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Design and Construction of a New Automated Device for Testing the Scratch Resistance of Polymeric Materials

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Abstract

This paper aims to design and construct of an automated device for testing the scratch resistance of the polymeric materials by measuring the force required to cause a scratch on the surface of polymeric materials as well as calculating the friction coefficient from the input and output of the device. The device was made of materials available in the local market and some parts were manufactured in local mechanical workshops. The device consists of four main parts were mechanical parts, scratching mechanism parts, electrical and electronic parts and the device operating program. The device designed in this work has the following specifications: normal load (0.1 N - 325 N), sliding speed (1 mm/s - 35 mm/s), tangential force measured by the load cell (0.1 N to 294 N), the samples dimensions (length: 10 - 195 mm, width: 10 - 125 mm, and thickness: 0.25 - 50 mm), maximum scratch length of 195 mm, and the height of the indenter from the platform surface (0.25 mm to 50 mm). Scratch test and calculation of the friction coefficient were performed for samples of pure and reinforced PMMA by Silicon Oxide nanoparticles (SiO_2). The results showed an increase in scratch resistance and a decrease in the friction coefficient with increasing the weight ratio of SiO_2. Also, the ability of the designed device to measure the tangential force required for scratching accurately and quickly and in simple steps.

Keywords: Scratch device, Polymer, Scratch resistance, Friction.

1. Introduction

The scratch resistance test is one of the most important tests conducted on polymeric materials to determine its surface properties so that researchers and manufacturers can then devise methods and treatments to improve these properties. Scratch resistance test for polymers depends on many parameters such as indenter geometry, applied normal load and sliding speed. Indenter geometry includes the indenter type (conical or spherical), radius and apex angle of the tip and attack angle of the indenter. The strain hardening and height of the polymeric material accumulated on both sides of the scratch groove increases as the attack angle of the indenter increases [1]. The value of applied normal load required to start scratching on the surface of the polymeric material is greatly influenced by the geometry of the indenter, this value does not mean anything without accurate description of the tip used [2]. When the scratching begins to occur on the surface of the polymeric material, tensile and compressive stresses are generated behind and in front of the indenter respectively, which associated mainly with the indenter geometry. The polymeric material begins fracturing at the bottom of scratch groove when the tensile stress value reaches the value of ultimate tensile strength [3]. The wear volume resulting from the scratching is proportional to the applied normal load [1, 4]. An increase in sliding speed lead to decreases the true contact radius and increases the mean strain in the contact region [5].

In the literature, the scratch test in all researches related to the study of scratch resistance of polymeric materials was performed using various devices with different specifications. For example, Amerio et al. used a CSM scratch device (Micro-Combi Tester) with specifications: normal load (0.05N-30N), sliding speed (0.4-600 mm/min) and maximum scratch length of 120 mm [6]. A new device based on the moving head of tensile machine (Instron 4502) with specifications: normal load (0.05 N - 5 N) and sliding speed (1 µm/s to104 µm/s) was used by Gauthier and Schirrer [7]. Christopher et al. used a CSM Nano-scratch tester with specifications: normal load (10 µN - 1 N), sliding speed (0.4 - 600 mm/min), tangential force measured by load cell (6 µN - 1 N) and dimensions of the sample (20 mm × 120 mm) [8]. Fond et al. used a micro-visioscratch device with specifications: normal load (0.05 N - 35 N), sliding speed (1 µm/s - 104 µm/s) and temperature range (- 70 °C to + 120 °C) [9]. Scratch apparatus with specifications such as sliding speed (1 µm/s - 15 mm/s), normal load (0.05 N - 5 N) and temperature range (- 70 °C to + 120 °C) was used by Lafaye et al. [10]. Manshaa et al. used a micro-visioscratch device with specifications: normal load (0.05 N - 35 N) and sliding speed (10^{-3} mm/s -15 mm/s) [5]. Surampadi et al. used a device with specifications: normal load (0.5 N - 10 N), maximum sliding speed of 10 mm/s and three indenters (loop, needle and Hoffman) [11].

Scratching occurs as a result of friction between the surface of the polymeric material and the body that causes the scratch, hence the importance of determining the friction coefficient of the polymeric material to improve its resistance to scratching. The ratio between the tangential force that causes the movement of the scratching tip and the vertical force due to the load applied on the scratching tip was called the apparent friction coefficient which includes two friction coefficients types, one of which was the result of separation at the region between scratched surface and tip was called coefficient of true local friction, the other was a result of plowing the material formed in front of the moving tip was called geometrical friction coefficient. The apparent friction can also be divided into adhesive friction and plowing friction and the second term may include two terms, one for viscosity and the other for plasticity.

