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Development and Characterization of Polyester–Titanium Functionally Graded Materials

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Abstract: The present study focuses on the fabrication and experimental investigation of polyester-based titanium functionally graded materials (FGMs). Titanium powder was produced by a conventional filing process using a 50 mm diameter titanium rod mounted on a lathe machine. The filing operation was performed at a rotational speed of 650 rpm using a coarse file. The resulting powder was subsequently demagnetized to eliminate iron particles and other unwanted ferromagnetic contaminants. The powder was then sieved to obtain titanium particles with a desired particle size of approximately 300 µm. Polyester-based FGMs containing 2.5 g, 5 g, and 10 g of titanium by weight were fabricated using the sedimentation technique. The fabricated FGMs were initially examined using liquid penetrant non-destructive testing to assess their surface integrity. Further characterization was carried out in terms of density, hardness, and microstructural features using optical microscopy. The experimental results obtained from these investigations were compared and analyzed to evaluate the influence of titanium content on the properties of the fabricated polyester–titanium FGMs.

Keywords: microstructural, evaluate, titanium, fabricated

1. Introduction

The continuous advancement of engineering and manufacturing technologies has resulted in the development of numerous innovative materials with enhanced functional and structural characteristics. Composite materials possessing high strength-to-weight and stiffness-to-weight ratios have attracted considerable attention because of their relatively low weight and superior performance. Such materials have found extensive applications in aerospace, automotive, energy, defense, biomedical, and other advanced engineering sectors.

Functionally graded materials (FGMs) represent a distinctive class of advanced composite materials in which the composition and properties vary gradually and continuously from one region to another. Unlike conventional composites, which generally possess distinct and relatively uniform phases, FGMs are designed by progressively varying the proportion of two or more constituent materials having different mechanical, thermal, or physical properties. This controlled compositional variation produces a gradual transition in properties across the material, thereby reducing abrupt interfaces and enabling the material to perform multiple functions within a single component.

The principal objective of developing FGMs is to achieve a desirable combination of properties, such as enhanced mechanical strength, thermal resistance, wear resistance, and structural integrity. The gradual distribution of constituent phases makes FGMs particularly suitable for applications involving severe thermal gradients. For example, in aerospace and spacecraft components, one surface may be subjected to extremely high temperatures while the opposite surface experiences considerably lower temperatures. An appropriately designed FGM can accommodate such variations more effectively than a conventional composite.

Because the material properties of FGMs vary spatially, analytical and numerical approaches developed for conventional homogeneous or discrete-phase composites may not always be directly applicable. The continuous variation in composition and properties must be considered when evaluating their mechanical, thermal, and structural behavior. Consequently, FGMs have received significant research attention because of their potential applications in aerospace, energy, defense, electronics, biomedical engineering, and other advanced technological fields.

The properties and performance of an FGM are strongly influenced by the processing technique employed during fabrication. In general, FGM manufacturing involves the formation of a graded structure and the consolidation of the constituent materials. A variety of processing methods have been developed to achieve controlled compositional gradients. In centrifugal casting, for example, the difference in specific gravity between the matrix and reinforcement particles can be utilized to obtain a graded distribution. The application of centrifugal force causes the reinforcement particles to redistribute during solidification, thereby producing a controlled material gradient.

Among the various processing techniques, sedimentation is a relatively simple method for producing particle-reinforced FGMs. This technique primarily relies on the difference in density between the reinforcement particles and the matrix material. Under the influence of gravitational force, particles settle at different rates, resulting in a gradual variation in reinforcement concentration along the specimen thickness. By appropriately controlling the processing conditions, a graded structure can therefore be obtained. Hassanin and Jiang [1] developed multilayer FGMs using highly stable alumina–zirconia suspensions. The stability of the suspension was controlled by adjusting the pH and the concentration of dispersant with respect to the powder content.

