Studying of DC Electrical Properties for Gold Sputtered PM-355 Thin Films after Annealing and Ion Beam Irradiation

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Abstract. This article investigated the dc electrical properties of Au / PM-355 thin films synthesized using magnetron sputtering. PM-355 specimens were cleaned by ethanol and then were deposited by gold of different thicknesses equal to 300, 400, 500, 700, 1000, 1300, and 1500 nm. From dc electrical measurements as resistance, resistivity and conductivity were investigated for these thin films due to determine the optimum Au thickness with good characteristic. Then, this optimum thickness was treated by annealing in air up to 140 °C for 30 minutes. Hence, another organic cleaner, chloroform, was used before the deposition of optimum thickness forming thin film. Also, study the effect of nitrogen ion beam extracted from conical anode - disc cathode ion source carried out for 30 minutes with operating conditions on the organic cleaners and annealing Au / PM-355 thin films. Moreover, Shore D hardness tester was used to measure micro-hardness for all thin films compared by blank PM-355. Then, the comparison was done between the thin films through dc electrical conductivity and micro-hardness data at the absence and presence of ion beam irradiation. Finally, the change in surface morphology of thin films due to different treatments can be observed by scanning electron microscope.

Keywords: Thin film, PM-355, gold, annealing, ion beam, sputtering, dc electrical properties, shore hardness test, and scanning electron microscope

1. Introduction
There are different techniques used for thin films deposition such as chemical vapor deposition [1, 2], pulsed laser deposition [3-6], atomic layer deposition [3], electron beam evaporation [7], sol-gel [8, 9], spray pyrolysis [10], ion beam sputtering [11], plasma heating [12-15] and magnetron sputtering [16-22]. The magnetron sputtering is one of the physical vapor deposition processes and well known technique due to the high growth rate fabrication of conductive films. In which the sputtering of a target, cathode, in a dissipate plasma forming thin films [23]. The prepared films are characterized by the higher hardness, better adhesion, and a smoother surface to a substrate compared with the dc traditional method at the similar parameters and the same deposition [24]. Moreover, the electrical and optical properties of thin films can be affected by the deposition parameters, working pressure of reactive gas, distance between target and substrate, substrate heating, plasma source, and the nature of applied power [16-18, 25-28]. Also, the thickness of thin film is a significant parameter that affected on the variation in its physical properties [29]. The ion irradiation plays an important
role for thin films of metal deposited on polymer in the enhancement of their properties [30]. In addition, the annealing effect on the properties of the prepared thin films [31-34].

The main goal of this work is to study the effect of different Au thicknesses deposited on PM-355 through the dc electrical properties and micro-hardness data of Au / PM-355 thin films. Also, study the effect of organic solutions cleaning for PM-355 on the deposition of optimum Au thickness properties' thin film. Moreover, study the effect of annealing on optimum Au thickness thin film to achieve good dc electrical properties and micro-hardness data. In addition, the effect of nitrogen ion beam irradiation on the organic cleaner and annealing for the formed optimum thin film. This is the first step for the catalyst-membrane layer in proton exchange membrane fuel cell (PEMFC) before the deposition of platinum and then immersed in Nafion solution.

2. Theory
The electrical conductivity of a material is an essential factor for studying its electrical properties. One technique to measure the electrical conductivity is the direct current, dc, conductivity measurements. Hence, dc electrical conductivity of a material, $\sigma_{dc}$, is calculated by using the measured electrical resistance, $R$, and specimen dimensions ($d$, $A$) is giving by the relation [35]:

$$\sigma_{dc} = \frac{d}{RA}$$  \hspace{1cm} (1)

Where $d$ and $A$ are the thickness and cross sectional area of the specimen, respectively.

3. Experiment
3.1 Sputtering Technique
It is essentially utilized for the deposition of metal and oxide films by controlling the crystalline structure and surface roughness [36,37]. In this work, the Au / PM-355 thin films are formed by deposition of different thicknesses nanometers gold, nm Au, on PM-355 specimens using DC magnetron sputtering [38]. Firstly, PM-355 specimens were cleaned by ethanol then ready to be deposited by different thicknesses Au forming thin films. From the dc electrical conductivity and micro-hardness measurements, the optimum Au thickness is determined that gives good electrical and mechanical properties. Secondly, the comparison between the effect of annealing and N$_2$ beam extracted from dc conical anode - disc cathode ion source for the optimum thin film at the same exposure time. Thirdly, another PM-355 specimen is cleaned by chloroform and then deposited by optimum thickness Au. The dc electrical conductivity measurements as resistance measured by 2-probes Keithley electrometer (model 617), $R$, resistivity, $\rho$, and conductivity, $\sigma_{dc}$, can be plotted for blank and different Au/PM-355 thin films.

