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Structural, Morphological and Thermal Study on PI and PI+ITO+GO Polymeric Composite Thin Films

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Abstract: ***Metal oxide incorporated polymeric composite thin films are of great interest to many applications because oxides within polymer matrix display a very wide range of properties like for the applications such as flexible thin films for solar cell, conducting transistors etc. The composite materials wherein inorganic particles are structured within a polymer matrix are likely to exhibit superior properties because of the size of the incorporated material. The study such as FT-IR, TGA, SEM and AFM has been discussed.***
Keywords: Polyimide, Indium Tin Oxide, Graphene Oxide, FT-IR, TGA, SEM, AFM.

I. INTRODUCTION

In mid of the nineteenth century polymeric material made up of human being are start replacing the conventional material. Due to different role of molecular shape and size, polymers or polymer based material are unique and belong to different class of material. The foregoing importance regarding the usefulness of metal oxide incorporated polymeric composite thin films opens the new dimension of study where the structure-morphological, thermal, micromechanical, electrical etc. can be correlated to develop new materials of desirable properties for advance technology. The controlling of disperse metal oxide particle in term of shape and size within the polymer matrix using different processessing technique is currently a frontier field of research with far reaching implications for new technologies. Polymer composites materials in which inorganic particles are dispersed in an organic polymer matrix at micro/ nano meter regime in order to significantly improve the performance properties of the polymer [1]-[3]

The conceptual key for selecting metal oxide and polymer is that, they have the ability of forming interaction between them on exothermic heat of mixing. Polyimide (PI) will play the role of host polymer matrix during the tenure of research work. PI had the fundamental aspects of macromolecular science, which will provide the base for developing innovative techniques for the synthesis of high temperature flexible thin film.

The Indium Tin Oxide (ITO) and Graphene Oxide (GO) possess excellent compatibility and reinforcing agent behaviour with other organic material like polymer. Moreover, apart from that both exhibits very good optical and electrical characteristic that makes them the great potential candidate to be incorporated within the PI matrix [4]-[7].

The PI and PI+ITO+GO polymeric composite thin films of different composition were prepared and explore to various characterization like FT-IR, TGA, SEM and AFM. The chemical functional groups within the composite thin films are explained using FT-IR technique. TGA characterization helps to understand the decomposition temperature of the PI and developed composite thin films. SEM and AFM micrograph helps to visualize the phase morphology and to detect the nano/ micro phase-domain structure within the composite thin films.

II. CHARACTERIZATION

A. Synthesis of ITO + GO Incorporated PI Composite Thin films

The ITO + GO + PAA composite solutions were weighed onto dry and clean glass plates and spread evenly using a spin-coating unit in a dust and moisture free chamber. Then it was conditioned at 70°C for 24 hours and thereafter subjected to curing for ½ hour each at 100, 150, 200, 250, 300 and 360°C for the first step, which partially imidizes the reactant. Subsequently, it was ultimately cured at 360°C for 2 hrs and then allowed to cool slowly to room temperature to get ITO + GO incorporated PI composite thin films. The pure PAA was also cured under identical conditions to form pure PI film. Most important number of mixed solution and film synthesis was tried, but the best was taken for further study. The different weight percentages of composite solutions were shown in table 1. The films were of 10 cm^2 in area and 15 ± 0.0002 µm in thickness, and the same were used for all the characterizations.

S. No	Solution Concentration	Value	Developed Films	Designation
1.	PAA	200 gm	Pure PI	PI
2.	PAA+ITO+GO	200 gm + ITO 1.0 wt% + GO 0.05 wt%	PI+ITO+GO	PITOGO-1
3.	PAA+ITO+GO	200 gm + ITO 1.0 wt% + GO 0.10 wt%	PI+ITO+GO	PITOGO-2
4.	PAA+ITO+GO	200 gm + ITO 1.0 wt% + GO 0.15 wt%	PI+ITO+GO	PITOGO-3
5.	PAA+ITO+GO	200 gm + ITO 1.0 wt% + GO 0.20 wt%	PI+ITO+GO	PITOGO-4
6.	PAA+ITO+GO	200 gm + ITO 1.0 wt% + GO 0.25 wt%	PI+ITO+GO	PITOGO-5

Table 3.1: Shows the various mixed solution concentrations and designations.