FGMs have demonstrated considerable potential in a wide range of engineering and biomedical applications. Al_2O_3/Y and ZrO_2-based FGMs have been investigated for biomedical applications such as knee and hip prostheses [2]. Functionally graded dielectric materials have also been explored for applications in the energy sector, high-current connectors, sensors, and capacitors [3–6]. Youngming et al. [7] investigated an Al_2O_3/Ti_3SiC_2 FGM system and reported that increasing the Al_2O_3/Ti_3SiC_2 content influenced the electrical conductivity of the fabricated FGM. An increase in hardness was also observed with changes in the composition of the constituent phases. Afzal et al. [8] developed a biocompatible FGM consisting of zirconia, hydroxyapatite, and alumina and evaluated its structural integrity.

Das et al. [9] reviewed recent developments in the design, processing, and engineering applications of functionally graded materials. Their study discussed various processing technologies employed for producing graded multilayered materials and highlighted potential future research directions. According to Jha et al. [10], the ability to tailor structural and morphological characteristics is one of the major advantages of FGMs over conventional composites. Pan [11] reported that functionally graded structures can exhibit lower residual stresses compared with corresponding non-graded materials. Kim et al. [12] investigated thermal barrier coating systems for gas turbine engine components and tungsten-coated dampers, emphasizing that thermal mismatch between the coating and substrate remains one of the important challenges in the development of thermal barrier systems.

Andrew et al. [13] demonstrated the successful fabrication of regular gradients in hydroxyapatite–316L stainless-steel biomaterial FGMs using thixotropic casting. Their work also highlighted the potential of impeller dry blending for producing approximately linear gradients in metal–ceramic FGMs. Hydrostatic shock forming was investigated as a potential densification technique for continuous bulk FGMs in systems containing metal and ceramic constituents with substantially different melting temperatures. Ti–TiB FGMs have also been fabricated using pressure-assisted densification for ballistic applications. Such graded materials have been reported to exhibit changes in properties such as elastic modulus, coefficient of thermal expansion, and ballistic performance [14]. Lucignano and Quadrini [15] developed functionally graded polyester materials and investigated their mechanical properties.

Spark plasma sintering (SPS) is another widely used solid-state technique for fabricating FGMs [16–18]. The SPS process has been employed for producing nanostructured materials, metal-matrix composites, fiber-reinforced metal-matrix composites, intermetallic compounds, FGMs, and other wear-resistant materials, including sandblasting nozzles [19]. The ability of SPS to process materials having different melting characteristics makes it suitable for the fabrication of graded structures. Compared with several conventional processing methods, SPS provides rapid heating, efficient powder consolidation, and improved control over material processing. El-Desouky et al. [20] investigated activated-tip-based sintering and reported a relationship between green density and the dimensions of the fabricated FGM.

Using phenolic resin, nanofibers, and powder metallurgy techniques, Bafekrpour et al. [21–23] developed polymeric functionally graded nanocomposites and investigated their viscoelastic and thermomechanical behavior. Their results indicated that composites containing higher graphite concentrations in the upper and lower layers and a lower concentration in the central region exhibited improvements in thermomechanical properties and creep resistance. Ashish et al. [24] presented an overview of FGMs, including their concept, requirements, manufacturing techniques, and mathematical models for predicting effective material properties. The study also discussed emerging applications of additive manufacturing techniques for the production of FGMs from both academic and industrial perspectives.

Watanabe et al. [25] investigated the feasibility of producing Ni-aluminide/steel clad pipes using centrifugal casting. Jedamark et al. [26] employed an electrochemical gradation technique to fabricate a W/Cu FGM system. The resulting material exhibited a controlled chemical gradient, while processing parameters such as electrolytic conditions, porosity, resistivity, density, and geometry influenced the resulting morphology and structure. Ankush Mehta et al. [27] proposed an FGM based on two different materials in the form of adhesive and silicone-based gels. A three-dimensional bioprinter was used to fabricate tensile specimens, and their mechanical and electrical characteristics were investigated. Two FGM configurations, namely A+B and B+A, were produced using the same weight proportion. Variations in thickness and non-uniformity of the fabricated specimens were observed, resulting in corresponding changes in their dielectric properties.