3.2 Micro-hardness Measurement
The most compatible characteristics of elastic material's mechanical properties is the hardness that defined as the capability of the composite material to resist abrasion depending on the permanent size of indentation measured by a hard ball under a spring load [39]. Shore hardness is an international standard instrument which is utilized for the estimation of the indentation plastics and rubber with types A and D durometers for softer and harder materials, respectively [39, 40]. The hardness indentation depends on the material's modulus of elasticity and the viscoelastic properties and inversely regarding to the penetration [41]. Gold is the most excellent noble metal and the lightest reinforcing factor used to increment the hardness of polymer.

Finally, the morphologies of the PM-355 blank and thin films treatments were shown by JFC 1500 scanning electron microscope with Energy Dispersive X-Ray Spectrometer.
4. Fabrication of Thin Films

4.1 Materials
Polycarbonate allyl diglycol, PM-355 with chemical composition C_{12}H_{18}O_{7}, is partially crystalline with predominant amorphous phase and purchased from Sigma-Aldrich, Germany. All specimens with dimensions 1.5cm x 1cm and thickness equal to 1mm, cleaned by 99.99% ethanol before Au deposition. Also, one specimen is cleaned by chloroform contains 1 to 2% v/v ethanol as a preservative, from AnalaR®- BDH Limited Poole England. The ethanol specimens were deposited with different Au thicknesses to determine the optimum thickness.

4.2 Au / PM-355 Thin Films Preparation
Firstly, the ethanol PM-355 specimens were deposited by different Au thicknesses equal to 300, 400, 500, 700, 1000, 1300, and 1500 nm using magnetron sputtering. Secondly, one sample was cleaned by chloroform and then deposited by the optimum Au thickness.

4.3 Characteristics of Annealing and Ion Beam Irradiation
The annealing was carried out in air using a thermostat oven at 140°C for 30min. with rate 5°C / min. for ethanol optimum Au thickness thin film. Also, another thin film is exposed to time equal to 30 minutes by the N_{2}^{+} ion beam that extracted from dc conical anode - disc cathode ion source. This ion source operating conditions are working pressure, discharge current, discharge voltage and output ion current equal to 3 x 10^{-4} mmHg, 0.5 mA, 1200 V, 165 μA respectively. Moreover, study the comparison of surface micro-hardness, dc electrical conductivity and surface morphologies for PM-355 blank and Au / PM-355 thin films at the absence and presence of nitrogen ion beam.

5. Results and Discussions

5.1 Without Ion Beam Irradiation

5.1.1 DC Electrical Conductivity. Resistance, resistivity and dc conductivity were measured for different Au thicknesses thin films using Keithley electrometer. Figures (1-3) show the effect of different Au nm thicknesses forming Au / PM-355 ethanol thin films on the resistance, resistivity and dc conductivity with respect to blank, respectively. Figure (1) illustrates the resistance of thin films are in range 0.74 - 0.9579 GΩ compared with 3.3 GΩ for blank PM-355. It is clear that 500 nm Au / PM-355 thin film has the maximum resistance equal to 0.9579 GΩ. Figure (2) indicates that the 500nm Au thickness gives the lowest resistivity equal to 0.92 x 10^{-4} Ωm comparison with 1.1 x 10^{-4} Ωm for blank PM-355. Also, the 500nm Au thickness thin film has the maximum dc conductivity equal to 1.084 x 10^{4} S/m compared with 0.9047 x 10^{4} S/m for blank PM-355, see fig. (3). From these figures, it is deduced that the optimum Au thickness is at 500 nm deposited on PM-355 to form good electrical thin film property. Hence, study the effect of annealing without exposure of N_{2}^{+} beam on this optimum Au thickness forming Au / PM-355 thin film. Moreover, one PM-355 specimen cleaned by chloroform and then deposited by Au forming thin film.

