MICRO HARDNESS STUDIES ON TITANIUM OXIDE DOPED PMMA COMPOSITE

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***Abstract*-**The micro-hardness measurement of titanium oxide doped poly (methyl methacrylate) samples is reported in the present work. Poly (methyl methacrylate) samples have been used as the host material in which titanium oxide is added in different weight % of doping (0%,0.0001%, 0.0005%, 0.005%, 0.001%, 0.05%, 0.01%). The sample preparation was done by solution casting method. The objective of the submitted work is to analyze the influence of the load on the micro-hardness of pure PMMA and titanium oxide doped PMMA composites. For the present studies Vicker's micro-hardness test has been used. The sample is subjected to loads of 10-200 grams. The micro-hardness value increases with increasing load up to 100 grams, after which it is constant for all the samples. After this load, micro-hardness tends to be independent of the applied load, also found micro-hardness of the samples increasing with increasing doping percentage of titanium oxide.

***Keywords*-Micro hardness, PMMA, titanium oxide**
PACS Nos61.41._e; 81.70.Bt; 81.40.Ef

1. Introduction

The mechanical behavior of polymers has been the subject of considerable research in the past. Mechanical properties are of relevance for the applications of polymers in industry, optics, electronics, photonics, medicine and others. Hardness is an unusual physical property in that it is the result of a defined measurement procedure and not an intrinsic materials property susceptible to precise definitions in terms of fundamental units of mass, length, and time. The term can apply to deformation from indentation, scratching, cutting or bending. The advantage of this test is that it involves only limited area for measurement so the specimen is relatively unaffected by the test. Hardness has been well established in characterizing metallic material and ceramics for many years, but only recently has it been widely employed for characterizing polymers[1].Hardness has a variety of meanings. To the metals industry, it may be thought of as resistance to permanent deformation. To the metallurgist, it means resistance to penetration [2].The conventional procedure of hardness testing consists of applying a fixed load on a diamond indenter and measuring , with the help of a microscopy , the dimension of the resultant indentation on the surface of the material after unloading. Among the variety of indenter geometries used in hardness testing, theVickers indenter is one in most widespread use. The Vicker hardness test follows the Brinell principle. An indenter of definite shape is pressed into the material to be tested, the load is removed, the diagonals of the resulting indentation are measured and hardness number is calculated [3].This test uses a square pyramid of diamond in which the included angles *a* between non-adjacent faces of the pyramid are 136°. The hardness is given by

$$\mathrm{HV} = \frac{2\mathrm{P}\sin\left(\frac{\alpha}{2}\right)}{\mathrm{d}^2} = 1.854\ \frac{\mathrm{P}}{\mathrm{d}^2} \ldots. (1.1)$$

where*P* is the force in newtons and *d* is the mean diagonal length of the impression in millimetres. The value of $H_V$is expressed in megapascals. The force is usually applied at a controlled rate, held for 30 s, and then removed [4-7].

2. Materials

2.1 Polymethyl methacrylate (PMMA)

Among polymer materials, PMMA is well known as a polymeric glass with a wide range of applications. Use of PMMA offers two fold advantages such as availability to carboxylate functional group for a chemical bonding with the metal ions and high solubility of PMMA in solvent. The material was developed in 1928. Poly (methyl methacrylate) PMMA is a polymer with several interesting physical properties, which are very useful in technical applications. Also PMMA is a widely used material in various engineering applications and its thermal properties are widely known from various studies and engineering databases [8]. Polymethyl methacrylate (PMMA) has been the most popular material for construction of dentures since the 1930s due to many advantages, including good aesthetics, accurate fit, stability in the oral environment, easylaboratory and clinical manipulation, and inexpensive equipment [9]. The PMMA is almost insulating in nature with high elastic strength and its conducting property can be enhanced by adding either metal oxide or supporting agent.

2.2 Titanium Dioxide

Titanium dioxide, also known as titanium oxide or titania, is the naturally occurring oxide of titanium, chemical formula TiO2. Itis a fine white powder pigment, has a good light scattering ability, and therefore a good whiteness, high color strength, hiding power, strong at the same time have a high chemical stability and good weather ability, non-toxic tasteless, no stimulating effect on the human body.

3.Preparation of pure PMMA and their polymer composite film samples

Polymer composites were prepared by doping different concentrations i.e. 0.0001%, 0.0005%, 0.001%, 0.005%, 0.01% & 0.05% wt% of titanium oxide in PMMA by solution casting method.