The scratching resulting from various contact conditions provides an appropriate and reliable method for studying the mechanical properties of polymers [5, 3, 12-16]. Level of the strain in contact region and the senility of polymer are factors on which the true local friction depends [17, 18]. The coefficient of actual friction between the indenter and the surface of the polymeric material is affected by the contact geometry, which in turn is greatly influenced by the indenter geometry [16]. The surface properties of polymeric materials are improved by adding certain materials in different ratios such as zirconium dioxide (ZrO_2), aluminum oxide (Al_2O_3), silicon oxide (SiO_2) and calcium carbonate ($CaCO_3$) [19-24].

The purpose of this work is to design and construct a new automated device for the scratch resistance test of polymeric materials to study the evolution in the surface properties of pure and reinforced polymeric materials.

2. Device Description

As stated in the introduction, the scratch resistance test of polymers depends on indenter geometry, sliding speed and applied normal load. In order to conduct scratch test with wide ranges for sliding speed and applied load as well as the ability to use samples in various dimensions and sizes, this device is designed to achieve all these requirements, as shown in Fig. 1.

Fig. 1 Scratch resistance device.

Fig. 2 shows the mechanical parts of the device, which consists of metal box, two sliding bar with two sliders, four serrated pillars (4 legs), stepper motor, stainless steel serrated shaft, flexible disk coupling, serrated flange, two metal plates with different dimensions, three iron hoops, set of iron discs and set of screws. Fig. 3 shows the parts of the scratching mechanism that consists of two iron pillars in L-shaped, set of serrated and non-serrated bars, connecting pieces and indenter holder.

The parts of the electrical circuit used to provide the device with electrical power are shown in Fig. 4, which consists of: (a) electric wire (2 × 2.5 mm), (b) power plug to connect the electrical wire to the device, (c) electrical fuse, (d) switch on/off, (e) operating indication lamp and (f) an electrical transformer to convert voltages from 220 Volts to 19.5 Volts. The parts of the electronic circuit used to control the device through the computer are shown in Fig. 5, which consists of :(a) stepper motor operator (Micro step Driver), (b) logical microcontroller (Lab Jack), (c) electric capacitors, (d) Integrating Circuit (IC), (e) electrical resistors and (f) Load cell. The connecting circuit of these parts is shown in Fig. 6.

Fig. 2 Mechanical parts of device.

Fig. 3 Scratching mechanism parts.

Fig. 4 Electrical parts.

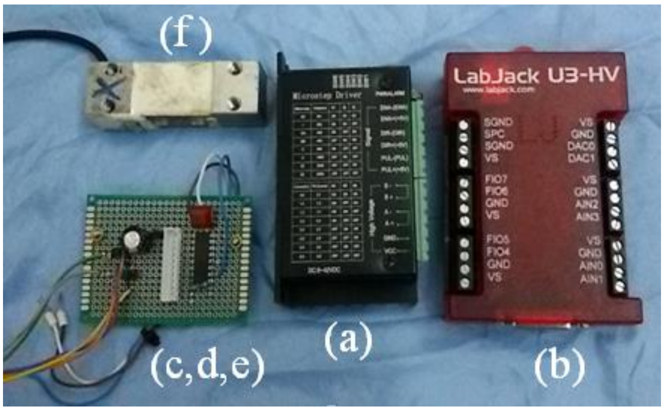

Fig. 5 Electronic parts.

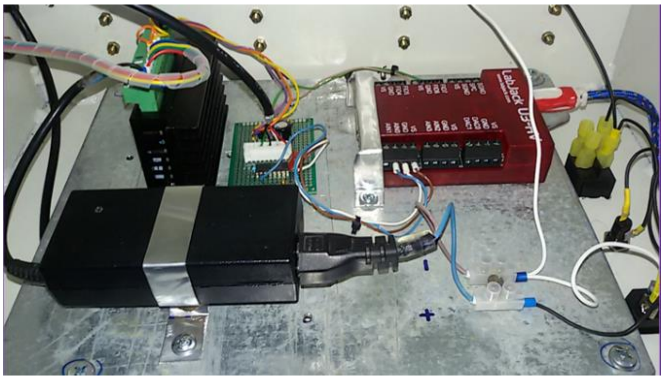

Fig. 6 The electrical and electronic circuit of the device.