B. Fourier Transform Infrared Spectrometry (FT – IR) Study

The FT- IR spectrometry widely used applicable method for obtaining the exploring the chemical functional group structure and miscibility of the oxides with polymer and gives quantitative approach, which can interpret the resulting data. The Fourier Transform Infrared Spectrometry (FT-IR) of Pure PI and PITOGO composite thin films were obtained with Perkin Elmer FTIR-LX 18-5255 instrument. The FT-IR is based on the correlation between IR frequency and chemical functional group. The energy difference ΔE between the states is related to the frequency of the radiation emitted or absorbed according to the quantum relation $\Delta E=h\nu$.

C. Dynamic Thermogravimetric Analysis (TGA) Study

The Perkin-Elmer TGA-7 instrument was used to perform Dynamic thermogravimetric analysis (TGA). The heating rate adopted was 10^{o}C/min in an inert atmosphere.

D. Scanning Electron Microscopy (SEM) Study

Many of the special properties of composite thin films can be explained in terms of oxide dispersion within polymeric matrix which helps to understand the surface morphological aspects. Thus SEM micrographs help analyzes the formation and texture and feature of pure PI and PITOGO composite thin films in micro/ nano domain regime and helps to yield useful data on the surface morphology and phase distribution. To obtain the micrographs a electron microscope model (SEOL-JJM-5600LV) instrument was used which was fully computerized.

E. Atomic Force Microscopy (AFM) Study

To confirm the SEM micrograph imaging AFM characterization was used as it has the ability to create three-dimensional micrographs with resolution down to the micro and nano meter regime. It also helps to evaluate the phase domain morphology and analyze the mechanical interactions between ITO and GO within PI matrix along with the surface regions of the samples with varied stiffness of the constituent phase. Thus, phase images represent a compositional mapping. AFM topography of pure PI and PITOGO composite thin films were imaged using (DIAFM-4 instrument) in tapping mode.

III.RESULT AND DISCUSSION

Figure 1 shows the FT-IR absorption spectrum shows the possible chemical bonding structure of pure PI and PITOGO composite thin films. During the prime examination the physical appearance of pure PI and PITOGO composite films seems to have smooth texture. The characteristic absorption spectra of the imide unit at 1776, 1777, 1724, 1374 and 722 cm^{-1} wave numbers were observed for pure PI film. From figure 1 at 3470 cm^{-1} broad peak appeared in the high frequency area indicates the stretching mode of O-H bond, shows the presence of hydroxyl groups in GO and the band observed at 1735 cm^{-1} was assigned to the carboxyl group. The peak at 1345 cm^{-1} represents the C-OH group and the peak at 1245 cm^{-1} denotes C-O-C stretching along with the vibrational mode of the C-O group. The interaction of C–O bonds was absorbed at stretching frequency at 178 cm^{-1} and bands corresponding to stretching vibration of In–O–O–Sn appear at 1497 and 1585 cm-1. Thus confirms show the presence of indium and tin compounds along with GO within PI matrix confirming the formation of the composite.