Wilson and Shin [28] employed laser deposition to fabricate Ni-based FGMs and reported that increasing the TiC content in Inconel 690 powder resulted in microstructural refinement. Chemical solution deposition (CSD) is another relatively simple technique used for producing thin films, in which a liquid precursor is dissolved in a suitable solvent. Put et al. [29] investigated the mechanical behavior of ZrO_2/Al_2O_3 and WC/Co FGMs fabricated using electrophoretic deposition. For the ZrO_2/Al_2O_3 system, the hardness varied from approximately 13.5 GPa at the core to 19.5 GPa at the edges. In the WC/Co FGM, the hardness variation was reported from approximately 21 GPa at the core to 9 GPa at the edges.

Katayama et al. [30] fabricated an Al_2O_3–W FGM using slip casting followed by sintering. Their results indicated that an increase in dispersant concentration improved the dispersibility of the raw materials. A gradual variation in tungsten grain size was also observed along the compositionally graded region. Gel casting is another commonly employed technique for fabricating complex-shaped ceramic components with either dense or porous structures. It provides advantages such as high productivity, relatively low processing cost, good green strength, and reduced forming time. Park et al. [31] fabricated an Al_2O_3/ZrO_2 FGM by dip-coating substrates prepared through gel casting and investigated its mechanical and microstructural characteristics. A gradual transition in hardness from the substrate toward the outer layer was observed.

Gas-phase processing techniques, such as ion plating, chemical vapor deposition, and plasma spraying, have also been employed to produce compositionally graded materials. In vapor deposition processes, the composition gradient of the deposited material can be controlled by adjusting the processing parameters and the relative reaction rates of the constituent phases. Liquid-phase processing techniques are also widely utilized for producing compositionally graded coatings and composite structures. The final properties of an FGM depend strongly on the processing technique and associated parameters. The major challenges in FGM fabrication include achieving controlled spatial gradation of the constituent phases and effectively consolidating the graded structure [32].

Although extensive research has been carried out on metal- and ceramic-based FGMs, comparatively fewer studies have focused on polymer-based functionally graded materials. In particular, research concerning the fabrication and characterization of polyester-based titanium FGMs using simple and economical processing methods remains limited. Therefore, the present study aims to fabricate polyester-based titanium functionally graded materials using the sedimentation technique and to investigate their density, microstructural characteristics, and hardness. The influence of titanium content on the resulting properties of the fabricated FGMs is also evaluated.

2. Experimental Procedure

2.1 Materials and Methods

In the present investigation, polyester resin was selected as the matrix material, while titanium powder particles were used as the reinforcing phase. The polyester matrix provides the continuous polymeric phase, whereas the titanium particles are progressively distributed within the matrix to develop the desired functional gradient. The relevant physical and mechanical properties of pure polyester resin and titanium are presented in Tables 1 and 2, respectively.

Table1. Properties of Polyester

Property	Value
Viscosity at 25^{0}C (ns/m^2)	250-350
Density (g/cm^3)	1.09
Modulus of elasticity E (Gpa)	3.3
Flexural strength (Mpa)	4.5
Tensile strength (Mpa)	40
Maximum elongation	1

Table2. Properties of Titanium

Property	Value
Density (g/cm^3)	4.5
Melting point (*c)	1670

Thermal conductivity (w/m-k)	15-22
Elastic modulus (Pa)	115
Reactivity with oxygen	High+
Corrosion resistance	High+

2.2 *Production of Reinforcements (particles)*

Titanium particles were produced using a conventional filing technique. The particle production process is illustrated in Figure 1. A titanium rod of 50 mm diameter was subjected to a filing operation on a lathe machine using a coarse file at a rotational speed of 650 rpm. The titanium powder obtained from the filing operation is shown in Figure 2.

Figure 1. Filing operation on lathe machine.

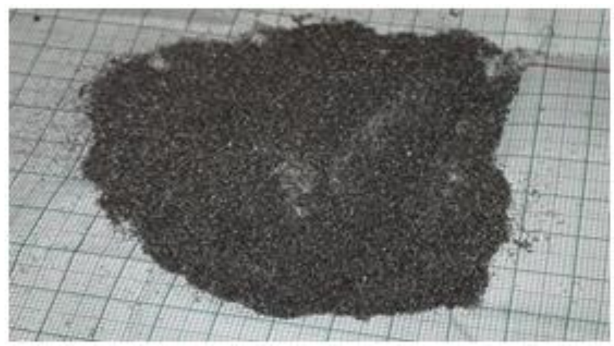

Figure 2 . Obtained particles after filing.