In this device, automation (control of the device by a computer program) was adopted to control the scratching mechanism, enter of variables and display results; the Visual Studio software was prepared using Visual Basic programming language. The input data through the Visual Studio software are the sliding speed, required length of the scratch path, required time to take the readings, the distance between readings along scratch path and the direction of platform movement. While the outputs are the tangential force required to scratch the polymeric surface and graph of the relationship between tangential force and time or scratch path. When click on the program icon, the window shown in Fig. 7, which contains the following options: speed (mm/s), distant (mm), scale (N/V), and sliding ratio (mm/rev).

After selecting both the sliding speed and the scratch path length, when click on the (Test-1) button a new window will be appeared in Fig. 8, which contains the following options: (1) Reading Time, (2) Direction, (3) Sampling distant, (4) Start motion, (5) Stop, (6) Read, (7) Set as Zero: to make the resulting reading as a zero reading, this feature is used at the beginning of the test and without load so as not to introduce the force resulting from the friction of the device parts in the following readings when placing loads, i.e., it is the zeroing process of the device, (8) Save Data: to save readings that is recorded as a table in a file inside the computer. Fig. 9 shows the flow chart of scratch resistance test.

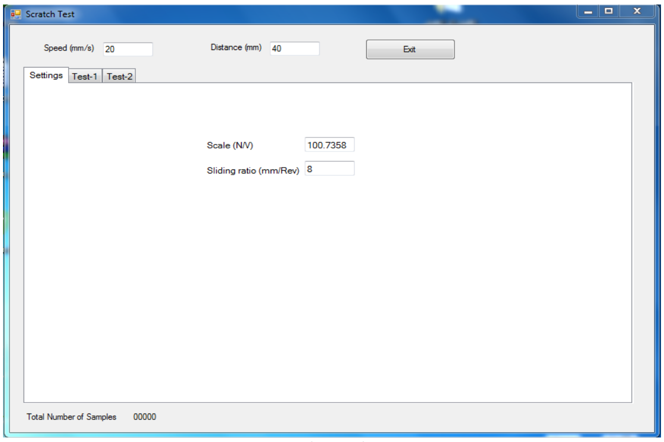

Fig. 7 The main interface of the device program.

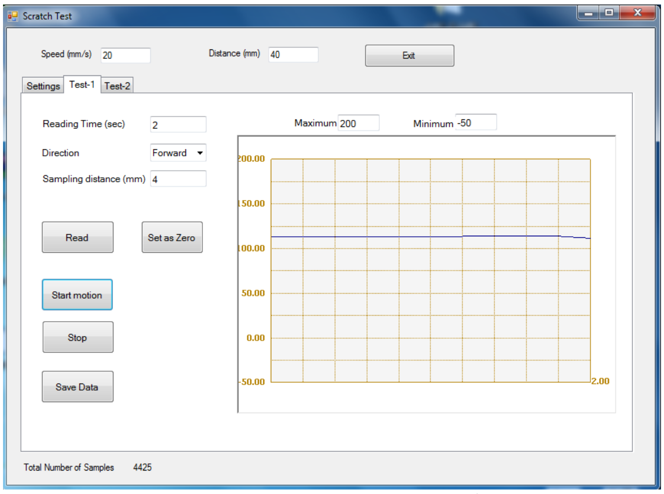

Fig. 8 Device operation settings interface.

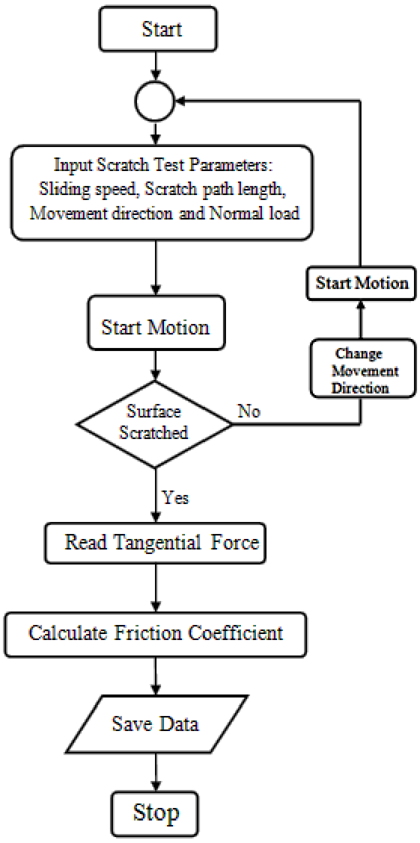

Fig. 9 Flow Chart of scratch resistance test.

