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Received on: 07/08/2017
Accepted on: 11/01/2018

Study the Effect of Nano-Al_2O_3 and Fiber Glass on Mechanical and Physical Properties of PMMA Composites for Prosthetic Denture

***Abstract**- In the present research, investigate the effect of adding some reinforcing materials which include nano-aluminum oxide (nano-Al_2O_3) particles that added with different volume fractions of (1%, 2%, 3% and 4%), and random fiber glass that added with fix volume fractions of (4%), into the PMMA matrix as new cold cure resin, and study some mechanical and physical properties of these hybrid laminated composite as prosthesis denture base material. Specimens' of this project were prepared by using (Hand Lay-Up) method to form a hybrid laminated composites materials. The mechanical tests were performed on these specimens include (tensile test, impact test, flexural test and hardness test), while the physical test was thermal behavior test. Results of this study showed the values of (tensile strength, hardness, thermal conductivity and thermal diffusivity) properties increased when adding the (nano-Al_2O_3) particles in PMMA composite materials for prosthetic denture base materials. While, the values of impact strength and flexural strength properties were decreased.*

***Keywords**- PMMA, Nano-Al_2O_3 Particles, Fiber Glass, Tensile, Impact, Hardness, Flexural, Thermal Properties.*

How to cite this article: Q.A. Hamad, "Study the Effect of Nano-Al_2O_3 and Fiber Glass on Mechanical and Physical Properties of PMMA Composites for Prosthetic Denture," *Engineering & Technology Journal*, Vol. 36, No. 7, pp. 715-722, 2018.

1. Introduction

The first known materials by human were the metals, ceramics and polymers and each one has their own specific properties, technology has developed rapidly over the past decade and so does the material industry, because those two are very much connected each material has their own specific property, some materials need more enhancement so by combining two or more materials together to get more developed materials and that's where the composites materials take place [1]. When the world developed so does the needs of the human body, a need for replacing an arm , a denture , a tooth and even a valve required special materials that were compatible and nontoxic with the human skin and human body. In addition, it is free from any side effects such as cytotoxicity, infection, bacteria, allergen city and does not cause any irritation on the skin [2]. Research found that out of the three main categories (Metals, Ceramics and Polymers) that Polymer is more preferred in the medical field, because of its properties that are close to a certain degree to the human body. So it became the matrix for most of the medical field materials, with the addition of reinforcements for strength and other properties (Metals and Ceramics) [3]. Polymer composites materials have good elastic modulus, high strength, high creep strength and high heat deflection compared to the pristine polymers and they are suitable for several orthopedic and orthodontic applications. Each type of material has good properties, which make them suitable for biomedical applications [4]. The most commonly polymers used in biomedical and dentistry application are acrylic resin. In the 20th century, acrylic resin and other plastic materials were used as denture base materials due to their attractive properties. Acrylic resins are the most widely used in dentistry application and accepted among all denture base materials, and it was estimated that they represent (95%) of the polymer in prosthodontics applications. Poly (methyl methacrylate) is favored for its good properties such as satisfactory mechanical properties, except the impact and fatigue strength, dimensional stability and it has high Tg temperature, the color which can similar to the human's internal skin. PMMA can be fabrication and processed by many methods such as casting, extrusion, injection and thermoforming method [5]. The poly (methyl methacrylate) (PMMA) properties are modified when reinforced by

https://doi.org/10.30684/etj.36.7A.3
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adding many particles or fibers, this is approach the scientist to study the effected of reinforced materials on the mechanical and physical properties for the composite material that using for complete and partial denture base materials.
In this study, new type of self-curing (cold curing) poly (methyl methacrylate) resin as new fluid resin matrix, reinforcing with (nano-Al_2O_3) particles and random fiber glass to prepare hybrid composite specimen for complete and partial denture base material with having excellent properties, in order to avoid or reduce many problems such as fracture that occur in for prosthetic denture base materials.