The produced powder was subsequently subjected to a demagnetization process to remove ferrous particles and other iron-based contaminants introduced during the filing operation. The powder was then sieved to obtain particles within the required size range. The sieving operation was carried out for 10 min, as shown in Figure 3. Titanium particles with a particle size of up to 300 µm were selected in accordance with IS 460:1982.

Figure 3. Sieving of Ti particles.

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2.2 Fabrication of Functionally Graded Materials

2.2.1 Sedimentation Method

A sedimentation technique was employed to fabricate polyester–titanium functionally graded materials. A PVC pipe having a diameter of 2.4 cm and a length of 4.2 cm was used as the mould for specimen fabrication. The sieved titanium particles were incorporated into the polyester matrix at three different quantities, namely 2.5 g, 5 g, and 10 g by weight, to produce FGMs with varying titanium concentrations.

For fabrication of the 2.5 g Ti FGM, a measured quantity of 2.5 g of titanium particles was mixed with polyester resin and poured into the vertically positioned PVC pipe. After filling, the mixture was allowed to remain undisturbed for approximately 1–2 h to facilitate gravitational sedimentation of the titanium particles. The sedimentation process is illustrated in Figure 4.

Figure 4. Fabrication of FGMs through Sedimentation process.

The same procedure was followed for fabricating polyester–5Ti and polyester–10Ti FGMs using 5 g and 10 g of titanium particles, respectively. For comparison purposes, a pure polyester specimen without titanium reinforcement was also fabricated under identical processing conditions. The fabricated specimens with different titanium contents—pure polyester, 2.5 g Ti, 5 g Ti, and 10 g Ti—are shown in Figure 5.

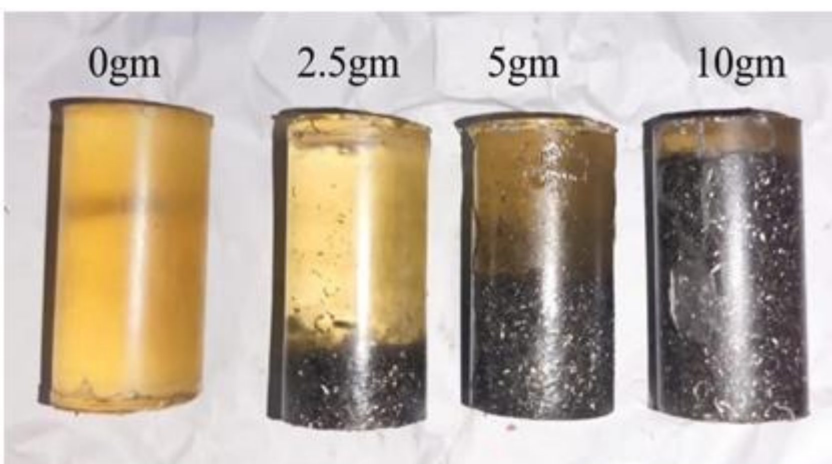

Figure 5. Obtained samples from sedimentation process.

2.3 Experimental Testing

The fabricated functionally graded specimens were subjected to different experimental investigations to evaluate their quality and characterize their physical, mechanical, and microstructural properties. The following tests were performed:

2.3.1 Liquid Penetrant Test

Liquid penetrant testing was employed as a non-destructive method for detecting surface-breaking defects in the fabricated specimens. The test was performed using a red-colored penetrant and a white developer. After application of the penetrant, sufficient time was allowed for it to enter any surface discontinuities present in the specimen. The excess penetrant was subsequently removed, followed by application of the developer.

The developer acts as an absorbent medium and draws the trapped penetrant out of surface defects, thereby producing visible indications. The appearance of red indications against the white developer provides information regarding the location and nature of surface flaws such as cracks or discontinuities.

