

Micro hardness of Injection Molded PP/MoS₂ Polymer Matrix Composites

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Abstract- Molybdenum disulphide (MoS₂) filled polypropylene (PP) composites were prepared using the horizontal ball milling machine followed by injection moulding. The content of MoS₂ was varied from 0 to 7.12 vol.%. Experimental density of the composites prepared by horizontal ball milling was close to the theoretical density. Flexural modulus increased by 21% up to 4.28 vol.% MoS₂, then it shows detrimental effect on flexural modulus. Flexural strength also increased from 13 MPa to 28 MPa at 4.28 vol.% MoS₂. The percentage deflection at break of the pure PP sample prepared by injection moulding was higher, as compared to other composites. % deflection at break decreases by 34.95%.

Keywords- Polymer matrix composite (PMC), Polypropylene (PP), Molybdenum disulphide (MoS₂), Injection Moulding, Density, Micro hardness

I. INTRODUCTION

1. POLYMER MATRIX COMPOSITE

Polymer matrix composites are comprised of a variety of short or continuous fibres bound together by an organic polymer matrix in which the strong reinforcing dispersed phase provides high strength and stiffness. They were designed so that the mechanical loads to which the structure is subjected in service are supported by the reinforcement. The function of the matrix is to bond the fibres together and to transfer loads between them. Polymer Matrix Composites are very popular due to their low cost and simple fabrication methods. Use of non-reinforced polymers as structure materials is limited by low level of their mechanical properties, in addition to relatively low strength; polymer materials possess low impact resistance.

Due to the favourable combination of easy process ability and attractive mechanical properties, the use of polymer materials in structural applications has assumed large proportions over the last decades. To ensure proper operation under heavy-duty conditions, these applications have to meet specific requirements regarding quality, safety, and mechanical performance (e.g. stiffness, strength and impact resistance). Mechanical performance is generally optimized by

trial and error until the functional demands of the design are satisfied. Chief among the advantages of PMCs is their light weight coupled with high stiffness and strength along the direction of the reinforcement. This combination is the basis of their usefulness in aircraft, automobiles, and other moving structures. Other desirable properties include superior corrosion and fatigue resistance compared to metals [1].

Reinforcement of polymers by strong fibrous network permits fabrication of Polymer Matrix Composite (PMC) characterized by the following properties High tensile strength, Good rupture resistance, High stiffness, Good corrosion resistance, High Fracture Toughness, Low cost, Good abrasion resistance & Good impact resistance.

The main disadvantages of Polymer Matrix Composites (PMC)

- Low thermal resistance
- High coefficient of thermal expansion.

2. Polypropylene Polymer (PP)

Polypropylene is a plastic polymer of the chemical designation C₃H₆. Polypropylene resins are a general class of thermoplastic produced from propylene gas. Propylene gas is derived from the cracking of natural gas feedstock's or petroleum by-products. Under broad ranges of pressures and temperatures, propylene generally polymerises to form very long polymer chains. However, to make Polypropylene resins with controlled configurations of molecules at reasonably acceptable commercial rates, special catalysts are required [3].

Table 1. Properties of PP

Properties	Units	Homopolymer (H110)
Density	Kg/m ³	905
Tensile Strength	MPa	15-35
Tensile Modulus	GPa	1.4
Elongation at Break	%	150
Hardness	“ R” scale	90
Notched Izod Impact	kJ / m	0.07
Heat Distortion Temp.	MPa / °C	105
Limiting Oxygen Index	%	18

3. Molybdenum Disulfide (MoS₂)

Molybdenum disulfide is a dark gray to black powder (the colour depending on its particle size). Generally molybdenum disulfide in its naturally occurring hexagonal form is chemically very inert. It is insoluble in both oil and water. Molybdenum disulfide is uncreative with most acids, however it is not resistant to attract of hot concentrated sulphuric and nitric acids. Also, molybdenum disulfide dissolved in strong oxidizing agents such as aqua regia. Molybdenum disulfide exists in two crystalline forms, hexagonal and rhombohedral. The hexagonal form is by far the most common and being the only type found in commercial ores. Also, the hexagonal form has been found in synthetic MoS₂. The rhombohedral was first identified in a synthetic material and subsequently found in several natural sources [4].