2. Prosthetic Dentistry

In prosthetic dentistry the partial denture are made to replace one or more of lost teeth and they have the advantage that support and retention can be derived from adjacent retained natural teeth and using design features. A complete denture replaces all the teeth in one dental arch. For people who are already edentulous, the objectives of prosthetic treatment they would be have good speech, good appearance, effective chewing and biting, comfort and the ability to engage in various social and interpersonal activities. Figure 1 shows images of complete and partial prosthetic denture [6].

3. Materials Used

I. Acrylic Resin Denture Base Material

In this study the matrix material included cold (self) curing PMMA that used as fluid resin matrix, type (Castavaria), made from (Vertex – Dental Company), to preparation specimens of composite prosthetic denture base. This type of materials distinguishes by softer feel, low molecular weight, color stable in the long run, minimized shrinkage, stable polymerization cycle with a perfect end result, the acrylic is long pourable and modelable for a long period of time. Nevertheless, have some disadvantage properties such as: low strength, low hardness and more difficult using during fabrication [7]. Table 1 shows some physical and mechanical properties of self-cure PMMA resin, type (Castavaria), which used in this study according to (Vertex – Dental Company).

Figure 1: Images of Prosthetic Denture.

Table 1: Mechanical and Physical Properties of Self Cure PMMA Resin

Tensile Modulus (GPa)	Impact Strength (KJ/m^2)	Flexural Strength (MPa)	Water Absorption (%)	Density (g/cm^3)
1.63-3	**8.3**	**79**	**2.5**	**1.19**

II. Reinforcing materials

Two types of reinforcing materials were used in this study with selected volume fraction it was added to the acrylic powder which including:

1. Nano Aluminum Oxide Particles

Aluminum oxide is supplied as (nano-particles) that added with different volume fraction of (1%, 2%, 3% and 4%), also it has high purity about (99.9%). The structure of aluminum oxides have strong bonding which gives its high strength, stiffness, hardness, wear resistant, and good thermal conductivity of alumina [8].
The result of particle size and particle size distribution of (nano-Al_2O_3) particles is obtained by using atomic force microscopy (AFM) was carried out in Baghdad University Laboratories, which shows the average diameter was (57.50 nm). While the result of particle size distribution for (nano-Al_2O_3) as shown in Figure 2. Table 2 shows some physical and mechanical properties of (Al_2O_3) particles that used in this study according to the manufacture's instruction.

SPM Scanning Probe Microscope

Granularity Cumulating Distribution Report

Sample:Al_2O_3	**Code:** Sample Code
Line No.:lineno	**Grain No.:**614
Instrument: CSPM	**Date:**2016-12-05

Avg. Diameter:57.50 nm

Diameter(nm) <	Volume (%)	Cumulation(%)	Diameter(nm) <	Volume(%)	Cumulation(%)	Diameter(nm) <	Volume(%)	Cumulation(%)
10.00	0.16	0.16	55.00	5.86	49.19	100.00	1.95	94.79
15.00	1.30	1.47	60.00	7.33	56.51	105.00	1.63	96.42
20.00	2.61	4.07	65.00	6.84	63.36	110.00	1.95	98.37
25.00	3.91	7.98	70.00	6.19	69.54	115.00	0.16	98.53
30.00	7.00	14.98	75.00	4.72	74.27	120.00	0.16	98.70
35.00	7.33	22.31	80.00	3.09	77.36	130.00	0.16	98.86
40.00	9.61	31.92	85.00	5.70	83.06	135.00	0.16	99.02
45.00	5.86	37.79	90.00	5.05	88.11	140.00	0.81	99.84
50.00	5.54	43.32	95.00	4.72	92.83	180.00	0.16	100.00

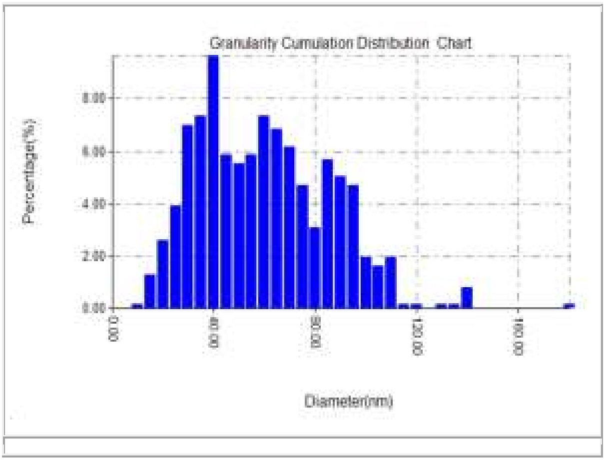

Figure 2: AFM Test of Nano-Al_2O_3 Particles.