2.3.2 Density Measurement

The density of each fabricated specimen was determined after fabrication by measuring its mass and volume. The density was calculated using:

$$P = \text{Mass}/\text{Volume}$$

2.3.3 Hardness Test

The hardness of the fabricated FGMs was evaluated using the Vickers hardness testing method. Measurements were obtained from both the polyester-rich and titanium-rich surfaces of each specimen using a constant load of 0.5 kg. Three measurements were taken at different locations on each surface, and the average value was considered as the representative hardness of the corresponding region.

2.3.4 Optical Microscopic Examination

Optical microscopy was employed to examine the microstructural characteristics and distribution of titanium particles within the polyester matrix. Prior to microscopic examination, the specimens were subjected to sequential polishing to obtain smooth and suitable surfaces for observation.

Both the top and bottom surfaces of the fabricated specimens were examined to identify the presence and distribution of titanium particles and the polyester-rich regions. The microscopic observations were used to verify the formation of the intended functional gradient.

3. Results and Discussion

3.1 Liquid Penetrant Non-Destructive Testing

Figure 6 presents the specimens examined using the liquid penetrant non-destructive testing method. The red-colored indications correspond to surface-breaking cracks or other discontinuities, which become visible after application of the white developer.

Figure 6. Defect identification through liquid penetrant test.

The test results indicate that the fabricated specimens were generally free from significant surface defects. Except for a minor indication observed near the tip region, no appreciable cracks or other significant discontinuities were detected. This observation indicates that the fabrication process produced specimens with satisfactory surface quality.

3.2 Density

The measured density values of pure polyester and the polyester–titanium FGMs containing 2.5 g, 5 g, and 10 g of titanium are summarized in Table 3. The corresponding variation in density is presented in Figure 7.

Table 3. Summary of densities for FGMs

S.NO	**Ti by weight (gm)**	**Density (g/cm^3)**
1	0	1.21
2	2.5	1.33
3	5	1.4
4	10	1.59

The densities obtained for the 2.5Ti, 5Ti, and 10Ti FGMs were 1.33 g/cm^3, 1.40 g/cm^3, and 1.59 g/cm^3, respectively. A progressive increase in density was observed with increasing titanium content.

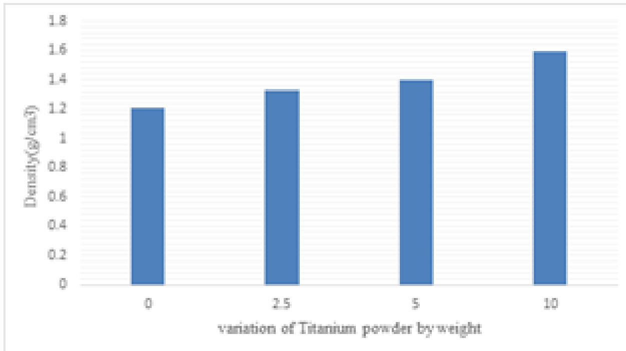

Figure 7. Comparison of densities for various FGMs.

This increase can primarily be attributed to the substantially higher density of titanium compared with that of the polyester matrix. Consequently, the incorporation of increasing quantities of titanium particles results in an overall increase in the density of the fabricated FGMs. The sedimentation process also facilitated the concentration of titanium particles in the corresponding region of the specimen, contributing to the formation of a denser titanium-rich zone.

For a given specimen volume, the resulting density is influenced by the amount and distribution of titanium particles incorporated into the polyester matrix. The progressive increase in measured density with increasing titanium content indicates that the sedimentation technique effectively facilitated the incorporation and grading of titanium particles within the polyester matrix.

3.3 Hardness

Figures 8 and 9 present the Vickers hardness measurements obtained for the 2.5Ti FGM on the polyester-rich and titanium-rich surfaces, respectively. The polyester-rich surface exhibited a relatively low hardness because of the comparatively softer nature of the polymer matrix. The larger indentation observed on this surface is consistent with its lower resistance to indentation.