Therefore, figure (4) shows the dc conductivity comparison between PM-355 blank, 500nm Au thin films treated with ethanol, chloroform and annealing. It is clear that the conductivity order is 500nm annealing > 500nm > blank > 500nm chloroform.
5.1.2 Surface Micro-hardness. Using Shore D tester, the micro-hardness is plotted versus different Au nm thicknesses thin films and blank PM-355 as shown in fig. (5). It is seen that the maximum micro-hardness value is for the optimum thin film and equal to 86.3 Shore D compared with 84.67 Shore D for blank. Moreover, the other thin films have micro-hardness in range 79.33 - 85.83 Shore D. Therefore, the micro-hardness comparison between PM-355 blank, 500nm Au thin films treated with ethanol, chloroform and annealing as shown in fig. (6) It is clear that the order of micro-hardness is 500nm annealing > 500nm > blank > 500nm chloroform.
5.2 With Ion Beam Irradiation

5.2.1 DC Electrical Conductivity. Figures (7-9) show the effect of N\textsubscript{2}+ beam for blank PM-355 and different Au thicknesses thin films on resistance, resistivity and dc conductivity, respectively. From figure (7), the resistance of optimum thickness and the other thin films are equal to 0.9629 G\(\Omega\) and in range 1.2013 - 1.329 G\(\Omega\) respectively compared with 1.4473 G\(\Omega\) for blank. It is clear that 500nm Au / PM-355 thin film has the minimum resistance under the effect of ion beam. Figure (8) indicates that the 500nm thickness has the lowest resistivity equal to 0.96 \(\times 10^{-4}\ \Omega \text{m}\) and the other thin films ranging 1.9 \(\times 10^{-4} - 2.6 \times 10^{-4}\ \Omega \text{m}\) compared with 0.48 \(\times 10^{-4}\ \Omega \text{m}\) for blank.

![Figure 5. Micro-hardness versus blank PM-355 and different Au thicknesses thin films.](image)

![Figure 6. Micro-hardness versus blank PM-355 and different Au/PM-355 thin films treatments.](image)

![Figure 7. Resistance versus blank PM-355 and different Au thicknesses thin films under the effect of ion beam.](image)

![Figure 8. Resistivity versus blank PM-355 and different Au thicknesses thin films under the effect of ion beam.](image)
Therefore, from fig. (9) the 500 nm thickness thin film has maximum dc conductivity equal to $1.0385 \times 10^4$ S/m and the other thin films ranging $0.3918 \times 10^4 - 0.5228 \times 10^4$ compared with $2.0629 \times 10^4$ S/m for blank. Then, the dc conductivity comparison between PM-355 blank, 500nm Au thin films treated with ethanol, chloroform and annealing after the effect of ion beam as shown in fig. (10). It is clear that the conductivity order is blank $>$ 500nm $>$ 500nm annealing $>$ 500nm chloroform.

5.2.2 **Surface Micro-hardness.** The micro-hardness, Shore D, is plotted versus different Au nm thicknesses thin films and blank PM-355 after the exposure of N$_2^+$ beam as shown in fig. (11). It is seen that the 500nm Au optimum thickness has the minimum micro-hardness value and equal to 57.67 Shore D compared with 75.67 Shore D for blank. Moreover, the other thin films have micro-hardness in range 60.67 - 73.33 Shore D. Then, the micro-hardness comparison between PM-355 blank, 500nm Au thin films treated with ethanol, chloroform and annealing under the effect of ion beam as shown in fig. (12). It is noticed that the order of micro-hardness is blank $>$ 500nm chloroform $>$ 500nm annealing $>$ 500nm.
5.3 Comparison between without and with Ion Beam Irradiation

5.3.1 DC Electrical Conductivity. Table (1) shows the effect of ion beam irradiation on dc conductivity values for PM-355 blank and different thin film treatments. It is noticed that the resistance and resistivity of 500nm Au / PM-355 thin film is decreased corresponding to PM-355 blank without ion beam effect reaching 0.29 GΩ and 0.8364 Ω·m with respect to PM-355 blank values. The effect of ion beam on the resistance and resistivity values of 500 nm Au thin film are increased than without ion beam irradiation reaching 0.665 GΩ and 2 Ω·m with respect to PM-355 blank values, respectively. Without the effect of ion beam, dc conductivity values for ethanol 500 nm Au and treated by annealing thin films are increased than PM-355 blank value and with reverse effect for chloroform thin film. But dc conductivity for ethanol, chloroform 500 nm Au and annealing thin films are decreased than PM-355 blank value under the effect of ion beam.