3. Device Features

Features of the device can be summarized in the following points:

1. Range of linear speed of the moving platform (1 mm/s - 35 mm/s).
2. Range of applied normal load (0.1 N - 325 N) or (10 gm - 33000 gm).
3. The tangential force measured by the load cell ranges (from 0.1 N to 294 N).
4. The length of the scratching path ranges (from 5 mm to 195 mm).
5. The height of the indenter ranges (from 0.25 mm to 50 mm).
6. The dimensions of the samples that can be tested are length ranges (from 10 mm to 195 mm), width ranges (from 10 mm to 125 mm), and thickness ranges (from 0.25 mm to 50 mm).
7. Ability to change the speed of the moving platform and the applied normal load in a single scratch path.
8. Ability to measure the force required for scratching directly and easily through which the coefficient of friction can be calculated directly.
9. Ability to measure the scratch force required for the path as a whole and for specific points on the path.
10. The possibility of lifting the scratch mechanism to the comfort position allowing the user to install and remove samples and indenter in an easy and safe way.
11. The method of installing and removing both sample and indenter is simple and fast.
12. The device can be used to test the scratch resistance of both plastic and elastic materials.
13. The space occupied by the device in the laboratory is small due to the small size of the device.
14. The method of device operation is simple and easy does not require the user a lot of experience or time to learn.
15. The user of the device does not need many safety requirements.

4. Validation and Calibration of the Device

In order to validate the results of the device, the tangential force measured by the device during the scratch resistance test of polymethyl methacrylate (PMMA) and polycarbonate (PC) was used to calculate friction coefficient of both materials. A comparison was made with the values of the friction coefficient mentioned by the manufacturer in material data sheet, the results showed an excellent convergence as shown in Fig. 10.

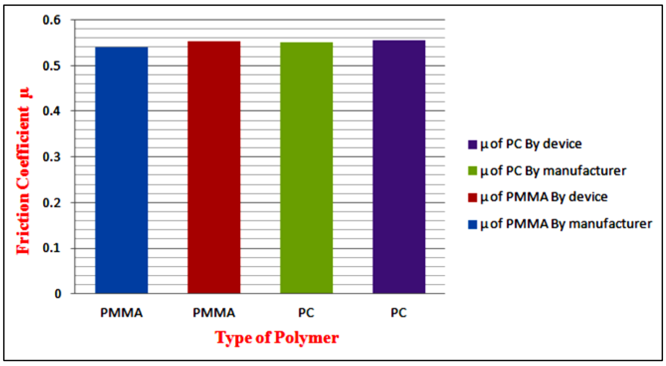

Fig. 10 Comparison of friction coefficient values of PMMA and PC.

Calibration of the linear velocity of the device and the load cell used to measure the tangential force were carried out in the laboratories of the Mechanical Engineering Department, College of Engineering in University of Basrah.

5. Experimental Work

In this part will be presentation the results obtained using the device during the current study. Samples of pure PMMA and reinforced with the addition of silicon oxide nanoparticles (SiO_2) in different weight ratios (1 wt %, 2 wt %, 3 wt % and 4 wt %) were prepared. The preparation of samples and the test of scratch resistance using the new device were all done in the laboratories of the Faculty of Materials Engineering in University of Babylon.

5.1. Materials

PMMA material used in this work is a spherical granule with diameter of 3 mm, the filling material is 99 % silicon oxide nanoparticles (SiO_2), APS: 10-30 nm, treated with silane coupling agent, the solvent used is tetrahydrofuran (C_4H_8O) with 99.7 % minimum assay (GC), as shown in Fig. 11. All materials used in this work are supplied by Areej Al Furat Company Bagdad, Iraq. In this work silicon oxide nanoparticles (SiO_2) treated with silane coupling agent is used as filler. Silicon oxide (SiO_2) is classified as a very hard material used as filler in many materials such as adhesive, paint, plastic, coatings and rubber to improve the hardness and scratch resistance for these materials. A well-known scientific fact is that covalent systems are always more hard, so the reason for the hardness of silica is that it forms a covalent bond with oxygen. The interphase region, which is the area between the inorganic filler (SiO_2) and organic polymer (PMMA), mainly affects the improvement of the properties of the resulting PMMA / SiO_2 composite, it depends on the type of coupling agent used. Silane coupling agent acts as a bonding agent to improve the adhesion between PMMA and SiO_2. Many desired changes can be obtained as a result of modification of the interphase region such as improve the SiO_2 distracting in PMMA, reduce viscosity and improve hardness and scratch resistance by formation of overlapping polymer networks.

