# Variation of Micro-Hardness of Titanium Oxide Doped Poly (Methyl Methacrylate) Composite samples with Different Annealing Temperature

P. Pendke[1,a)], K. Das[1,b)]and J.M.Keller[3, c)]

[1]Department of Post Graduate Studies and Research in Physics, St. Aloysius College, Jabalpur, 482001, M.P., India
[2] Department of Post Graduate Studies and Research in Physics, R.D.V.V Jabalpur, 482001, M.P., India
[a)]Corresponding author: email:poonampendke52@gmail.com
[b)]kallolkumardas@gmail.com
[c)]jagmohankeller@gmail.com

Abstract.The variation of micro-hardness of titanium oxide doped poly (methyl methacrylate)compositesamples with different annealing temperature 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. For the present studies Vicker's micro-hardness test has been used. The sample is subjected to loads of 10-200 grams.

## INTRODUCTION

Hardness measures material resistance to indentation. The term 'hardness' can have various meanings in different contexts, for example implying resistance to elastic deformation in the case of elastomeric materials or resistance to groove formation in scratching[1].Hardness is known as "Resistance of solid material to plastic deformation", usually by indentation. However, the term may also refer to stiffness or temper or to resistance to scratching, abrasion, or cutting. It is the property of a solid, which gives it the ability to resist being permanently, deformed (bent, broken, or have its shape changed), when a load is applied. The greater the hardness of the solid, the greater it has resistance to deformation. Hardness measurement does not depend on a single physical property, but it involves both the elastic and plastic deformation characteristics of materials. PMMA is frequently preferred because of its moderate properties, easy handling and processing, and low cost. The micro hardness technique was used for many years for characterization of such "classical materials" as metals, alloys and inorganic glasses. Its application to polymeric materials developed during the last several decades [2]**.**For the present studies Vicker's micro hardness test has been used. 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

$$H_v = 1.854\ \frac{P}{d^2}$$

where $P$is the force in Kg and $d$ is the mean diagonal length of the impression in millimeters. The value of $H_V$ is expressed in Kg/mm$^2$. The force is usually applied at a controlled rate, held for 30 s, and then removed [3-6].

## MATERIALS

### Polymethyl methacrylate (PMMA)

PMMA selected for the present work is one of the most important polymeric materials with good physical properties.PMMA, an ethylene derivative, is a synthetic polymer made by the chain growth method of polymerization [7]. PMMA is a transparent [8], high-strength commercially available amorphous thermoplastic polymer. PMMA exhibits prominent mechanical, dimensional, and thermal stabilities, as well as a high optical transparency with a relatively low glass transition temperature [9].

## Titanium oxide

Titanium dioxide, also known as titanium oxide or titania, is the naturally occurring oxide of titanium, chemical formula $TiO_2$. 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.

## Preparation of pure PMMA and their polymer composite film samples

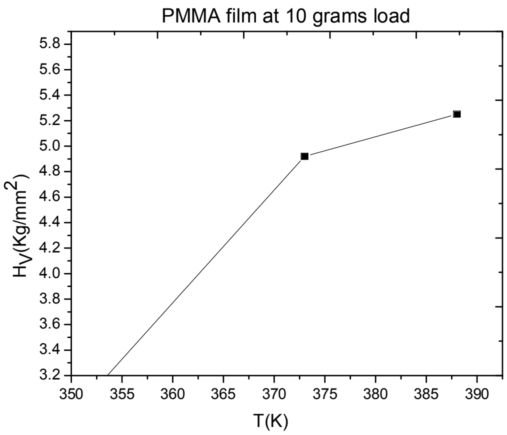
Polymer composites were prepared by doping different concentrations i.e. 0.0001%, 0.0005%, 0.001%, 0.005%, 0.01% & 0.05% wt% of titanium oxide in PMMA with the help of solution casting method.

## 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 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.. 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. A number of measurements were carried out and average of the hardness values was taken.

## RESULTS AND DISCUSSION

Annealing effects on the microstructure and morphology of polymer materials have been a subject of major technological and scientific interest for a number of years. Annealing has been performed to polymers for improving their crystalline order. There are many studies about annealing influences on the crystallization behavior of neat polymers [10].Annealing processes is time and temperature dependent [11].The effect of annealing temperature on pure PMMA and PMMA doped with titanium oxide has been investigated by annealing samples at temperatures 40°C, 60°C, 80°C,100°C and 115°C for 2 hours. $H_v$ is plotted against annealing temperature for each load for pure as well as doped PMMA samples. The effect of annealing temperature on hardness of pure and composite samples has been shown by figures 1- 7. Fig8 shows the variation of microhardness with temperature of pure PMMA for an applied load of 10 gm with error bars.