Table (1) the effect of ion beam on dc electrical properties for 500 nm Au / PM-355 thin films and their treatments compared with PM-355 blank.

|                        | Without ion beam | With ion beam |
|------------------------|------------------|---------------|
| **Resistance (GΩ)**    |                  |               |
| 500nm Au thin film     | 0.29 blank value | 500nm Au thin film | 0.665 blank value |
| 500nm Au thin film     | 0.8364 blank value | 500nm Au thin film | 2 blank value |
| **Resistivity (Ω·m)**  |                  |               |
| ethanol 500nm Au thin film | 1.198 blank value | 500nm Au thin film | 0.534 blank value |
| 500nm Au annealing thin film | 1.217 blank value | 500nm Au annealing thin film | 0.38 blank value |
| chloroform 500nm Au thin film | 0.732 blank value | chloroform 500nm Au thin film | 0.3363 blank value |
| chloroform 500nm Au thin film | 0.61 ethanol 500nm Au thin film | chloroform 500nm Au thin film | 0.67 ethanol 500nm Au thin film |
| **Conductivity (S/m)** |                  |               |
| 500nm Au annealing thin film | 1.015 ethanol 500nm Au thin film | 500nm Au annealing thin film | 0.76 ethanol 500nm Au thin film |

Figure (13) shows the conductivity comparison between PM-355 blank, thin films treatments at the absence and presence effect of ion beam. It is found that the dc conductivity values of blank and 500nm chloroform thin film is increased by the irradiation of ion beam. But, the conductivity values of 500nm Au / PM-355 thin film and annealing is decreased under the effect of ion beam. Then, the effect of ion beam on 500nm Au chloroform thin film and 500 nm Au annealing ethanol thin film have the lowest dc electrical conductivity. Moreover, it is a large compatible with ac electrical conductivity values of these thin films at low frequencies ranging KHz reported in [38]. This is the first step before Pt addition that used to design the membrane - catalyst layer (Au / Pt) for proton exchange membrane fuel cell (PEMFC) before impregnated in Nafion solution.
Figure 13. DC conductivity versus different Au/PM-355 thin films treatments and PM-355 blank with and without ion beam effect.

5.3.2 Surface Micro-hardness. Table (2) shows the micro-hardness comparison between PM-355 blank and different thin films treatments at the absence and presence of ion beam. It is noticed that the effect of ion beam on thin films treatments decreased their micro-hardness values than without ion irradiation corresponding to PM-355 blank. In case of without ion beam effect, the micro-hardness values for 500nm ethanol, annealing and chloroform thin films are nearly equal to PM-355 blank value. But these thin films are decreased with respect to blank micro-hardness value under the effect of ion beam.

Table (2) the effect of ion beam on micro-hardness for 500 nm Au / PM-355 thin films and their treatments compared with PM-355 blank.

| Without ion beam | With ion beam |
|------------------|---------------|
| 500nm Au thin film | 1.02 blank value | 500nm Au thin film | 0.76 blank value |
| 500nm Au annealing thin film | 1.03 blank value | 500nm Au annealing thin film | 0.77 blank value |
| chloroform 500nm Au thin film | 0.988 blank value | chloroform 500nm Au thin film | 0.846 blank value |
| chloroform 500nm ethanol 500nm Au thin film | 0.61ethanol 500nm | chloroform 500nm ethanol 500nm Au thin film | 0.67ethanol 500nm |

Figure (14) shows the comparison of micro-hardness, Shore D, between PM-355 blank, thin films treatments at the absence and presence of ion beam irradiation. It is found that the micro-hardness values for PM-355 blank and thin films treatments are decreased under the effect of ion beam irradiation. Also, the effect of ion beam on 500 nm Au chloroform thin film and 500 nm Au annealing ethanol thin film have the highest micro-hardness values between the ion irradiated thin films.
5.3.3 Scanning Electron Microscope Morphology. Scanning electron micrographs of PM-355 blank, chloroform, annealing, and ion beam for 500 nm Au / PM-355 thin films are shown in fig. 15(a-d), respectively. Figure 15(a) shows the typical surface topography of PM-355 blank that has smooth surface. Then, due to the deposition of Au on PM-355 cleaned before by chloroform as shown in fig. 15(b).