(a) (b) (c)

Fig. 11 (a) PMMA granules, (b) silicon oxide nanoparticles (SiO_2) and (c) tetrahydrofuran solvent.

5.2. Casting Equipment

Casting molds are glass molds with dimensions of 220 mm length, 130 mm wide and 20 mm high. These molds are placed on horizontal adjustment tables to ensure uniform thickness of the cast material. The molds are covered by glass containers to protect them from unwanted external factors such as dust and wind as well as to ensure a saturated perimeter around the casting mold, as shown in Fig. 12.

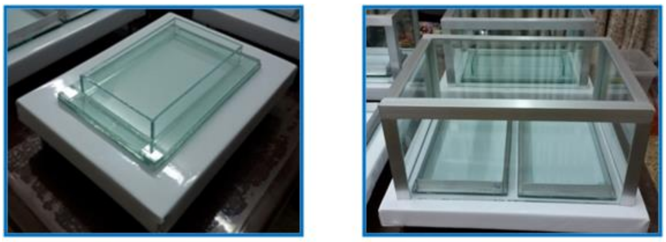

Fig. 12 (a) glass molds with (b) glass containers and horizontal adjustment tables.

5.3. Samples Preparation

Test samples are prepared by casting method, where the PMMA granules are mixed with nanoparticles (SiO_2) and solvent in a closed flask, placed on the stirrer with 1500 rpm for 2 hours at room temperature (20 °C). The solution is then poured into the glass mold placed on horizontal adjustment table and covers by a glass container and left under the sun for 5 days to ensure complete evaporation of the solvent, as shown in Fig. 13. The samples are then removed from the mold by placing them in a cold water tank for 30 minutes. Table 1 shows the ratios of mixing nanoparticles (SiO_2) with PMMA. The final stage of preparation of the samples is to compress them at a pressure of 0.5 MPa and temperature of 95 °C for 2 minutes using a hydraulic thermal press to ensure a smooth surface and free of bubbles, as shown in Fig. 14. The samples required for each test are cut using a laser cutting device.

Fig. 13 Preparation of PMMA/SiO_2 composites.

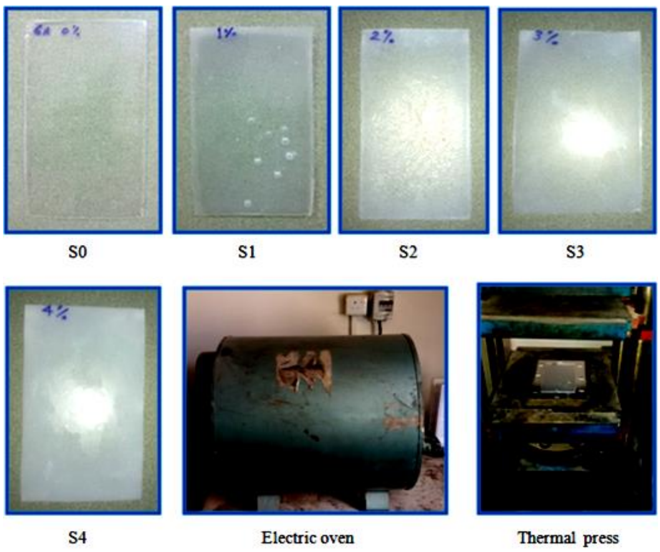

Fig. 14 Pressing process of samples.

Table 1 The mixing ratios between PMMA and SiO_2.

Sample Code	Wt % PMMA	Wt % SiO_2
S0	100	0
S1	99	1
S2	98	2
S3	97	3
S4	96	4

5.4. Scratch Test

A scratch resistance test were conducted at 20 °C and sliding speed of 20 mm/s, the applied load ranging from 1 N to 10 N and scratch path length of 40 mm. All samples were scratched by stainless steel indenter with conical tip, which has a radius of 0.2 mm and apex angle of 60°. The dimensions of the scratch test sample were 80 mm width, 130 mm length and 3 mm thickness. Tangential force required for scratching the surface of each sample measured by the device and the corresponding applied load value and the friction coefficient calculated are listed in Table 2.

Table 2 applied normal load (F_n) required for scratching, measured tangential force (F_t) and friction coefficient of PMMA/SiO_2 composites.