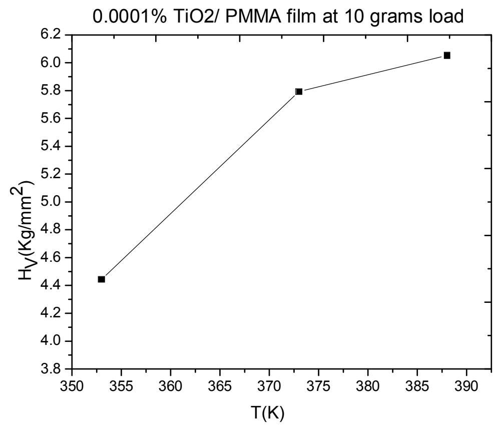
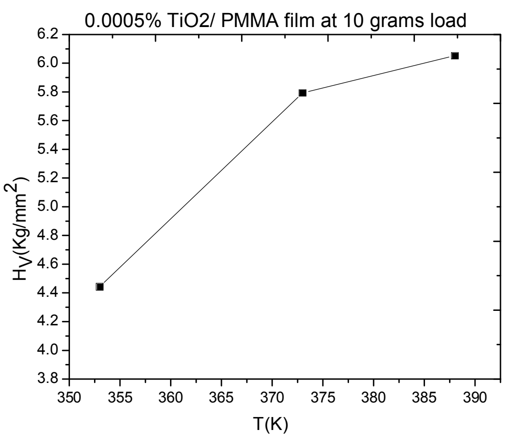




**FIGURE 1.**Variation of $H_v$ with annealing temperature **FIGURE 2** Variation of $H_v$with annealing temperature for
for pure PMMA film at 10 gm load0.0001% $TiO_2$/ PMMA composite at 10 gm load



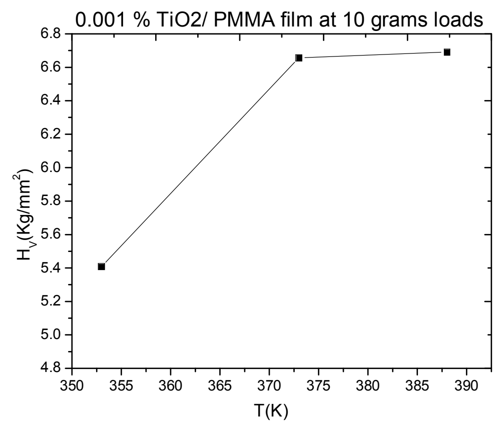
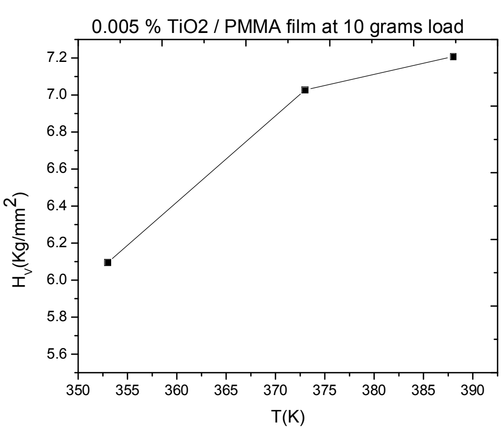




**FIGURE 3** Variation of $H_v$with annealing temperature for **FIGURE 4** Variation of $H_v$with annealing temperature for
0.0005% $TiO_2$/ PMMA composite at 10 gm load0.001% $TiO_2$/ PMMA composite at 10 gm l



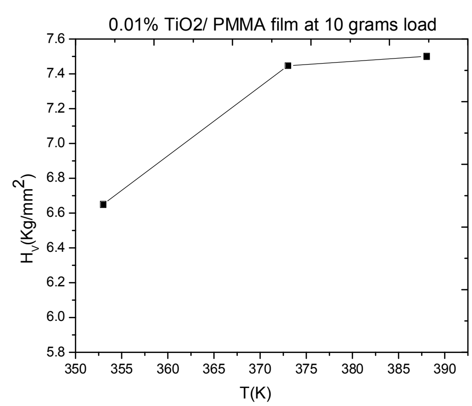




**FIGURE 5** Variation of $H_v$with annealing temperature for**FIGURE 6** Variation of $H_v$with annealing temperature for0.005%
$TiO_2$/ PMMA composite at 10 gm load 0.01% $TiO_2$/ PMMA composite at 10 gm load