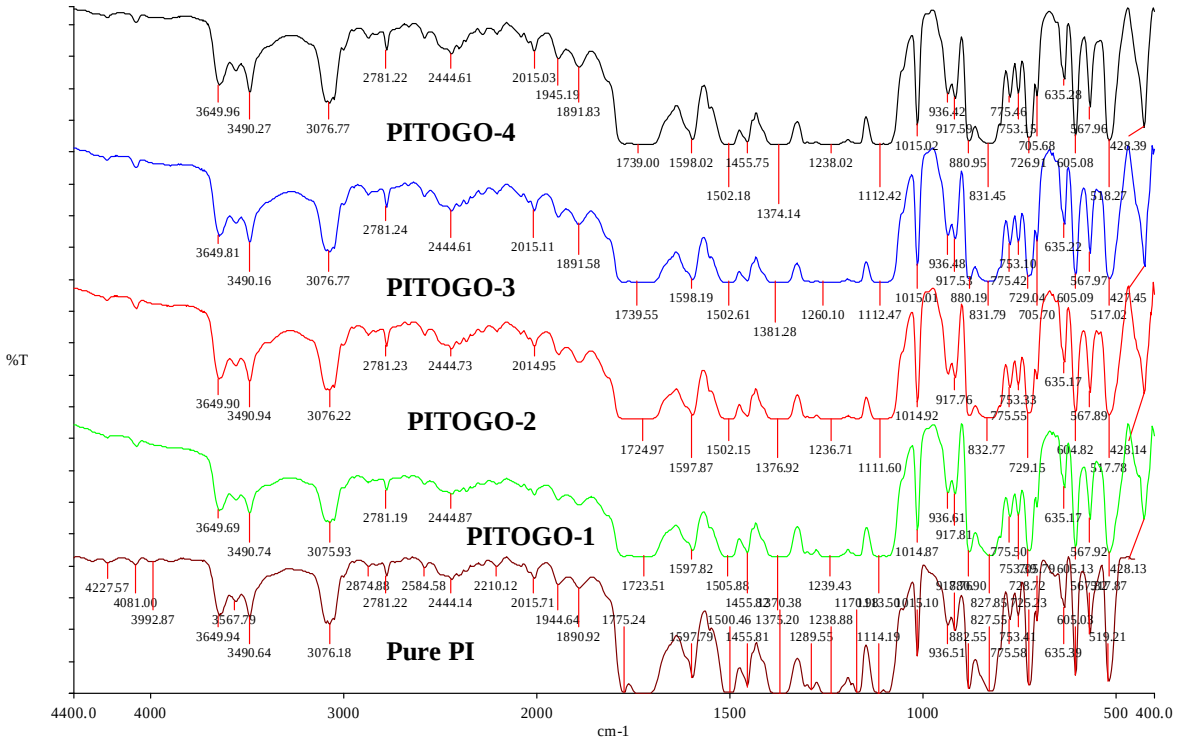

Figure 1: Shows the FT-IR absorption spectrum of PI and PITOGO composite thin films.

The decomposition temperature for PI and PITOGO composite thin films are shown in table 2 and for PITOGO composite thin films it was found to be higher. The best was found for PITOGO-2 composite thin films. At 630°C the initial weight loss for the composite thin films were observed which shows the thermal stability. The maximum decomposition temperature for PITOGO composite thin films is due continuous dispersion of ITO+GO within PI matrix along with cross-link consequence. Moreover, the requirement of high temperature for the degradation of PITOGO composite thin films arises due to the increase in potential energy barrier formed by the electrostatic attraction between the disperse particle and matrix. It can also be said that the formation of charge transfer complexes due to interact with the excited electronic states of disperse particle with PI molecules and hence these complexes may enhance the potential energy barrier for the decomposition. Thus it needs higher energy than normal for decomposition and the final residue is the ITO+GO content.

Inert atmosphere parameters (°C)						
Sample Designation	$D_{0.1}$ (C)	$D_{1/2}$ (C)	UDT (°C)	MRDT (°C)		Residue content %
				I^{st} Step	II^{nd} Step	
PI	600.0	632.2	672.0	625.0	-------	53.5
PITOGO-1	622.0	647.2	698.0	640.7	--------	26.1
PITOGO-2	629.5	656.0	699.7	648.0	--------	18.4
PITOGO-3	616.0	646.1	692.0	649.3	--------	28.4
PITOGO-4	610.0	641.0	690.0	651.4	--------	32.7
PITOGO-5	608.2	638.0	688.8	653.9	--------	36.9

Table 2: Inert atmosphere TGA analysis of PI and PITOGO composite thin films. Where UDT is ultimate decomposition temperature and MRDT is maximum rate decomposition temperature.