2. Random Glass Fiber (RFG)

Fiber glass is a strong, lightweight material, cheaper, more readily molded into complex shapes and it is used for many applications which include aircraft, boats, automobiles, bath tubs and, swimming, hot tubs, water tanks, roofing, pipes, cladding, casts and possible using in dentistry field as denture. The E-glass fiber used in this study made from (Mowding LTD. UK Company), as random woven with constant volume fraction of (4 %). Table 2 shows some mechanical and physical properties of glass fiber that used in this study according to the manufacture's instruction.

4. Test Specimens Preparation

I. Proportioning and Mixing of Acrylic

The PMMA denture base materials consist of polymer powder and monomer liquid (methyl methacrylate, MMA). The standard proportion in mixing ratio for cold cure (self-cure) acrylic resin is usually about (17 g) polymer powder (PMMA) and (9.5 g) monomer liquid (MMA) (1.7 g/0.95g) by weight according to the manufacturer's instructions of manufacturer company. This ratio was effect on the workability of the mixture, dimensional changes and toxicity of acrylic resin specimens [9]. This type of cold cure acrylic resin is moldable for a long period of time, where the mixture was mixed of liquid (MMA) in the clean and dry container (glass beaker), follow after that by slow addition of dry powder (PMMA) to liquid (MMA), the mixture was stirred at room temperature and poured with thin straight line in the center of opening glass mould with maximum time about (4.5 min). Woven fibers cutting in rectangular shape of dimensions (19×19) cm which slightly less than those of the test mould (Glass mold) to avoid the exposure of fiber to the environment. The part of acrylic mixture was poured into the mould which nearly filled half of the mould then the pre-prepared woven mat fiber (Glass fiber) were placed over the lower half of the mixture then the other half of the mixture was poured into the mould over the woven mat fibers in this stage using glass rod during pouring of mixture in order to ensure the fiber well wetted with acrylic dough mixture. Then the mould was vibrated and vibrated from side to remove any entrap gas bubbles that may be found in this mixture, which may cause creating the voids that may be affecting on properties of the final hybrid composite materials and left mould to stand on the bench top at room temperature for (8-13 min) from beginning of mixing process as working time to increase the viscosity of mixture and surface of casting become hard and matt, this is process as shown in Figure 3.

II. Curing Cycle and Cooling Process

The curing done for all specimens after casting process by place it's inside the oven at (60 ºC) for (30 min), for complete polymerization of specimens under this condition in short time and give minimum level of residual monomer. The castings of composite specimens after curing process stand outside of the oven at room temperature about (30 min) to complete the cooling and hardening of specimen. After cooling, the specimens was de-mould to remove from the mould carefully and cleaning, as shown in Figure 4.

Table 2: Some Mechanical and Physical Properties of (Al_2O_3) Particles and (E-Glass) Fibers.

Material	Compressive Strength (MPa)	Tensile Strength (MPa)	Thermal Conductivity (W/m.K)	Density (g/cm^3)
Al_2O_3	2600	260	35	3.9
E-Glass Fiber	4500	3450	1.3	2.58

Figure 3: Casting of Composite Specimens in Glass Mould.

Figure 4: Composite Prosthetic Dentures Specimens after Curing Process.

All specimens before any test is carried out in air at room temperature (23 ± 2) ℃ after complete finishing and polishing processes was done with the sand paper or silicon carbide papers with continuous water cooling. While polishing was done by using fine cloth with diamond paste in grade of (2 µm), immersion the specimens in distilled water at (37 ± 1) ºC for (48 hr), in order to remove any residual monomer and release residual stress, also to ensure that the denture base materials remains in semi oral environment [10].