II. EXPERIMENTAL WORK

High purity commercial grade polypropylene granules (REPOL-110 Homo-polymer) was purchased from Reliance Industries Limited and was grinded to fine powder by liquid nitrogen grinding.

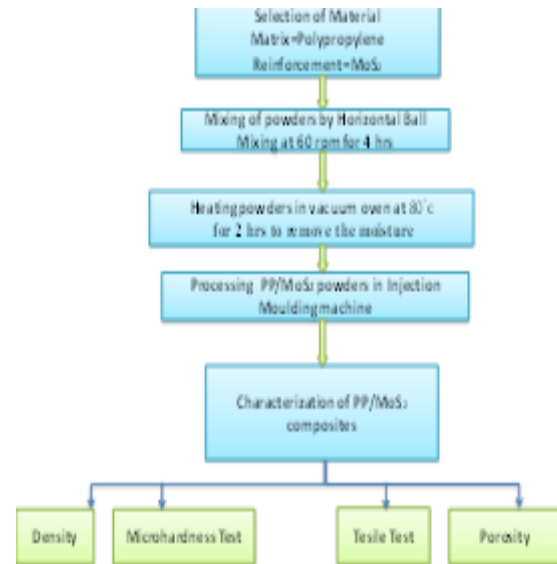


Figure 1. Flowchart of experimental work

III. DENSITY

A high precision electronic balance AFCOSET model ER200A is used to weigh the specimen. Specimen is weighed in air and later when immersed in ethanol medium, its weight is recorded. Theoretical density is calculated by Rule of Mixture. Procedure followed for density calculations are as below,

- The experimental density of the pure and composites can be determined with the help of Archimedes principle.
- The specimen is weighed in air and weight is recorded.
- The same specimen weighed in ethanol medium and weight is recorded.
- Theoretical and experimental density is calculated by equation 6 & 7.



Figure 2. Electronic weighing balance

Determination of wt% and vol % of PP/MoS2 composite

For pure PP, required weight for sample preparation is calculated by

- Mass (m) = Volume (V) * Density (ρ), (gm)
- V = volume of sample, cm³
- P = density, g/cm³
- Density of pure PP = 0.905 g/cm³
- Density of pure MoS₂ = 5.06 g/cm³

For a given weight fraction, the volume fraction of reinforcement can be calculated by following eqn 5

$$V_f = \text{Volume fraction} = \frac{W_f}{\left(W_f + (1 - W_f) \left(\frac{\rho_f}{\rho_m} \right) \right)} \dots \dots \dots \text{Equation 5}$$

W_f = Weight fraction of filler.
 All sample calculations are carried out for PP-1.95 vol % MoS₂ composite

$$\text{Volume fraction, } V_f = \frac{(0.1)}{(0.1) + (1 - (0.1)) \left(\frac{0.905}{5.06} \right)} = 0.01948$$

Determination of theoretical density ρ_{th}, by Rule of Mixtures (ROM)

Theoretical density,
 $\rho_{th} = (\rho_m * V_m + \rho_f * V_f), (g/cm^3)$
Equation 6

- V_m = (1- V_f)
- V_m = Volume fraction of matrix
- V_f = Volume fraction of filler
- ρ_f = density of filler,(g/cm³)

$$\rho_{the.} = (0.905 * (1 - 0.01948)) + (5.06 * 0.01948) = 0.9858 \text{ g/cm}^3$$

Determination of experimental density (ρ_{exp}), by Archimedes principle

The experimental density is obtained by using Archimedes principle.

$$\text{Experimental density, } \rho_{exp} = \left[\frac{W_{air}}{W_{air} - W_{medium}} \right] * \rho_{medium}, (g/cm^3) \dots \dots \dots \text{Equation 7}$$

- ρ_{medium} = Density of ethanol = 0.791(g/cm³)
- W_{air} = Weight of specimen in air (gm)
- W_{med.} = weight of specimen in ethanol (gm)
- ρ_{exp.} = experimental density (g/cm³)

$$\text{Experimental density, } \rho_{exp.} = \frac{(19.333)}{(19.333 - 3.790)} * 0.791 = 0.9820 \text{ g/cm}^3$$