Moreover, figure 15(c) illustrates the effect of annealing on Au layer deposited on PM-355 cleaned by ethanol. It shows a decrease in the number of dislocations by redistribution of atoms leading to a change in the hardness. Finally, figure 15(d) illustrates the effect of nitrogen ion beam on Au layer that placed in the interstitial sites and then decreased the hardness.

**Figure 14.** Micro-hardness versus different Au/PM-355 thin films treatments and PM-355 blank with and without ion beam effect

**Figure 15.** (a-d) Scanning electron microscope morphologies for PM-355 blank, chloroform, annealing, and ion beam for 500nm Au / PM-355 thin films respectively
6. Conclusion
The present work investigated how the Au thickness deposited on PM-355 polymer and annealing the optimum thickness then irradiated by nitrogen ion beam effected on the dc electrical properties micro-hardness measurements of the formed thin films. It is deduced that the resistance of blank PM-355 is decreased after ion beam irradiation to 43.86%. But, there is no effect of ion beam irradiation on the resistance value of optimum Au thickness thin film. The effect of ion beam on the annealed and chloroform thin films have an increase in the resistance value by 140.42% and 152.8% respectively. Also, the increasing in conductivity for blank PM-355, 500 nm Au chloroform, ethanol and annealed thin films under the effect of ion beam irradiation by 228%, 104.7%, 95.8% and 71.2%, respectively. In addition, the chloroform Au / PM-355 thin film has the lowest dc conductivity value in case of with and without ion beam irradiation. Also, the annealing thin film has the highest conductivity value without ion beam irradiation and in a second order after chloroform thin film in case of ion beam irradiation.

There is a decrease in micro-hardness values for PM-355 blank, 500 nm Au chloroform, ethanol and annealed thin films under the effect of ion beam to 89.4%, 76.5%, 66.8% and 66.4% respectively. In general, the nitrogen ion beam irradiation decreased the micro-hardness of PM-355 blank and different thin films treatments. Moreover, the effect of ion beam on chloroform thin film move the micro-hardness value from the lowest order to the highest with comparison with blank PM-355 and other thin films. Also, there is a decrease in the micro-hardness value for annealed thin film under the effect of ion beam. Then, it is concluded that the annealing and ion beam irradiation plays an important effect on electrical properties and micro-hardness of thin films treatments. The chloroform thin film properties is lower than the annealing effect on the thin film in the two cases with and without ion beam irradiation. The ion beam irradiation effect on the annealed thin film has a good properties for the Au / PM-355 thin film.

Finally, it is necessary to deposit Au on PM-355 cleaned by chloroform before platinum deposition forming membrane - catalyst layer used in PEMFC then impregnated in Nafion solution. Hence, the best way to design this layer is to deposit Au on the polymer cleaned by ethanol followed by annealing or chloroform only. That is a large compatible between the dc and ac electrical properties that presented in another article for the different thin films treatments.

