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Effect of pressure on electrical resistance of WSe_2 single crystal

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Abstract. The results of electrical resistance measurements under pressure on single crystals of WSe_2 are reported. Measurements up to 8.5 GPa are carried out using Bridgman anvil set up and beyond it using diamond anvil cell (DAC) up to a pressure of 27 GPa. There is no clear indication of any phase transition till the highest pressure is reached in these measurements.

Keywords. Pressure dependence of resistance; transition metal dichalcogenides; WSe_2 single crystal; Bridgman anvil; diamond anvil cell.

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1. Introduction

Lamellar solids of transition metal dichalcogenides (TMDC) MX_2 ($M =$ Mo, W, Nb, Ta and $X = S$, Se, Te) have been extensively studied because of their excellent lubricating properties and photovoltaic behaviour. These materials being semiconducting and of layered structure, may undergo structural and electronic transitions under pressure. They typically have a hexagonal structure, which is composed of two-dimensional sheets stacked on top of one another. Each sheet is tri-layered with a metal atom in the middle that is covalently bonded to chalcogen atoms in the top and bottom layers. The covalently bonded sheets are held together by weak van der Waals forces, which permit them to shear easily. At high pressures the fluid lubricants squeeze out from in-contact surfaces, causing high friction and wear. With lamellar solids such as TMDCs, shearing takes place more easily when loads are high. So lamellar solids are well-suited to extreme pressure lubrication. Structural transitions and metallization are of relevance in its application as lubricant. In the present work, we have investigated the high pressure behaviour of one such lamellar solid, i.e., WSe_2 for pressure induced phase transition by electrical resistance measurements.

2. Experimental technique

Single crystals of WSe_2 were grown by direct vapour transport technique using stoichiometric proportion of tungsten (Koch-Light Laboratories Ltd., England, purity: 99.95%) and selenium powder (Koch-Light Laboratories Ltd., England, purity: 99.99%). Ten gram of the powdered compound was introduced into a quartz ampoule. The ampoule was then evacuated to a pressure of about 10^{-5} torr and sealed. Ampoule was placed coaxially and horizontally in a two-zone furnace with powder in the region of the hotter section of the furnace. The temperature of the hot and cold zones for crystal growth was 1080°C and 1020°C respectively. The ampoule was kept in this temperature gradient of the furnace for 10 days. At the end of this period the furnace was allowed to cool slowly at the rate of 50°C/h to room temperature. Large shining single crystals of WSe_2 were obtained by this technique. Energy dispersive analysis of X-ray (EDAX) gave confirmation about the stoichiometry of the grown crystals. Initially the pressure variation of electrical resistance measurements were carried out using Bridgman opposed anvil apparatus [1] up to 8.5 GPa where we have good control of pressure while increasing or decreasing. The sample is contained in a pyrophyllite gasket with talc as pressure transmitting medium [2]. Pressure was calibrated with bismuth transitions at 2.5 and 7.65 GPa. A four probe method is used to obtain the resistance of the WSe_2 sample up to 8.5 GPa [3]. However the pressure is limited to approximately 10 GPa using this apparatus.

We have extended these measurements up to 27 GPa using recently standardized technique of electrical resistance measurements using DAC [4,5]. All over the world, diamond anvil cells (DACs) are being used for experiments involving high hydrostatic pressures. This is because of their small external dimensions, which make them compatible with most other equipments and also because of the very high pressures that can be obtained routinely and safely [6].

A pre-indented stainless steel gasket with 400 μm hole stuffed with alumina powder of 0.05 μm grain size was used to contain the sample. This layer of alumina helps to insulate the sample and leads from the gasket. Two 20 μm parallel stainless steel wires centered on one of the anvils, serve as leads for quasi four probe measurements. Fine powder of ruby filled between the wires was employed for pressure measurements using ruby fluorescence technique [7–9]. Thin flakes of crystal sample are kept over the wires. As the cell is slowly tightened, the voltage across the small segment of ($\sim$20 μm) the sample is measured by passing a small amount of constant current. The full details of the technique are given elsewhere [5].

3. Results and discussion

Pressure variation of electrical resistance using Bridgman anvil apparatus and DAC are shown in figures 1 and 2 respectively. As seen in the figures, the resistance decreases continuously as pressure is increased till the highest pressure of these measurements is reached. The changes in resistance observed by Bridgman anvils up to 8.5 GPa are well-reproduced with the DAC. The sample becomes more conducting as pressure increases and around 27 GPa the electrical resistance decreases by a factor of 5. We noticed slight slope change in resistance variation with pressure beyond 20 GPa. However the value of resistance remained quite high in comparison to metallic resistance. Hence we rule

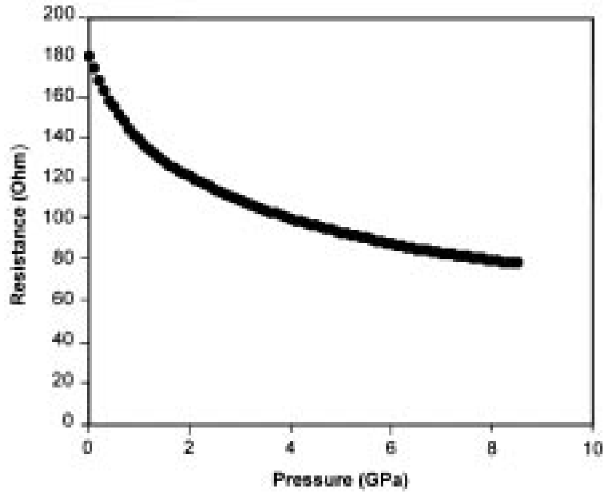

Figure 1. Pressure variation of electrical resistance of WSe_2 using Bridgman anvils.

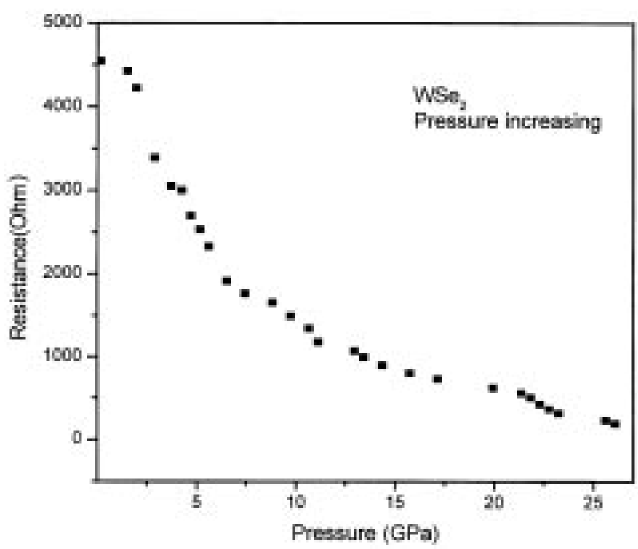

Figure 2. Pressure variation of electrical resistance of WSe_2 using DAC.

out the possibility of semiconductor to metallic transition up to the highest pressure of 27 GPa. However, good quality X-ray powder diffraction employing synchrotron source can identify the origin of slight slope change in resistance variation with pressure beyond 20 GPa. It will be interesting to carry out Raman and X-ray diffraction measurements under pressure to associate the weak anomaly observed in resistance measurements with any structural transition.

4. Conclusion

Good quality single crystals of WSe_2 have been prepared. Its resistance decreases gradually under pressure. There is no clear indication of any structural transition till the pressure of 27 GPa, the highest pressure reached in this work.

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