The SEM micrographs of pure PI (Fig. 2) shows the formation of a single phase with a smooth surface morphological feature. While observing the SEM micrographs (Fig. 3-7) of PITOGO composite thin films chain interaction was observed along with globular structure morphology. Moreover, a complete chain interaction was also observed. These globular structures are found to be in a spherical domain shape at micro/ nano level regime with chain type regularity within them. Varying the weight percentage of ITO and GO within the PI matrix plays very important role in the formation of smooth composite thin films. Thus the best result was observed for PITOGO-2. Moreover, as the varying weight percentage increasing about PITOGO-3 overall results starts decreasing might be due to coagulation with been ITO and GO However, this feature can be more convincingly understood by the AFM studies discussed below. Hence single phase morphology with a smooth texture is obtained and also indicates better compatibility between ITO and GO within PI matrix, which increases the performance in terms of mechanical and electrical properties of PITOGO composite thin films.

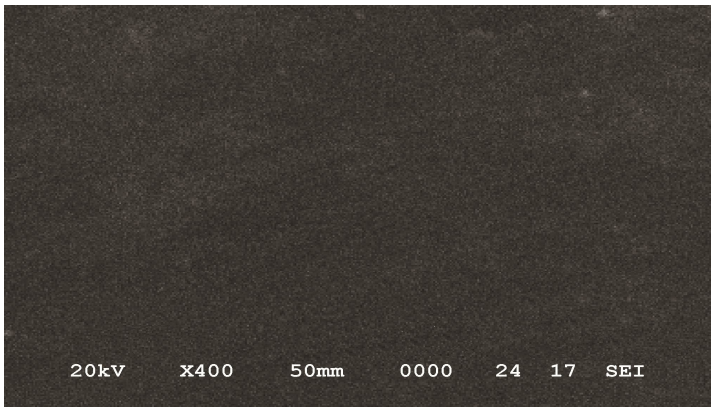

Figure 2: SEM Micrograph of pure PI.

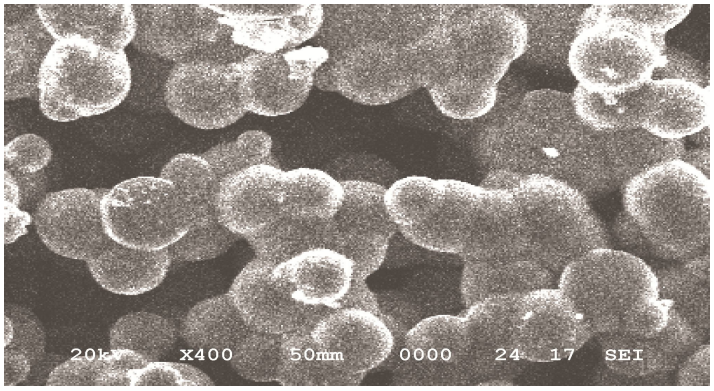

Figure 3: SEM Micrograph of PITOGO-1.

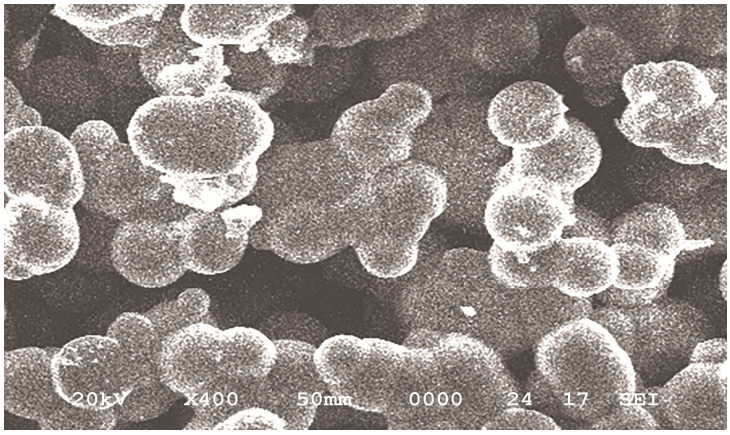

Figure 4: SEM Micrograph of PITOGO-2.