5. Results and Discussion

I. Tensile Test for Modified Composites

The tensile test is performed according to (ASTM D638-03) by using tensile machine (universal testing machine), type (Instron) at strain rate of (5mm/min) and load was applied equal to the (5 kN) until break the specimen occur. Figure (5) shows the standard specimen of tensile test [11]. Figure 6, shows the effect of adding (nano-Al_2O_3) particles with different volume fractions on the tensile strength of (PMMA-F.G) composite for prosthetic denture base materials that prepared in this study.

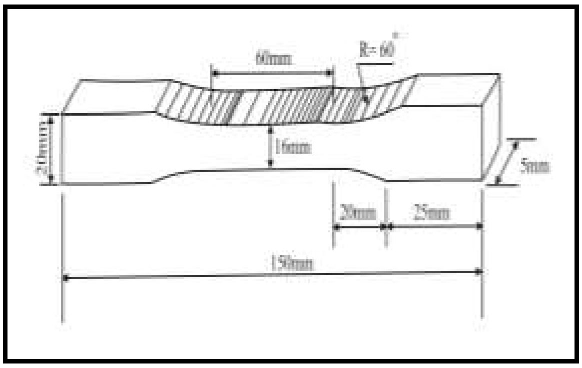

Figure 5: Schematic Specimen for Standard Specimen of Tensile Test.

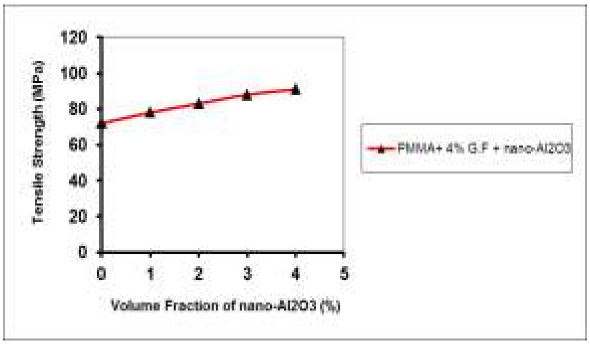

Figure 6: Tensile Strength of PMMA Hybrid Laminated Composite as Function of (Nano-Al_2O_3) Particles (Vol. %) in Composite.

Above Figure, it can be noticed the tensile strength values of PMMA hybrid laminated composite specimens increased with increasing the volume fraction of (nano-Al_2O_3) particles, because of this is due to the contribution of (nano-Al_2O_3) particles, glass fiber to be carried out of the load applied on the hybrid laminated composite specimen with suitable the nature and volume fraction of them. In addition to regular and randomly distribution of (nano-Al_2O_3) particles inside the PMMA resin and ease penetration the matrix material through the particle and fiber, this leads to create fully interfaces between the matrix material and reinforcing material, and leads to an increase in the ability of stress transfer from the PMMA composite to the hybrid laminated composite [12]. Thus, the tensile strength value increased from (72 MPa) for (PMMA-F.G) composite specimen (as referenced) to reach higher value of tensile strength (91 MPa) for hybrid laminated composite materials (PMMA-F.G- 4% nano-Al_2O_3).

II. Impact Test for Modified Composites

The impact test is performed according to (ISO-180) and (ADA Specification No.12, 1999), by using unnotched Izod Impact test machine type is (XJU series pendulum Izod impact testing machine), Figure 7 shows the standard of unnotch specimen of impact test [13]. Figure 8, shows the effect of adding (nano-Al_2O_3) particles with different volume fractions on the impact strength of (PMMA-F.G) composite for prosthetic denture base materials that prepared in this study.

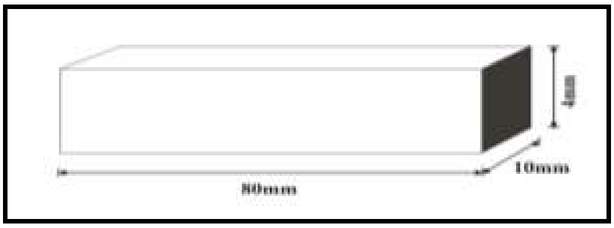

Figure 7: Schematic Specimen for Standard Specimen of Impact Test.