7. References
[1] Martín A, Espinós J P, Justo A, Holgado J P, Yubero F and González-Elipe A R 2002 Surf. Coat. Technol. 151–152 289
[2] Nystrom M J, Wessels B W, Lin W P, Wong G K, Neumayer D A and Marks T J 1995 Appl. Phys. Lett. 66 1726
[3] Meyer J, Görrn P, Hamwi S, Johannes H H, Riedl T and Kowalsky W 2008 Appl. Phys. Lett. 93 073308
[4] Schwyn Thöny S, Youden K E, Harris Jr J S and Hesselink L 1994 Appl. Phys. Lett. 65 2018
[5] Shen Z R, Ye H, Mak C L, Yum T Y and Wong K H 2007 Thin Solid Films 515 3475
[6] Gupta S, Paliwal A, Guo R, Bhalla A S, Gupta V and Tomar M 2018 Opt. Mater. 85 26
[7] Sahu D R, Lin S Y and Huang J L 2007 Sol. Energy Mater. Sol. Cells 91 851
[8] Lin J P and Wu J M 2008 Appl. Phys. Lett. 92 134103
[9] Kraya R, Baskar J, Arceo A, Katz H E and Thakor N 2018 Thin Solid Films 664 41
[10] Pandey R, Yuldashev S, Nguyen H D, Jeon H C and Kang T W 2012 Curr. Appl. Phys. 12 S56
[11] Valentini A, Quaranta F, Penza M and Rizzi F R 1993 J. Appl. Phys. 73 1143
[12] Duncan M A 2012 Rev. Sci. Instrum. 83 041101
[13] Slotte M and Zevenhoven R 2017 R. Energies 10 1605
[14] Rajput N 2015 Int. J. Adv. Eng. Technol. 7 1806
[15] Koten M A and Voeller S A 2016 J. Appl. Phys. 119 114306
[16] Sahu B B, Jin S B, Xiang P J, Kim J B and Han J G 2018 J. Appl. Phys. 123 205107
[17] Hamasha M M, Dhakal T P, Vasekar P, Alzoubi K, Lu S, Vanhart D and Westgate C R 2013 Sol. Energy 89 54
[18] Yen W T, Lin Y C and Ke J H 2010 Appl. Surf. Sci. 257 960
[19] Zhu Y Y, Xiao R F and Wong G K L 1997 J. Appl. Phys. 82 4908
[20] Kumar G R, Gokulraj S and Yathavan S 2016 Mater. Today Proc. 3 3982
[21] Shirokov V B, Pavlenko A V, Stryukov D V and Revinskii Y V 2018 Phys. Solid State 60 1005
[22] Raj S G, Mathivanan V, Kumar G R, Yathavan S and Mohan R 2016 AIP Conf. Proc, AIP Publishing 1728 20585
[23] May G S and Sze S M Fundamentals of Semiconductor Fabrication 2004 (Wiley, New York)
[24] Engwalla A M, Shin S J, Bae J and Wang Y M 2019 Surface & Coatings Technology 363 191
[25] Guillén C and Herrero J 2010 Vacuum 84 924
[26] Kim D K and Kim H B 2011 J. Alloys. Compd. 509 421
[27] Nishimotoa Naoki, Fujihara Junko, Yoshino Katsumi 2017 Applied Surface Science 409 375
[28] Cristea D, Crisan A, Cretu N, Borges J, Lopes C, Cunha L, Ion V, Dinescu M, Barradas N P, Alves E, Apreuteseii M and Munteanu D 2015 Applied Surface Science 354 298
[29] Singh D, Kumar S, Thangaraj R and 2013 Physica B: Condensed Matter 408 119
[30] Imran M, Ahmad R, Afzal N and Rafique M 2019 Vacuum 165 72
[31] Miliutina E, Kalachyova Y, Burtev V, Postnikov P, Elasnikov R, Švorčík V and Lyutakov O 2019 Nano-Structures & Nano-Objects 17 77
[32] Shang X, Yu H, Choi W, Lee E K and Oh J H 2016 Organic Electronics 30 207
[33] Aruna P and Joseph C M 2017 Materials Science in Semiconductor Processing 61 39
[34] Jůřík P, Slepíčka P, Kolská Z and Švorčík V 2016 Materials Letters 165 33
[35] Elliott S R 1987 Adv. Phys. 36 135
[36] Barranco A, Borras A, Elipe A R G and Palmero A 2016 Progress Mater. Sci. 76 59
[37] Macleod H A 2013 from Materials to Applications Woodhead Publishing Series in Electronic and Optical Materials 3
[38] Radwan S I, Rashad A M, Tantawy H R and Abdel Samad S, Effect of Annealing and Ion Beam Irradiation on AC Electrical Properties for Gold Sputtered PM-355, under review
[39] Morgans R, Lackovic S and Cobbold P 1999 American Chemical Society
[40] Plastics and ebonite—Determination of indentation hardness by means of a durometer (Shore hardness), ISO 868-1985
[41] Mohamed M I and Aggag G A 2003 Measurement 33 251
[42] Ozsoy I, Demirkol A, Mimaroglu A, Unal H and Demir Z 2015 J. J of Mech. Eng. 6110 601