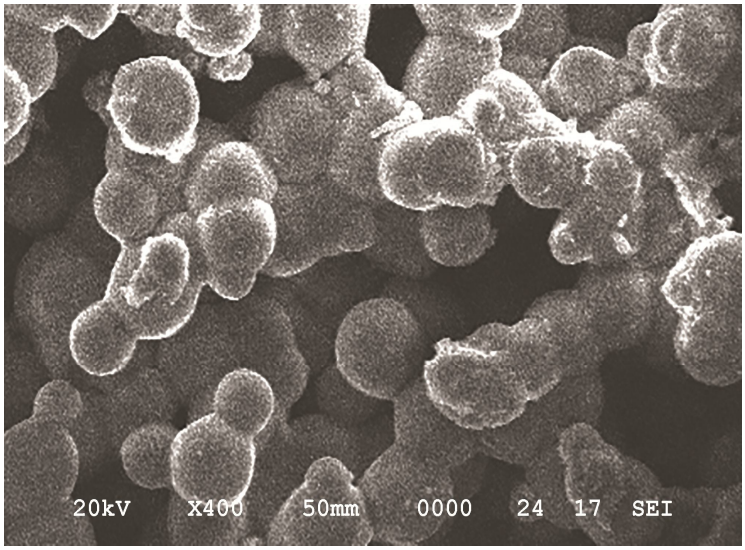

Figure 5: SEM Micrograph of PITOGO-3.

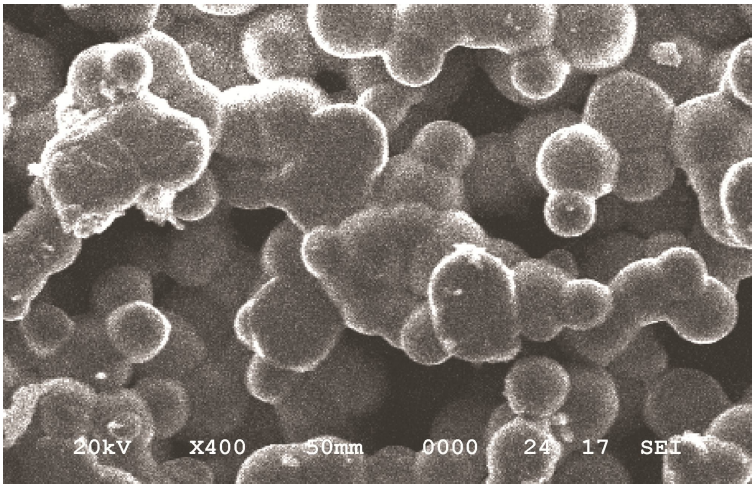

Figure 6: SEM Micrograph of PITOGO-4.

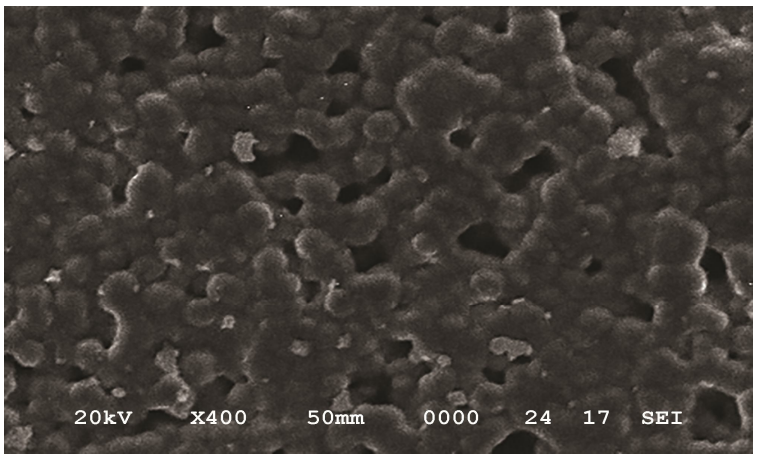

Figure 7: SEM Micrograph of PITOGO-5.