Sample code	Normal Load Required For Scratching F_n (N)	Measured Tangential Force F_t (N)	Friction Coefficient μ $\mu = F_t / F_n$
S0	3.581	1.987	0.554
S1	4.493	2.192	0.488
S2	5.268	2.297	0.436
S3	6.112	2.353	0.385
S4	7.122	2.421	0.340

6. Results and Discussion

Fig. 15 presents the tangential force distribution (F_t) against the length of the scratch path (40 mm) for each sample of the PMMA/SiO_2 composite. It should be noted that the values of tangential force (F_t) are approximately equal at all points along the scratch path and this is good evidence of the uniform distribution of nanoparticles (SiO_2) within the PMMA matrix. Fig. 16 shows the applied loads required (F_n) to start scratching for each PMMA sample as a function of the ratio of nanoparticles (SiO_2). It should be noted that the applied load increases with the increasing of nanoparticles (SiO_2) due to increase scratch resistance as a result of increase hardness due to the addition of nanoparticles which are characterized by high hardness.

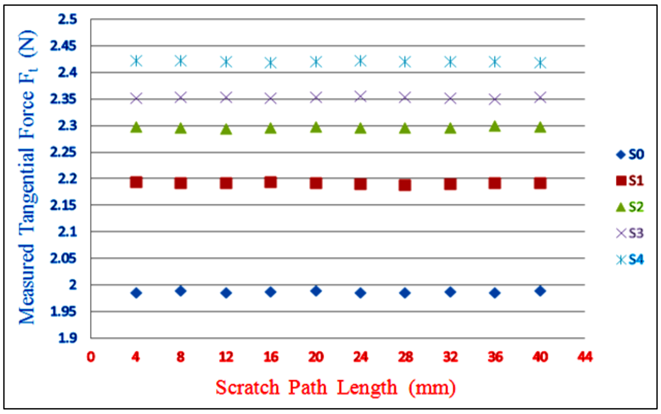

Fig. 15 Measured tangential force (F_t) against the scratch path length of PMMA/SiO_2 composites.

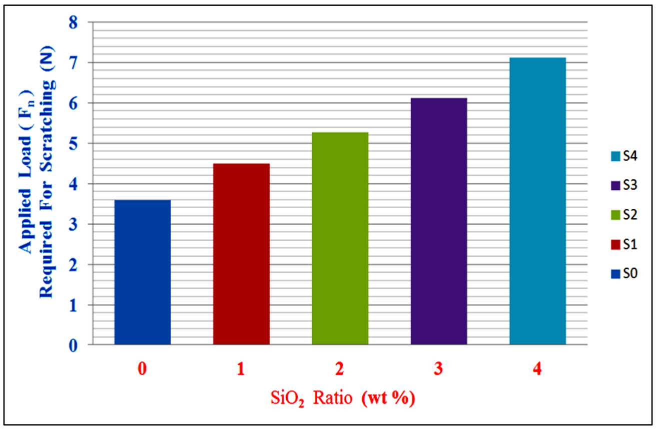

Fig. 16 Applied loads required for scratching (F_n) as a function of nanoparticles (SiO_2) ratio.

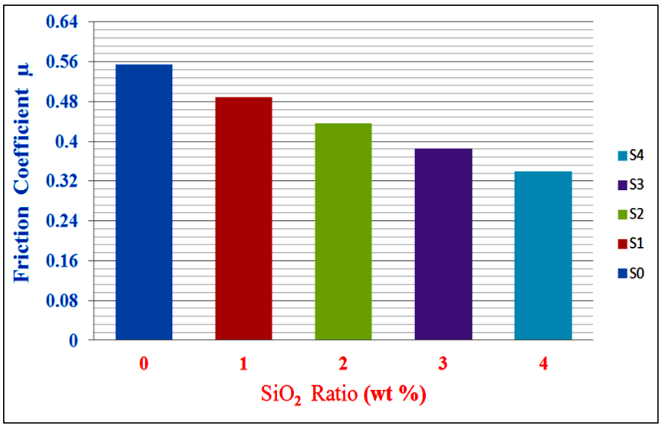

Fig. 18 Friction coefficient μ for PMMA/SiO_2 composites as a function of wt % SiO_2.

The friction coefficient μ of each sample can be calculated from the division of the measured tangential force (F_t), which represents output of the device on the applied normal load (F_n), which represents input of the device. Fig. 17 presents the friction coefficients of pure PMMA and reinforced PMMA against the scratch path length. The values of the friction coefficient μ for each sample were approximately equal along the scratch path. This indicates the homogeneity of properties along the scratch path at all points of the sample due to the uniform distribution of nanoparticles.