4. Procedure

The PMMA samples have been used as the host material in which titanium oxide is added in different wt% doping (0.0001%,0.0005%,0.005%,0.001%,0.05%,and .01%). In The present work "Vaiseshika" Micro Hardness Tester (Type: 7005) has been used.The measurement of Vickers hardness provides a Vickers hardness number H_v for the material. The hardness number is defined as the ratio of the load applied to the indenter (gram or kilogram force) divided by the contact area of the impression (square millimeters). The Vickers hardness test method consists of indenting the test material with a diamond indenter. The indenter is in the form of a right pyramid with a square base and an angle of 136 degrees between opposite faces. The sample is subjected to loads of 100-200 gms. The two diagonals of the indentation left on the surface of the material after removal of the load are measured using a microscope and their average is calculated. The area of the sloping surface of the indentation is calculated. The Vickers hardness is the quotient obtained by dividing the kgf load by the square mm area of indentation. In the present investigation the samples were fixed on an optically plane glass in such a way that the surface to be indented was always perfectly horizontal. The plate with the sample is mounted on the stage of the microscope to avoid displacement of the sample. The indenter was kept in contact with the surface for 30 seconds. The length of diagonal of the square was measured through a microscope. The shape of the indenter depends on structure, face and material. A number of measurements were carried out and average of the hardness values was taken.

5 Results and discussion

The result of variation of microhardness with varying ratio of pure and titanium oxide doped pmma with different load and composition are discussed below.

Effect of load

The effect of load on microhardness of the samples has been investigated by taking load values 10gm-200gms. The graph between Vicker's hardness number and applied load for samples with different doping of titanium oxide bywt% (0, 0 .0001, 0.0005, 0.001, 0.005, 0.01 & 0.05 wt.%) at 25ºC (room temperature) are shown in fig1 . From this figure it can be interpreted that the microhardness value increases with increasing load and after initial increase attains a limiting value.Hardness increases with increasing applied indentation test load [10] and after the certain load hardness remain constant[11].The load at which Hv attains a limiting value is 100gms, which is constant for all the samples. After this load, H_V tends to be independent of the applied load. The increase in microhardness may be explained on the basis of strain hardening phenomenon

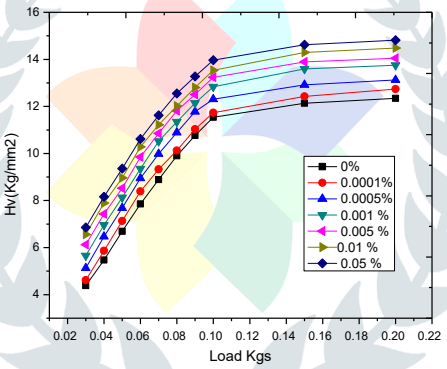

Fig 1 Variation of Hv with load for Pure and Tio2/PMMA composites at room temperature.

Effect of doping

The physical properties of polymers may be affected by doping and thickness. For investigating effect of doping on microhardness, PMMA samples with titanium oxide content of (0, 0 .0001, 0.0005, 0.001, 0.005, 0.01 & 0.05) wt% were subjected to various load values from 10 gms to 200 gms. The variation of microhardness number H_v with wt % content of titanium oxide in PMMA can be seen in fig2 & fig3 .From the various graphs it can be interpreted that hardness increases with increasing wt % content of titanium oxide.

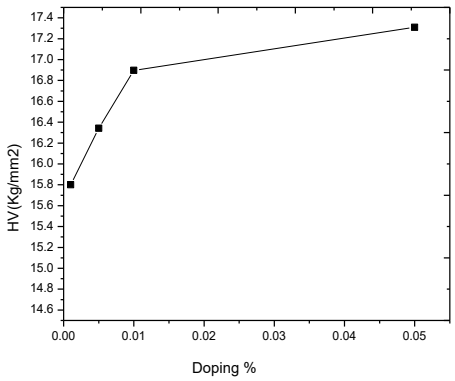

Fig 2 Variation of Hv with doping % for Pure and Tio2/PMMA composites at room temperature for 100 gm load.

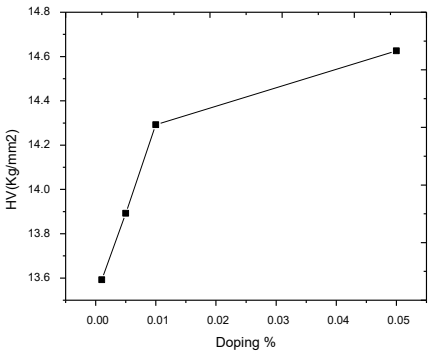

Fig 3 Variation of Hv with doping % for Pure and Tio2/PMMA composites at room temperature for 115 gm load.

Strain hardening index

The load dependent nature of microhardness of materials can be determined by strain hardening index of Meyer's law. Meyer's law is useful to analyze dependence of hardness on load L gives the following relation between the applied load *L* and indentation diagonal *d* [12].

This gives the relation between load L and length of diagonal d:

$$L = ad^n$$

Taking logarithm both sides of the equation, we have

log L = log a + n log d

Where, 'a' is constant i.e. load for unit dimension and 'n' is the logarithmic index number, which is the measure of strain hardening.