Figures 8-13 shows the 3D AFM topographic image pure PI and PITOGO composite thin films. The figure 9-13 i.e. AFM topographic image of PITOGO composite thin films clearly shows the formation of spherical micro/ nano domain with uniform regularity. Figure 8 shows the AFM topography of pure PI films clearly defines the amorphous characteristic of the thin film where spherical micro/ nano phase are dispersed. Thus, this result suggests that spherical micro/ nano domain structure within PI matrix could be controlled by the specific interaction when ITO and GO are mixed in different weight proportion. Moreover, the result suggests that the surface enrichment in this study is more strongly affected by the interaction parameter and it should be noted that the degree of surface enrichment is controlled by the magnitude of interaction. Form PITOGO AFM topographic image the best result was observed for PITOGO-2, which is also confirmed by SEM image. In case of AFM image same trend was observed which was observed in SEM image i.e. after PITOGO-3 weight percentage configuration ITO and GO start coagulating and no appreciable result was observed.

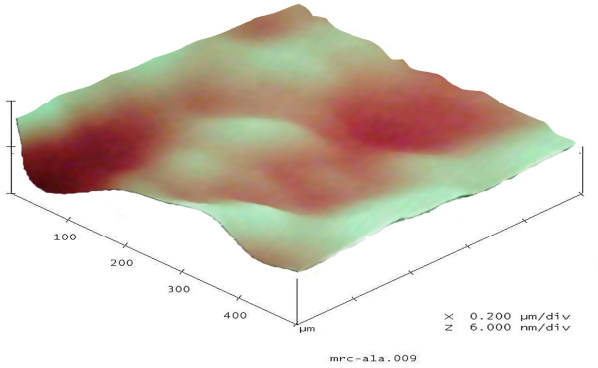

Figure 8: AFM 3D topography image of pure PI.

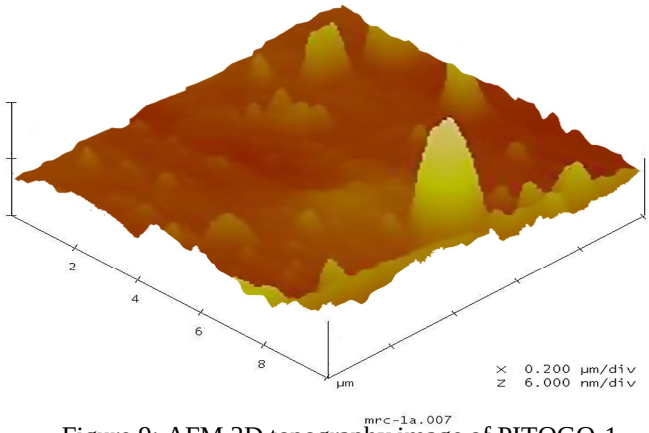

Figure 9: AFM 3D topography image of PITOGO-1.

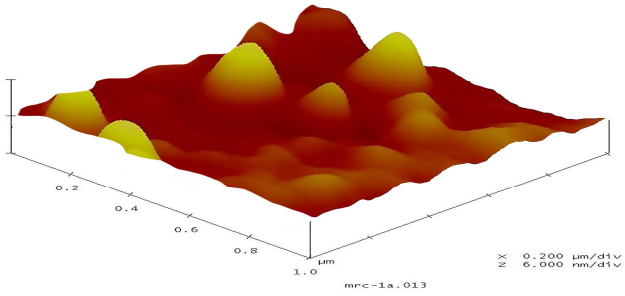

Figure 10: AFM 3D topography image of PITOGO-2.