Determination of Porosity

Porosity in the composite is calculated by
 $\text{Porosity} = \frac{(\text{Theoretical density} - \text{Experimental density})}{\text{Theoretical density}} * 100$

$$\text{Porosity} = \frac{(0.9858 - 0.9820)}{0.9858} * 100 = 0.385\%$$

Similarly, Theoretical & Experimental densities are calculated for other compositions

Vickers Micro hardness Test

The Vickers Micro hardness test is often easier to use than other hardness tests since the required calculations are independent of the size of the indenter can be used for all materials irrespective of hardness. The basic principle, as with all common measures of hardness, is to observe the questioned material's ability to resist plastic deformation from a standard source. The Vickers test can be used for all materials and has one of the widest scales among hardness tests. The unit of hardness given by the test is known as the Vickers Pyramid Number (VPN) or Diamond Pyramid Hardness (DPH). The hardness number is determined by the load over the surface area of the indentation and not the area normal to the force range, the Vickers test can be used on almost any material. The part size is only limited by the testing instrument's capacity.

The specification of Vickers Micro hardness Machine are Make: Future-Tech, Model: FM 700, Tokyo, Japan, Load range: 10 – 1000 gm, Loading/ unloading: Automatic, Dwell time: 5 – 60 sec, Magnifications: 10X, 50X and 100X, Specimen height: 65 mm, Test Table: 100 x 100 mm.



Figure 3. Vickers Microhardness machine

Determination of Theoretical Micro hardness

Theoretical Micro hardness,

$$H_v = (1.8544 \cdot P) / d^2, \text{ (kg/mm}^2\text{)} \dots \dots \dots \text{Equation 9}$$

P = applied load in kg.

d = Average indentation diameter in mm.

Experimental Vickers Microhardness is recorded directly from machine.

IV. RESULTS AND DISCUSSIONS

Specimens fabricated by injection moulding machine are characterized for various properties like density, microhardness, tensile strength, impact strength, flexural strength, SEM & limiting oxygen index testing.

Table 2. Composition of PP/MoS2 composites

Specimen Composition	By Weight(% of MoS ₂)	By volume(% of MoS ₂)
Pure PP	0	0.00
PP-0.93 vol % MoS ₂	5	0.93
PP-1.95 vol % MoS ₂	10	1.95
PP-3.06 vol % MoS ₂	15	3.06
PP-4.28 vol % MoS ₂	20	4.28
PP-5.63 vol % MoS ₂	25	5.63
PP-7.12 vol % MoS ₂	30	7.12

1. Density of PP/MoS2 Composites

Theoretical density is calculated by Rule of Mixture while experimental density is calculated by Archimedes principle.

Table 3. Theoretical and Experimental Density of PP/MoS2 Composites

Sr. No.	Composition	Theot. Density (g/cm ³)	Expt. Density(g /cm ³)	Porosity (%)
1	Pure PP	0.905	0.899	0.6629
2	PP-0.93 vol % MoS ₂	0.9435	0.9370	0.680
3	PP-1.95 vol % MoS ₂	0.9858	0.9820	0.385
4	PP-3.06 vol % MoS ₂	1.0316	1.0210	1.06
5	PP-4.28 vol % MoS ₂	1.0826	1.0790	0.332
6	PP-5.63 vol % MoS ₂	1.1384	1.1175	1.835
7	PP-7.12 vol % MoS ₂	1.200	1.1740	2.166

Alumina balls with 5.5 mm diameter size used for milling the PP/MoS2 powder for 4 hours and 200 RPM speed. Ball to composite weight ratio is 10:1, (100 gm composite powder batch size and 10 gm balls were used). Experimental density of PP/MoS2 composite is nearly equal to theoretical density as shown in table 4.2. Alumina balls are used as they have high hardness and inertness; hence no contamination occurs during milling of PP/ MoS2 powder.