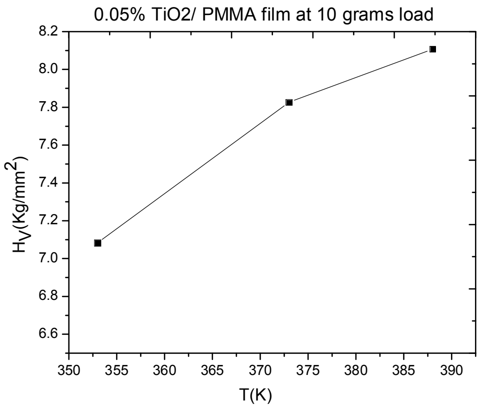



**FIGURE 7** Variation of $H_v$with annealing temperature for 0.05% $TiO_2$/ PMMA composite at 10 gm load

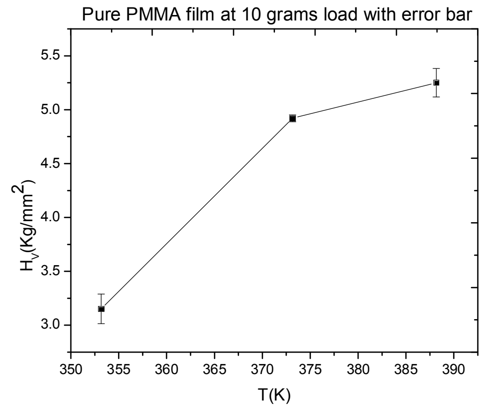



**FIGURE 8** Variation of $H_v$with annealing temperature for pure PMMA composite at 10 gm load with error bars

These graph shows that when temperature increases the hardness of samples increases because annealing of polymers improve their mechanical properties. Ayman A. Aly[12]reported in his work Polymers heat treatment is considered one of the most effective methods of modification to widen their application. The heat treatment of polymers can improve their mechanical and tribological properties. This effect is a result of crystal phase increase in the polymer structure, where the elastic part of polymer viscoelasticity increases causing significant increase in compressive strength and heat conductivity.

## 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 by any substance to localized plastic deformation. This paper presents the results of microhardness studies on pure and doped thin films of PMMA for various annealing temperatures 40, 60, 80, 100 and 115$^o$C.The effect ofannealing temperatures on microhardness have been studied and the variation of microhardness with temperature were analyzed. It was observed that as the annealing temperature is increased the hardness also increases. The increase in hardness with annealing temperature can be explained on the basis of formation of local orders in glassy polymers. With increasing temperature the thermal expansion in the crystalline regions will lead to an enlargement of the chain cross-section in the crystalline phase

## REFERENCES

1. M. I. Hutchingsa, *"Journal of Materials Research",*24581-589 (2009).
2. F. B. Calleja, Z. Roslaniec, *"Block Copolymers"*, CRC Press, 17(2000).
3. F J Balta and SFakirov ,*"Microhardness of Polymers"*,Cambridge University Press, 161(2007).
4. B.Agrawal and C.M.Agrawal, "*Basic Mechanical Engineering*", Wiley India, 32-33(2008).
5. H AYuehueiand R ADraughn, "Mechanical testing of Bone and the Bone implant Interface", CRC Press, 88-90(2000).
6. B. Bramfitt and A. OBenscoter, "*Metallographer's Guide: Practice &Procedures for Iron and Steels"*, ASM International,166-167(2002).
7. Chandramohan, B.Rajam, V.V.BasavaRao, "*International Journal of Scientific &Engineering Research",* 7(2016), 1766-1782.
8. B. K. Kizilduman et al, "*Advances In Materials Science"*, 171-23 (2017).
9. S.B. Azizet al, "*Polymers"*,9 1-15( 2017),.
10. M. Talukdar, "*International Journal of Recent Research and Applied Studies"*, 16,405-410 (2017).
11. R. C. Mikkelson (private communication).
12. A. A. Aly, "International Journal of Materials Chemistry and Physics", 1, 132-140(2017).