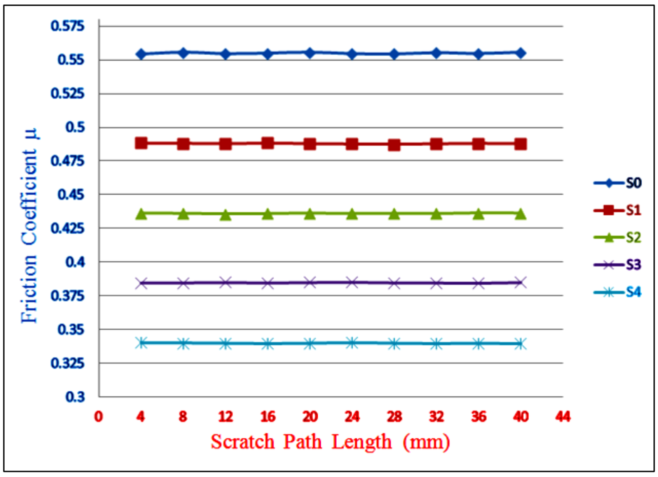

Fig. 17 Friction coefficient μ for PMMA/SiO_2 composites against the scratch path length.

Fig. 18 presents the friction coefficients of pure PMMA and reinforced PMMA against the nanoparticles (SiO_2) ratio, it can be noted that the friction coefficients of the reinforced PMMA are lower compared to pure PMMA. A decrease in the friction coefficient of the reinforced PMMA is also observed when the ratio of nanoparticles increases and the lowest value at 4 wt %. Decreasing in friction coefficient refers to the increase in the hardness and scratch resistance of the PMMA due to the addition of high hardness silica nanoparticles.

7. Conclusions

The most important points in this work can be included as follows:

1. The study proved the ability of the designed device to measure the tangential force required for scratching accurately and quickly and in simple steps as well as the possibility of calculating the coefficient of friction directly from the inputs and outputs of the device.
2. The study also proved the effectiveness of the program designed to control the device in terms of ease of enter variables and accuracy of outputs.
3. Scratch test results for PMMA showed an increase in scratch resistance with increasing the ratio of SiO_2, also showed a decrease in the friction coefficient with increasing the ratio of SiO_2 and the smallest value at 4 wt %.

References

[1] Michael J. Adams, Alexander Allan, Brian J. Briscoe, Peter J. Doyle, David M. Gorman, and Simon A. Johnson, "An experimental study of the Nano-scratch behavior of poly (methyl methacrylate)", Elsevier, Wear, Vol. 251, Issue 1-12, pp. 1579-1583, 2001.

[2] Vincentd D. Jardret and Warren C. Oliver, "On the robustness of scratch testing for thin films: the issue of tip geometry for critical load measurement", Materials Research Society, MRS Proceedings, Vol. 594, pp. 395-400, 1999.

[3] Vincent Jardret and Pierre Morel, "Viscoelastic effects on the scratch resistance of polymers: relationship between mechanical properties and scratch properties at various temperatures", Elsevier, Progress in Organic Coatings, Vol. 48, pp. 322-331, 2003.

[4] Ernest Rabinowicz, Friction and Wear of Materials, John Wiley & Sons, Inc. 2nd Edition, ISBN: 978-0-471-83084-9, 2013.

[5] M. Manshaa, C. Gauthiera, P. Gerardb and R. Schirrera, "The effect of plasticization by fatty acid amides on the scratch resistance of PMMA", Elsevier, Wear, Vol. 271, Issue 5-6, pp. 671-679, 2011.

[6] Ezio Amerio, Paola Fabbri, Giulio Malucelli, Massimo Messori, Marco Sangermano, Rosa Taurino, "Scratch resistance of nano-silica reinforced acrylic coatings", Elsevier, Progress in Organic Coatings, Vol. 62, Issue 2, pp.129-133, 2008.

[7] C. Gauthier and R. Schirrer, "Time and temperature dependence of the scratch properties of poly (methylmethacrylate) surfaces", Springer, Journal of Materials Science, Vol. 35, pp. 2121- 2130, 2000.

[8] Christopher M. Seubert, Mark E. Nichols, "Scaling behavior in the scratching of automotive clearcoats", Springer, Journal of Coatings Technology Research, Vol. 4, No. 1, pp. 21-30, 2007.