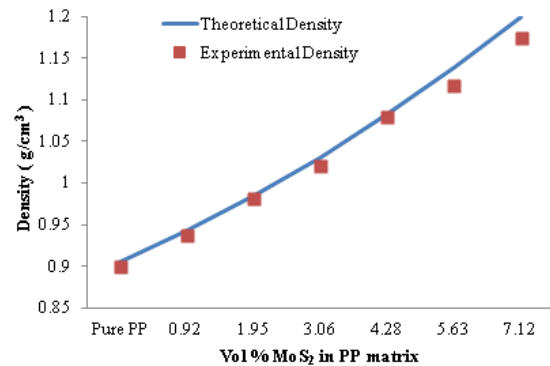


Figure 4. Theoretical and Experimental Density of PP/MoS2 Composites

Fig. 4.1 shows theoretical and experimental densities of the PP/MoS2 composites. The pure PP has theoretical density of 0.905 g/cm³. The density of the composites increased with increasing MoS₂ content. It is due to the higher density (5.06 g/cm³) of MoS₂ than that of PP. The experimental density of the composites containing up to 4.28

vol % MoS₂ is close to that of theoretical density. It indicates that the prepared composites are almost porosity free. However, the experimental density of the 5.63 vol % and 7.12 vol % composites is lower than that of the theoretical density by approximately 1.9% and 2.2% respectively. This is due to the formation of MoS₂ aggregates which hinder the infiltration of molten polymer through the aggregates during injection moulding process [19]. More porosity was observed for 5.63 vol % & 7.12 vol % MoS₂ samples.

2. Micro hardness of PP/MoS₂ Composites

Table 2. Composition of PP/MoS₂ composites

Sr. No	Composition	Avg. Microhardness (kg/mm ²)
1	Pure PP	6.7
2	PP-0.93 vol % MoS ₂	7.0
3	PP-1.95 vol % MoS ₂	8.1
4	PP-3.06 vol % MoS ₂	8.4
5	PP-4.28 vol % MoS ₂	9.0
6	PP-5.63 vol % MoS ₂	6.46
7	PP-7.12 vol % MoS ₂	6.4

The micro hardness was measured using Vickers micro hardness tester (Future Tech FM-700, Tokyo, Japan) at a constant load of 100 gm and dwell time of 15 seconds. Five readings were noted at different locations and average value is reported as the micro hardness of the composites as shown in table 4.3.

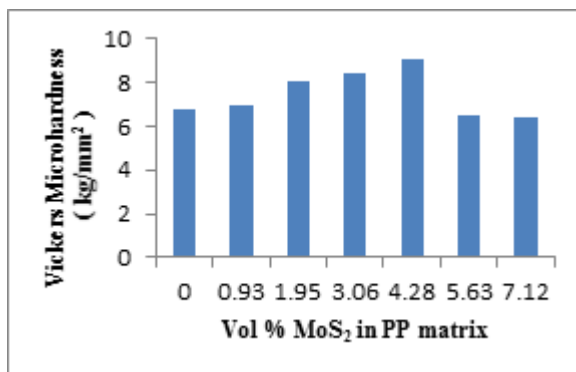


Figure 5. Micro hardness for PP/MoS₂ composites

Fig. 4.2 shows the micro hardness of composites as a function of MoS₂ content. The micro hardness of the composites increased significantly with increasing content of MoS₂ in the matrix. The micro hardness increased from 6.7 kg/mm² for pure PP to 9.0 kg/mm² for PP-4.28 vol % MoS₂

composite. It increased due to the resistance to the plastic deformation of the PP matrix from comparatively hard MoS₂ particles [19]. The significant improvement in microhardness may be attributed to the better distribution of MoS₂ powder in PP matrix. The increase in micro hardness might be attributed to higher micro hardness of MoS₂ compared to pure PP. Further addition of MoS₂ results in decreased value of micro hardness. This was observed due to the more amount of porosity present at 7.12 vol. % of MoS₂

V. CONCLUSION

Polymer matrix composites (PMCs) based on polypropylene (PP) as matrix, and MoS₂ powders as reinforcements were fabricated successfully using horizontal ball milling followed by injection moulding. Experimental density of the PMCs was close to those of theoretical density up to 4.28 vol % loading of MoS₂, indicating porosity free samples. Porosity will be higher i.e 2.1% for 7.12 vol % loading of MoS₂. Micro hardness of PMCs increased by 34% with loading of MoS₂ content up to 4.28 vol %, and then it shows detrimental effect on it.

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