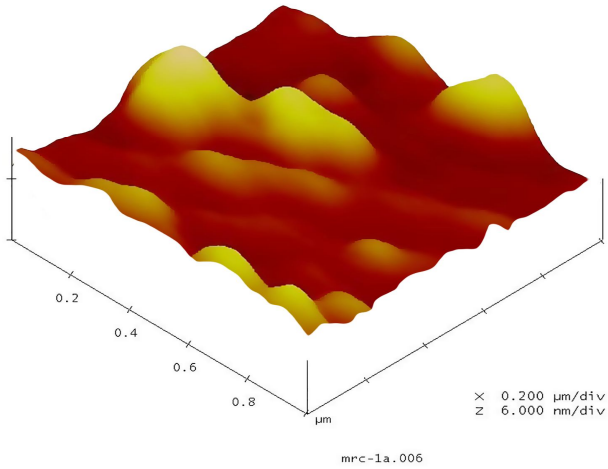

Figure 11: AFM 3D topography image of PITOGO-3.

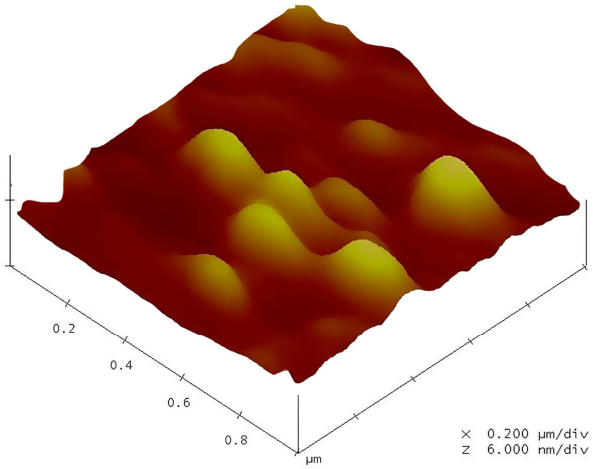

Figure 12: AFM 3D topography image of PITOGO-4.

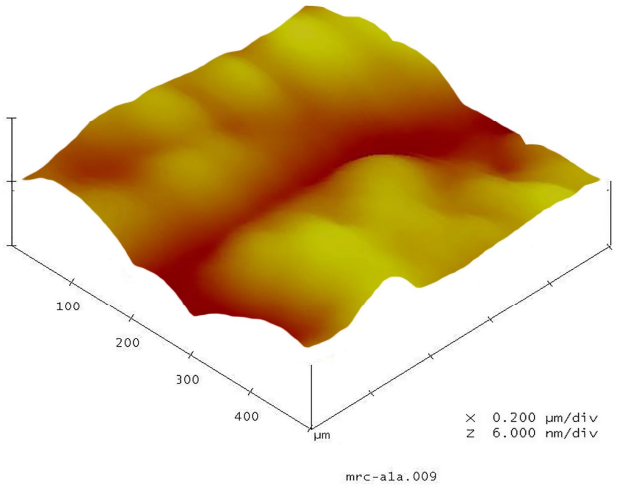

Figure 13: AFM 3D topography image of PITOGO-5.

IV. CONCLUSIONS

The PITOGO composite thin films were prepared using In-situ Generated Inter-chain Crosslinking Moieties (IGICM) approach along with pure PI thin film. The most challenging aspect was the incorporation of ITO and GO indifferent weight percentage with in PI matrix. Thereafter various characterizations was carried out like FT-IR, TGA, SEM and AFM to study and understand the issues related to the processing and structure-property relationship. These issues include decomposition temperature, chemical bonding, compatibility, morphology, chain interaction structure at micro/ nano domain region. The pure PI film was taken as a reference to compare PITOGO composite thin films and the overall best result was found for PITOGO-2 due to uniform dispersion of ITO+GO within the PI matrix at micro/ nano level regime.