Strain hardening is an increase in the strength and hardness of the metal due to a mechanical deformation [13].The slope of log L versus log d gives the value of MeyersIndex. The Meyer index number can be calculated from the Meyer's law. The plots of log L versus log d for pure and annealed specimens (with different wt% doping of titanium oxide) of PMMA are shown in Figs.4 - 6. It can be observed from these graphs that for all specimens, the strain hardening index has two values, one for the low load region ranging from 10 to 60 gms with slope n_1 and the other for the high load region ranging from 60 to 200 gms with slope n_2. It is clear from the figures that the value of *n* is greater for high load region than for low load region. Meyer's law indicates that the value of $H_v$increases continuously with load.

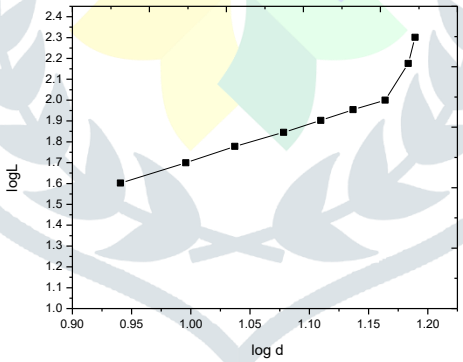

Fig 4 Variation of Log d with Log L for pure pmma at room temperature.

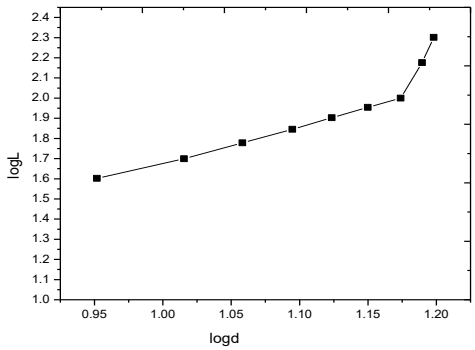

Fig 5 Variation of Log d with Log L for 0.0001% Tio2/Pure PMMA film at room temperature

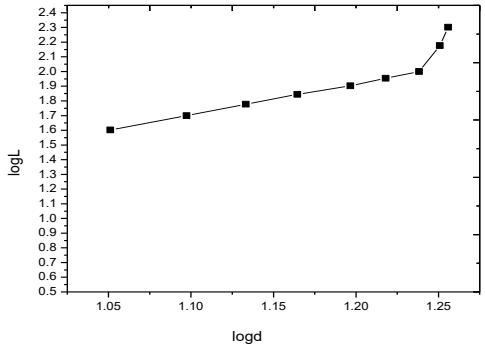

Fig 3 Variation of Log d with Log L for 0.05% Tio2/Pure PMMA film at room temperature.

From the slope of log L Vs. log d plot, the value of *n* was estimated, which gives the Meyer index number or work hardening index.Meyer's law indicates that the value of Hvincreases continuously with load when n is greater than 2 and the value of *n* approaches 2 in the saturation load region when Hvbecomes independent of load. Hence, the logarithmic index number, *n*, can be considered as a measure of strain hardening in different specimens. In fact the different values of n in the different load regions reflect the elastic and plastic characteristics of deformation[14].The value of *n* is expected to be 2 but most of the experimental data show that it is always less than 2[15].The strain hardening index (n) is increasing at higher load region. The variation in strain hardening index shows, the material becomes tough at higher load region due to formation of crystalline region inside the specimen.

Table 1. Calculated value of n for pure and doped PMMA at roomtemperatures in the two load regions

Temperature	Tio2 (wt %)	Slop in low load region	Slop in high load region
Samples at 25°C	0%	1.375	1.65
	0.0001%	1.38	2.25
	0.05%	1.69	2.34

6. Conclusion

The microhardness of a substance is an important parameter to define the strength of its material. This property is basically related to the crystal structure of the material or the way in which the atoms are packed. Hardness is the resistance offered any substance to localized plastic deformation. Hardness testing provides useful information about the mechanical properties of material.

The microhardness study on pure and doped thin films of PMMA helped in understanding the modification in properties due to inter and intra - molecular interactions. The effect of various parameters like applied load and effect of doping on microhardness have been studied and the variation of microhardenss with these parameters were analyzed.

It was observed that initially microhardness increases non linearly with load till 100gms. Beyond this value of load the microhardness of the specimen becomes independent of load and attains a level of saturation due to strain hardening. As the load is increased the specimen is subjected to a greater strain hardening which increases H_v. The H_v tends to saturate when the polymer specimen is fully strain hardened and no appreciable change in the value of H_v is observed.

The microhardness of doped PMMA increases with increasing doping of titanium oxide. By this we can say that samples show strengthen, when doping % of titanium oxide increases.

7.References

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