[9] C. Gauthier, A. -L. Durier, C. Fond, and R. Schirrer, "Scratching of a coated polymer and mechanical analysis of a scratch resistance solution", Elsevier, Tribology International, Vol. 39, Issue 2, pp. 88-98, 2006.

[10] S. Lafaye, C. Gauthier and R. Schirrer, "Analysis of the apparent friction of polymeric surfaces", Springer, Journal of Materials Science, Vol. 41, pp. 6441-6452, 2006.

[11] N. L. Surampadi, T. C. Pesacreta and R. D. K. Misra, "The determining role of scratch indenter radius on surface deformation high density polyethylene and calcium carbonate-reinforced composite", Elsevier, Materials Science and Engineering: A, Vol. 456, Issue 1-2, pp. 218-229, 2007.

[12] Brian J. Briscoe, Paul D. Evans, Enrico Pelillo, Sujeet K. Sinha, "Scratching maps for polymers", Elsevier, Wear Vol. 200, Issue 1-2, pp.137-147, 1996.

[13] Sujeet K. Sinha, Desmon B. J. Lim, "Effects of normal load on single-pass scratching of polymer surfaces", Elsevier, Wear, Vol. 260, Issue 7-8, pp. 751-765, 2006.

[14] Eric Felder, Jean-Luc. Bucaille, "Mechanical analysis of the scratching of metals and polymers with conical indenters at moderate and large strains", Elsevier, Tribology International, Vol. 39, Issue 2, pp. 70-87, 2006.

[15] B. J. Briscoe, S. K. Sinha, "Scratch Resistance and Localised Damage Characteristics of Polymer Surfaces - a Review", Materials Science & Engineering Technology, Mat.-wiss. u. Werkstofftech, Vol. 34, No. 10/11, pp. 989-1002, 2003.

[16] V. Jardret, H. Zahouani, J. L. Loubet and T. G. Mathia, "Understanding and quantification of elastic and plastic deformation during a scratch test", Elsevier, Wear, Vol. 218, pp. 8-14, 1998.

[17] C. Gauthier, S. Lafaye and R. Schirrer, "Elastic recovery of a scratch in a polymeric surface: experiments and analysis", Elsevier, Tribology International, Vol. 34, pp. 469-479, 2001.

[18] S. Lafaye, C. Gauthier and R. Schirrer, "A surface flow line model of a scratching tip: apparent and true local friction coefficients", Elsevier, Tribology International, Vol. 38, pp. 113-127, 2005.

[19] Vipul Asopa, S. Suresh, Meenakshi Khandelwal, Vivek Sharma, Shivalika S. Asopa, Laxman Singh Kaira, "A Comparative Evaluation of Properties of Zirconia Reinforced High Impact Acrylic Resin with that of High Impactacrylic Resin", Elsevier, The Saudi Journal for Dental Research, Vol. 6, pp. 146-151, 2015.

[20] Jyothi Atla, Prakash Manne, A. Gopinadh, Anche Samphat, Suresh Babu Muvva, Krishna Kishore, Chiramana Shandeep, and Harika Chittamsetty, "The Effect of Al_2O_3 Addition on the Thermal Diffusivity of Heat Activated Acrylic Resin", Journal of Clinical and Diagnostic Research, Vol. 7, No. 8, pp. 1797-1798, 2013.

[21] Hasanen A. Alnamel and Mohammed Mudhaffer, "The effect of Silicon dioxide Nano-Fillers Reinforcement on Some Properties of Heat Cure Polymethyl Methacrylate Denture Base Material", J Bagh College Dentistry, Vol. 26, No. 1, pp. 32-36, 2014.

[22] Mohamed Ashour Ahmed and Mohamed I. Ebrahim, "Effect of Zirconium Oxide Nano-Fillers Addition on the Flexural Strength, Fracture Toughness, and hardness of heat-Polymerized Acrylic Resin", World Journal of Nano Science and Engineering, Vol. 4, pp. 50-57, 2014.

[23] Hussein Karim Hameed and Hanan Abdul Rahman, "The Effect of Addition Nano Particle ZrO_2 on Some Properties of Autoclave Processed Heat Cure Acrylic Denture Base Material", J Bagh College Dentistry, Vol. 27, No. 1, pp. 32-39, 2015.

[24] Maurizio Avella, Maria Emanuela Errico, and Ezio Martuscelli, "Novel PMMA/$CaCO_3$ Nanocomposites Abrasion Resistant Prepared by an in Situ Polymerization Process", Nano Letters, Vol. 4, No. 1, pp. 213-217, 2001.