V. ACKNOWLEDGMENT

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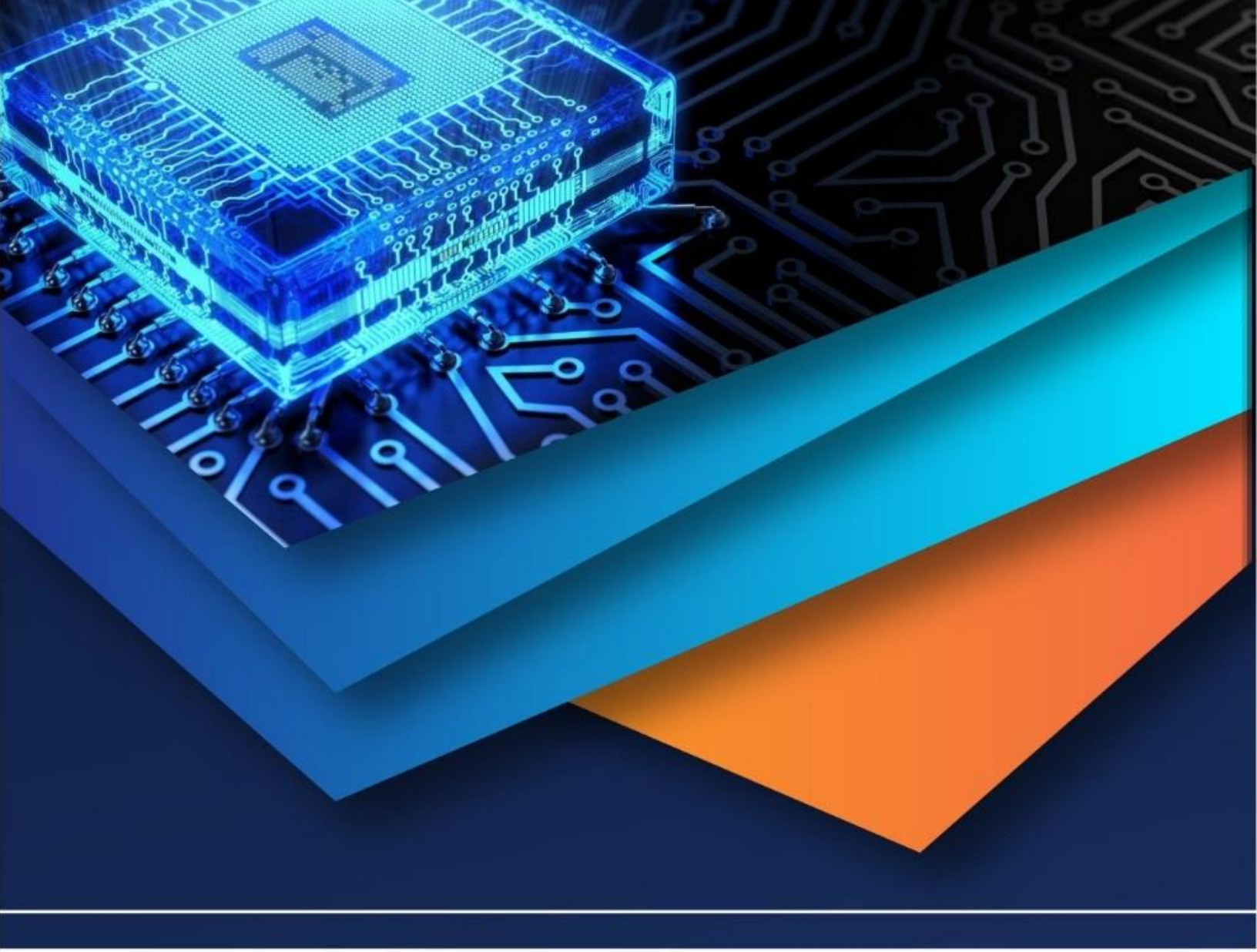

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