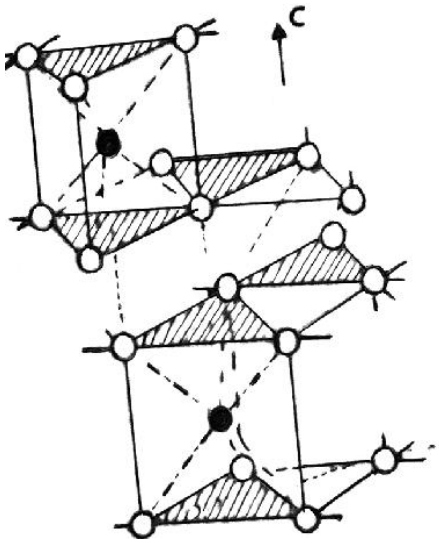

Figure 1: Crystal structure of MoS_2 single crystal

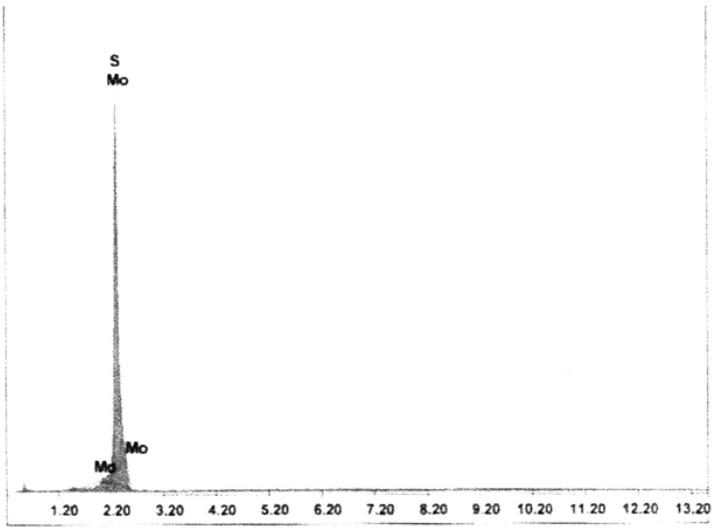

Figure 2. EDAX of MoS_2 single crystal

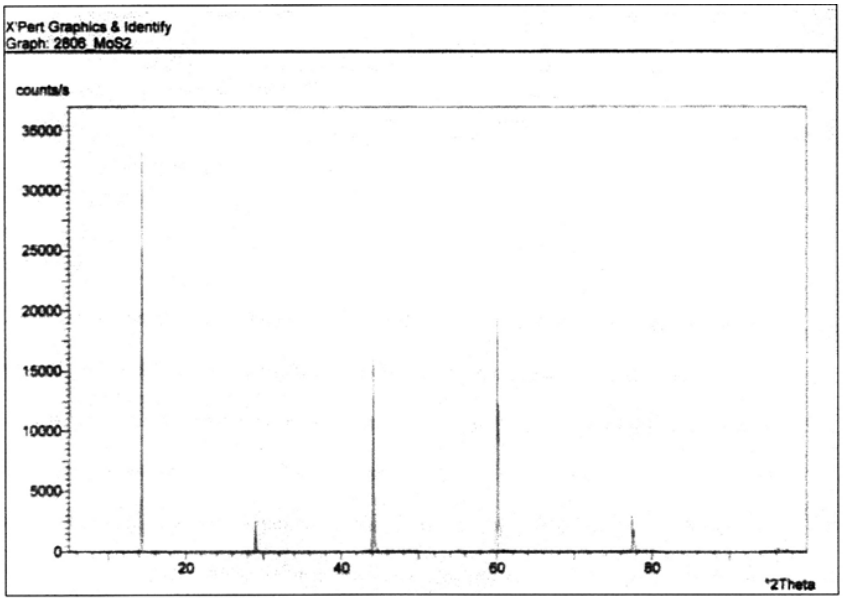

Figure 3. XRD of MoS_2 single crystal

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