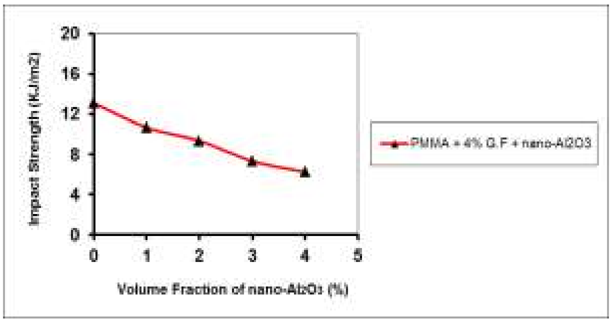

Figure 8: Impact Strength of PMMA Hybrid Laminated Composite as Function of (Nano-Al_2O_3) Particles (Vol. %) in Composite.

From this Figure can be noticed the impact strength values of PMMA hybrid laminated composite specimens decreased with increasing the volume fraction of (nano-Al_2O_3) particles, because of any increasing in these particles numbers, it will be act as points for localized stress concentration regions from which the failure will begin. Furthermore, the natural of these particles is brittleness and weakness in the ability of resistance to impact loads comparing with PMMA resin and fiber glass. Also may be because aggregated of nano-particles, which have high surface energy [14]. Thus, the impact strength value decreased from (13.1 KJ/m^2) for (PMMA-F.G) composite specimen (as referenced) to reach lower value of impact strength (6.25 KJ/m^2) for hybrid laminated composite materials (PMMA-F.G- 4% nano-Al_2O_3).

III. Flexural Test for Modified Composites

The flexural test is performed according to (ASTM D790). All data measured from three point bending test machine by using the same tensile machine at strain rate of (5 mm/min) and load was applied equal (5 kN) until break the specimen occur, Figure 9 shows the standard specimen of flexural test [15]. Figure 10 and Figure 11, shows the effect of adding (nano-Al_2O_3) particles with different volume fractions on the flexural strength and flexural modulus of (PMMA-F.G) composite for denture base materials that prepared in this study.

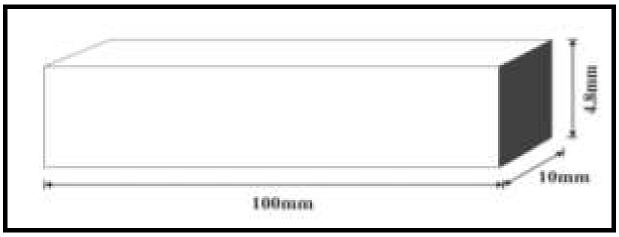

Figure 9: Schematic Specimen for Standard Specimen of Flexural Test.

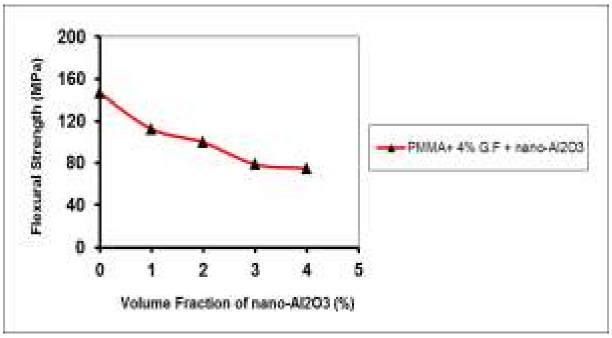

Figure 10: Flexural Strength of PMMA Hybrid Laminated Composite as Function of (Nano-Al_2O_3) Particles (Vol. %) in Composite.