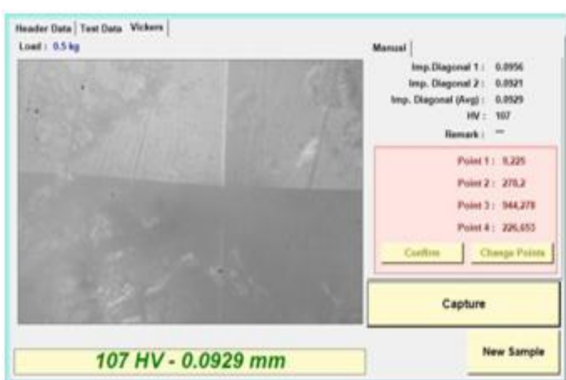

Figure 8. Hardness values of Pure Polyester Side(2.5 gm).

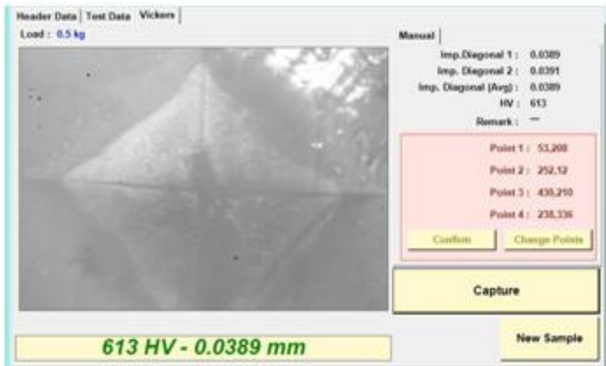

Figure 9. Hardness values of Titanium rich side (2.5gms).

In contrast, the titanium-rich surface exhibited a significantly higher hardness, with a measured value of approximately 613 HV. For a constant applied load, a smaller indentation generally indicates greater resistance to deformation and, consequently, higher hardness.

Figures 10 and 11 show the hardness measurements obtained for the 5Ti FGM. A hardness value of approximately 101 HV was measured on the polyester-rich surface, whereas the titanium-rich surface exhibited a hardness of approximately 771 HV. The higher hardness on the titanium-rich side compared with the corresponding region of the 2.5Ti FGM can be attributed to the increased titanium content.

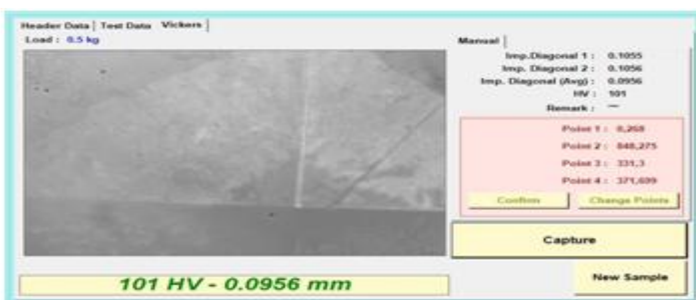

Figure 10. Hardness values of Titanium (5gm) pure Polyester Side.

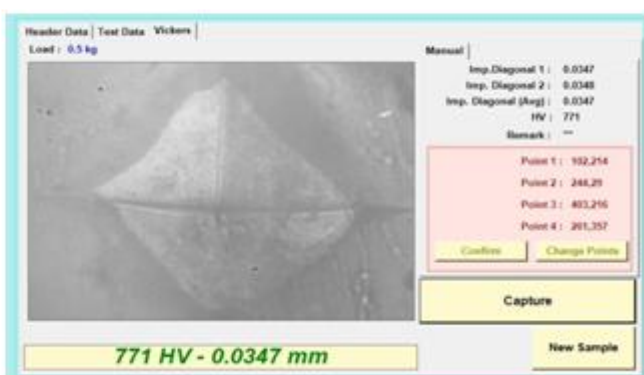

Figure 11. Hardness values of Titanium rich side (5gms).

The indentation dimensions provide further evidence of the difference in hardness between the two regions. The relatively smaller indentation associated with the titanium-rich surface indicates greater resistance to localized plastic deformation, while the larger indentation on the polyester-rich side indicates comparatively lower hardness.