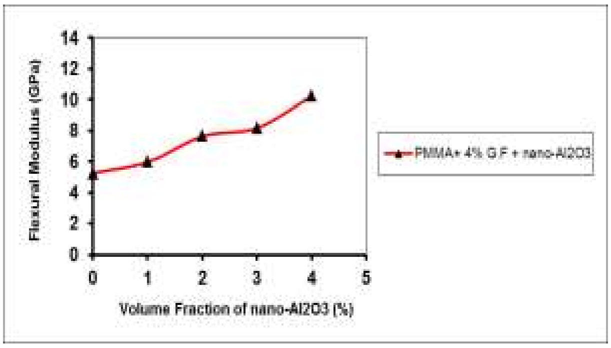

Figure 11: Flexural Modulus of PMMA Hybrid Laminated Composite as Function of (Nano-Al_2O_3) Particles (Vol. %) in Composite.

Above Figures it can be noticed the flexural strength values of PMMA hybrid laminated composite specimens decreased with increasing the volume fraction of (nano-Al_2O_3) particles, because of any increasing in the particle number lead to agglomerate its by its high surface energy, which it will be act as localized stress concentration regions from which the failure will begin, also this decreasing may be due to the fact the flexural strength of (nano-Al_2O_3) particles is lower than PMMA matrix and random woven fiber glass. Furthermore, the (nano-Al_2O_3) particles are brittleness and weakness in ability to resistance of flexural loads comparing with PMMA resin and fiber glass [14]. Thus, the flexural strength value decreased from (146.25 MPa) for (PMMA-F.G) composite specimen (as referenced) to reach lower value of flexural strength (74.92 MPa) for hybrid laminated composite materials (PMMA-F.G- 4% nano-Al_2O_3). While, the flexural modulus value increased with increasing the volume fraction of (nano-Al_2O_3) particles, from (5.242 GPa) for (PMMA-F.G) composite specimen (as referenced) to reach higher value of flexural modulus (10.23 GPa) for hybrid laminated composite materials (PMMA-F.G- 4% nano-Al_2O_3). This is due to the contribution of each (nano-Al_2O_3) particles, and glass fiber to be carried out of the load applied on the hybrid laminated composite specimens that suit their mechanical properties, nature and geometry as well as their volume fraction. Therefore, using of (Al_2O_3) particles which have flexural modulus more than glass fiber and PMMA matrix materials. As well as the regular and randomly distribution of (Al_2O_3) particles inside the PMMA matrix and ease penetration the matrix material through the particles and woven mat fibers to formation good interfaces to give strong bonding between the matrix and reinforcing material, all these reasons lead to increase of flexural modulus values [16].

IV. Hardness Test for Modified Composites

The hardness test is performed according to (ASTM D2240) by used dorumeter hardness test, type (Shore D) at load applied equal to 50 N and depressing time of measuring equal to (15sec). The surface of specimens must be smooth in zone testing, and the minimum thickness of the specimen is (3 mm) with diameter more than (30mm). Each specimen was tested seven times on different areas of each specimen at same time and take in the middle of specimen not on the edge of its, finally the average value was taken. Figure 12 shows the standard specimen for hardness test [17]. Figure 13, shows the effect of adding (nano-Al_2O_3) particles with different volume fractions on the hardness of (PMMA-F.G) composite.

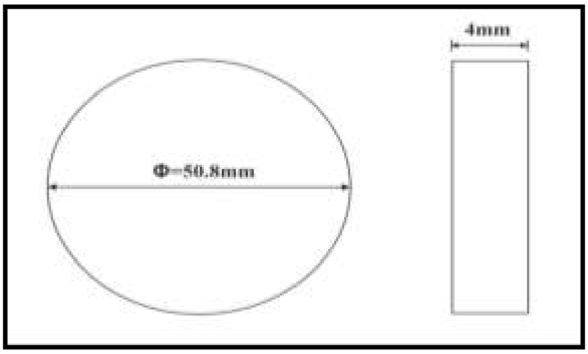

Figure 12: Schematic Specimen for Standard Specimen of Hardness Test.

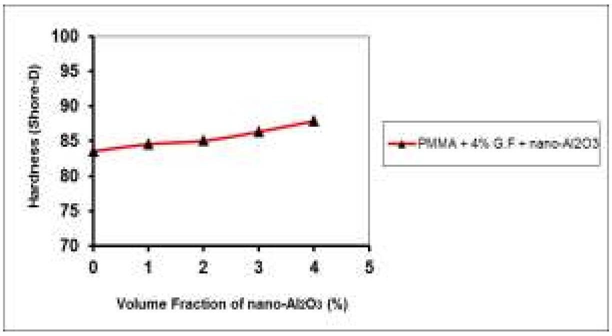

Figure 13: Hardness of PMMA Hybrid Laminated Composite as Function of (Nano-Al_2O_3) Particles (Vol. %) in Composite.

It can be noticed the hardness values of PMMA hybrid laminated composite specimens increased with increasing the volume fraction of (nano-Al_2O_3) particles, due to the high hardness and brittleness of Al_2O_3 particles as compared with glass fiber and PMMA matrix materials which have low hardness values. Furthermore, the high wettability between the matrix and these particles, which lead to make the harder surface of hybrid laminated composite that lead restricting the movement of the matrix and low resistance to the load applied on it. Therefore find result, the slightly increases in the values of hardness with increasing the volume fraction of (nano-Al_2O_3) particles [18]. Thus, the hardness value increased from (83.53) for (PMMA-F.G) composite specimen (as referenced) to reach higher value of hardness (87.86) for hybrid laminated composite materials (PMMA-F.G- 4% nano-Al_2O_3).

V. Thermal Behavior Test for Modified Composites

This test is performed according to apparatus manual and standard specifications instrument, by using (Hot Disk Thermal Constant Analysis) which supplied by heating power was (0.022 Watt), and resistance of (11.56 Ω) to transform the thermal energy which passes through the specimen, when its placed inside device to measure of thermal transport properties (thermal conductivity and thermal diffusivity), the Hot Disk sensor as shown in Figure 14 [19]. The specimen that used in this study have dimensions (20mm × 20mm), thickness (5mm), and must be clean both surface and free from oil, grease and other foreign matters, the basic hot disk sensor operation as shown in the Figure 15 [20].

1. Thermal Conductivity for Modified Composites

Figure 16, shows the effect of adding (nano-Al_2O_3) particles with different volume fractions on the thermal conductivity of (PMMA-F.G) composite.

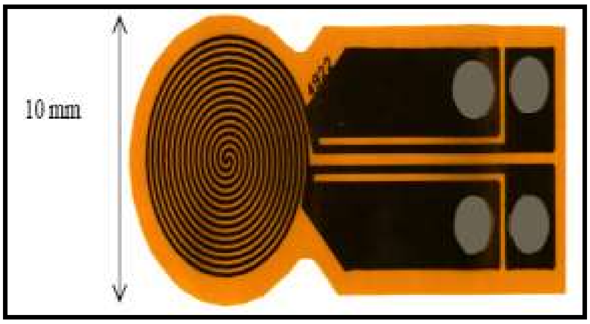

Figure 14: Hot Disk Sensor

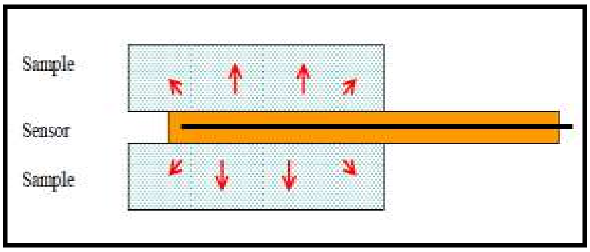

Figure 15: Hot Disk Sensor Operation.

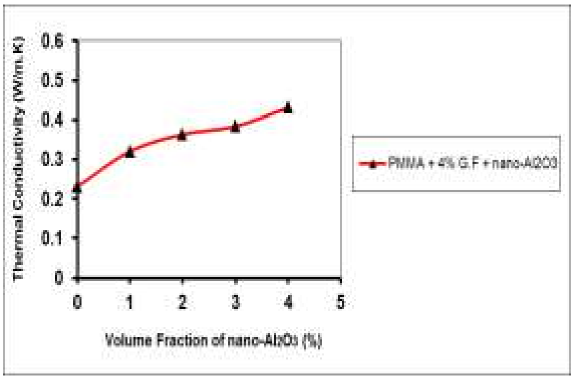

Figure 16: Thermal Conductivity of PMMA Hybrid Laminated Composite as Function of (Nano-Al_2O_3) Particles (Vol. %) in Composite.