For the 10Ti FGM, the titanium-rich surface exhibited a hardness value of approximately 897 HV, which was higher than the corresponding values obtained for the 2.5Ti and 5Ti FGMs as can be seen from Figures 12 and 13. This increase in hardness can be associated with the greater concentration of titanium particles in the titanium-rich region. In contrast, the polyester-rich surface exhibited a considerably lower hardness of approximately 103 HV.

The observed variation in hardness across the specimen thickness demonstrates the graded nature of the fabricated materials. The hardness of the FGM is influenced by the relative proportions of the polymer matrix and titanium reinforcement, as well as by factors such as elastic modulus, yield strength, particle distribution, and interfacial bonding. Overall, the experimental results indicate that increasing titanium content leads to a progressive enhancement in the hardness of the titanium-rich region.

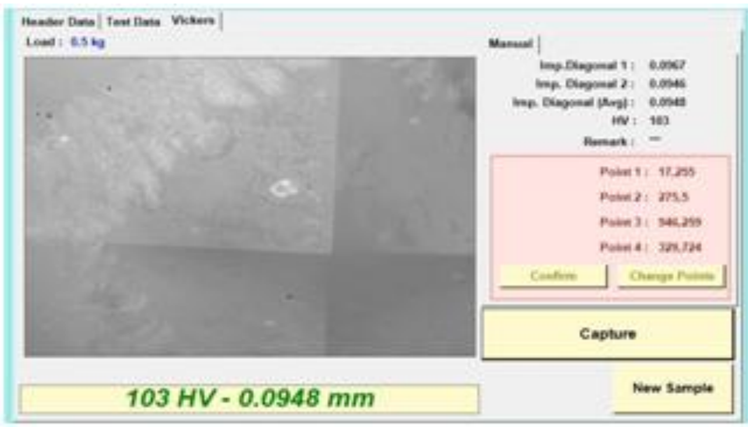

Figure 12. Hardness values of Pure Polyester Side.

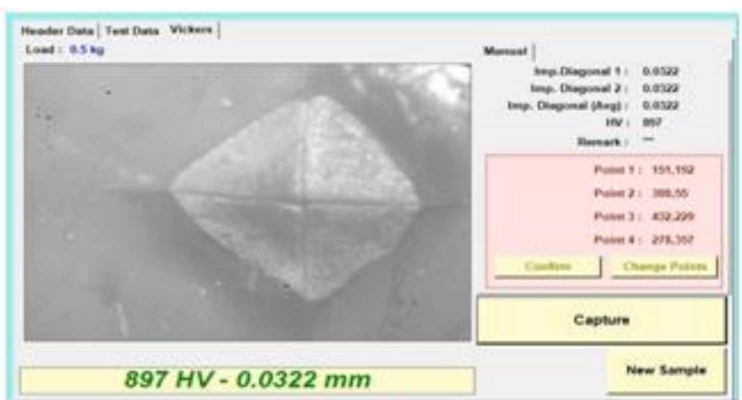

Figure 13. Hardness values of Titanium rich side(10gms).

3.4 Optical Microscopy

The microstructural characteristics of the fabricated specimens were examined using optical microscopy. Figure 14 shows the optical micrograph of the pure polyester specimen. Since the observed region consists exclusively of the polyester matrix, no metallic inclusions or titanium particles are visible.

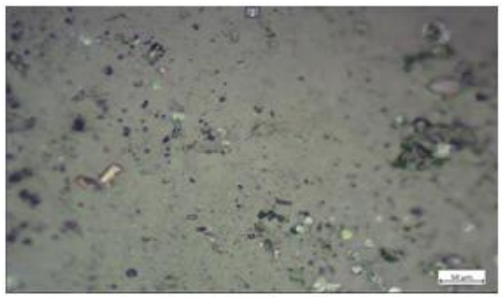

Figure14. Optical microscope image of pure polyester resin.