Above Figure it can be noticed the thermal conductivity values of PMMA hybrid laminated composite specimens increased with increasing the volume fraction of (nano-Al_2O_3) particles, this is due to the fact the thermal conductivity value of (nano-Al_2O_3) particles is higher than PMMA resin and fiber glass. In addition, the presence of these particles is made to fill or diminish the spaces and voids which were inside the PMMA matrix and fibers. Finally, the result leads to facilitate the process of the heat transfer through composite specimens. Therefore, that leads to an increase of the thermal conductivity values of the hybrid laminated composite specimens [21]. Thus, the thermal conductivity values increased from (0.23115 W/m.K) for (PMMA-F.G) composite specimen (as referenced) to reach to the higher value of thermal conductivity (0.43108 W/m.K) for hybrid laminated composite materials (PMMA-F.G- 4% nano-Al_2O_3).

2. Thermal Diffusivity for Modified Composites

Figure 17, shows the effect of adding (nano-Al_2O_3) particles with different volume fractions on the thermal diffusivity of (PMMA-F.G) composite. From this Figure can be noticed the thermal diffusivity values of PMMA hybrid laminated composite specimens increased with increasing the volume fraction of (nano-Al_2O_3) particles, for the same review reasons for the thermal conductivity values results. Thus, the thermal diffusivity values increased from (0.1969 mm^2/S) for (PMMA-F.G) composite specimen (as referenced) to reach to the higher value of thermal diffusivity (0.3826 mm^2/S) for hybrid laminated composite materials (PMMA-F.G- 4% nano-Al_2O_3).

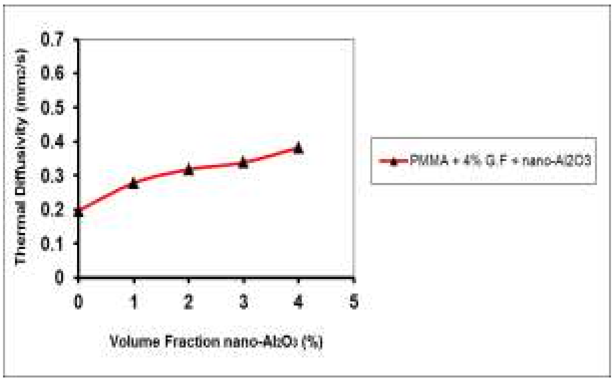

Figure 17: Thermal Diffusivity of PMMA Hybrid Laminated Composite as Function of (Nano-Al_2O_3) Particles (Vol. %) in Composite.

6. Conclusions

According to the experimental results of hybrid laminated composite materials in this study, can be the conclusions the following sentences:

1- The addition of fiber glass and (nano- Al_2O_3) particles has a noticeable effect on most mechanical and physical properties of PMMA hybrid laminated composite for prosthetic denture base materials with increasing the volume fractions of (nano- Al_2O_3) particles more than using fiber glass only, specially (tensile strength, flexural modulus, hardness, thermal conductivity and thermal diffusivity).

2- Some mechanical properties values such as (flexural strength and impact strength) of PMMA hybrid laminated composite materials for prosthetic denture base materials, were decreased with increasing of the volume fraction of (nano-Al_2O_3) particles.

3- The maximum values for (tensile strength, flexural modulus, hardness, thermal conductivity and thermal diffusivity) properties, were obtained in the PMMA hybrid laminated composite materials (PMMA-F.G-4% nano-Al_2O_3). While the minimum value for (flexural strength and impact strength) properties, were obtained in the PMMA hybrid laminated composite materials (PMMA-F.G-4% nano-Al_2O_3).

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