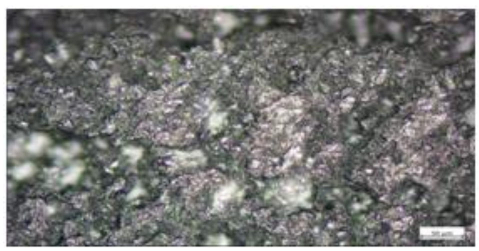

Figure15. Optical microscope image of10TiFGM

Figure 15 presents the optical micrograph of the titanium-rich region of the 10Ti FGM. A predominant presence of titanium particles can be clearly observed in this region. A relatively small amount of polyester matrix is also visible between and around the metallic particles, indicating that the polymer has penetrated and occupied the interparticle spaces.

Figure 16 shows the optical micrograph of the titanium-rich region of the 5Ti FGM. Compared with the 10Ti FGM, a lower concentration of titanium particles is evident. The increased presence of polyester in this region partially covers the titanium particles. The observed distribution suggests effective interaction between the titanium particles and the polyester matrix, which can contribute to improved structural integrity of the graded material.

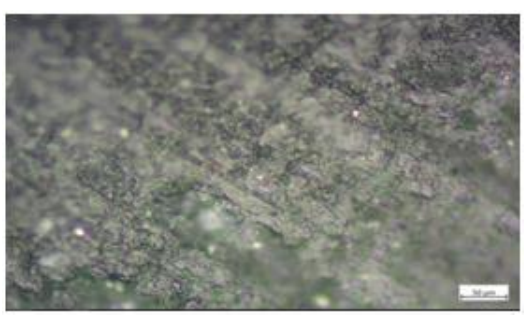

Figure 16. Optical microscope image of 5 Ti FGM on rich side.

Figure 17 presents the optical micrograph of the titanium-rich region of the 2.5Ti FGM. A relatively lower concentration of titanium particles is observed compared with the 5Ti and 10Ti FGMs. The polyester matrix is more prominently visible and appears to occupy the spaces surrounding the metallic particles.

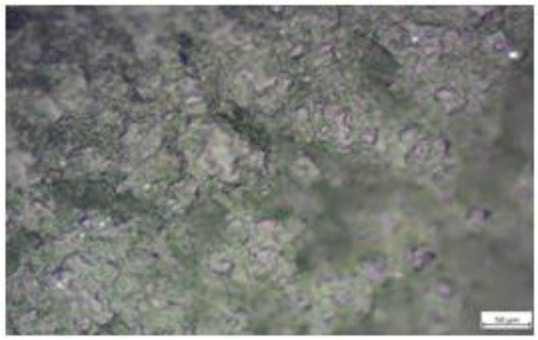

Figure 17. Optical microscope image of 2.5 Ti FGM.

The optical microscopy results demonstrate a systematic variation in the distribution of titanium particles with increasing titanium content. The gradual change in the relative proportions of titanium and polyester across the specimens provides microstructural evidence of the formation of functionally graded polyester–titanium materials.

4. Conclusions

Based on the experimental investigation of polyester–titanium functionally graded materials fabricated using the sedimentation technique, the following conclusions can be drawn:

1. Polyester-based titanium functionally graded materials with different titanium contents were successfully fabricated using the sedimentation technique.
2. Liquid penetrant non-destructive testing indicated satisfactory surface quality of the fabricated specimens, with no significant cracks or surface discontinuities except for a minor indication near the specimen tip.
3. The hardness of the fabricated FGMs increased progressively from the polyester-rich region toward the titanium-rich region. An increase in titanium content resulted in higher hardness values in the titanium-rich regions.
4. The density of the FGMs increased with increasing titanium content. The measured densities of the 2.5Ti, 5Ti, and 10Ti FGMs were 1.33, 1.40, and 1.59 g/cm³, respectively.
5. Optical microscopic examination confirmed the variation in titanium particle concentration within the polyester matrix. The increasing presence of titanium particles in the titanium-rich regions provided microstructural evidence supporting the formation of the intended functionally graded structure.
6. Overall, the experimental results demonstrate that the sedimentation technique is a simple and effective approach for fabricating polyester–titanium FGMs with controlled variation in density, hardness, and microstructural characteristics.

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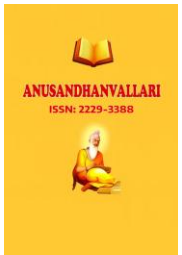

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