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APPENDICES

APPENDIX A

CALIBRATION CURVE FOR MEASURING THICKNESS OF SILVER LAYER BY PLOT TRANSMITTANCE VERSUS THICKNESS OF SIVER LAYER BY TEM

In this work, the thickness of silver layer was measured from the transmittance of light through the film. The calibration curve was constructed from the absorbance data versus the actual thickness of the silver layer on the film measures from the cross-section TEM images.

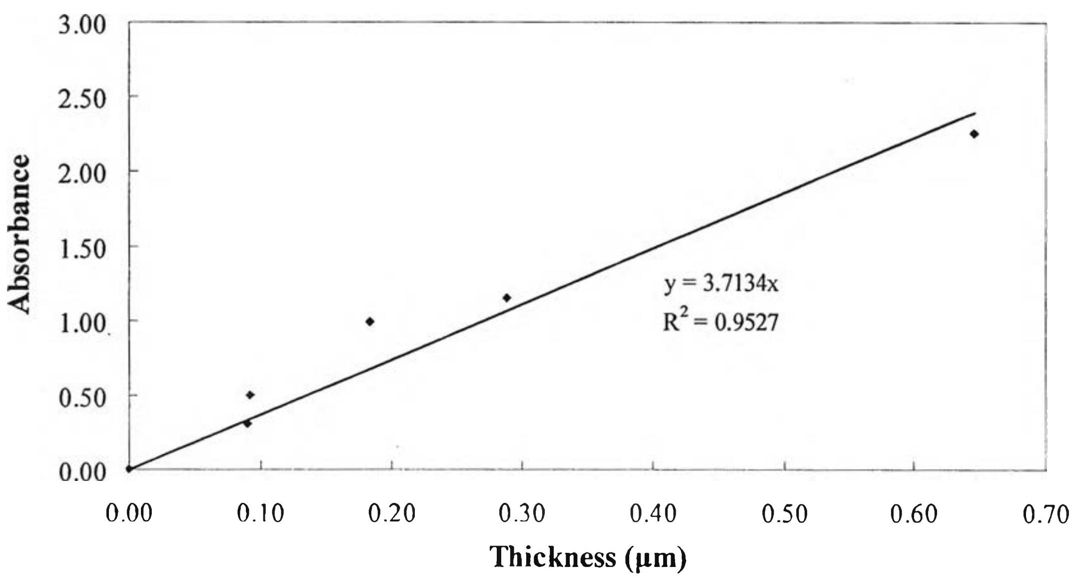

Figure A.1 Calibration curve for measuring thickness of silver layer.

APPENDIX B

SIZE DISTRIBUTION OF SILVER NANOPARTICLES ON THE SURFACE OF POLYIMIDE FILM PREPARED BY ULTRAVIOLET IRRADIATOIN

Table B.1 Size distribution of silver nanoparticles on polyimide prepared by conventional irradiation.

Number of particle	Particle size (µm)	Number of particle	Particle size (µm)
1	0.02	65	0.04
2	0.03	66	0.04
3	0.03	67	0.04
4	0.03	68	0.04
5	0.03	69	0.04
6	0.03	70	0.04
7	0.03	71	0.04
8	0.03	72	0.04
9	0.03	73	0.04
10	0.03	74	0.04
11	0.03	75	0.04
12	0.03	76	0.04
13	0.03	77	0.04
14	0.03	78	0.04
15	0.03	79	0.04
16	0.03	80	0.04
17	0.03	81	0.04
18	0.03	82	0.05
19	0.03	83	0.05

Number of particle	Particle size (μm)	Number of particle	Particle size (μm)
20	0.03	84	0.05
21	0.03	85	0.05
22	0.04	86	0.05
23	0.04	87	0.05
24	0.04	88	0.05
25	0.04	89	0.05
26	0.04	90	0.05
27	0.04	93	0.05
28	0.04	94	0.05
29	0.04	95	0.05
30	0.04	96	0.05
31	0.04	97	0.05
32	0.04	98	0.05
33	0.04	99	0.05
34	0.04	100	0.05
35	0.04	101	0.05
36	0.04	102	0.05
37	0.04	103	0.05
38	0.04	104	0.05
39	0.04	106	0.05
40	0.04	107	0.05
41	0.04	108	0.05
42	0.04	109	0.05
43	0.04	110	0.05
44	0.04	111	0.05
45	0.04	112	0.05
46	0.04	113	0.05
47	0.04	114	0.05
48	0.04	115	0.05
49	0.04	116	0.05

Number of particle	Particle size (μm)	Number of particle	Particle size (μm)
50	0.04	117	0.06
51	0.04	118	0.06
52	0.04	119	0.06
53	0.04	120	0.06
54	0.04	121	0.06
55	0.04	122	0.06
56	0.04	123	0.06
57	0.04	124	0.06
58	0.04	125	0.06
59	0.04	126	0.06
60	0.04	127	0.06
61	0.04	128	0.06
62	0.04	129	0.06
63	0.04	130	0.07
64	0.04		

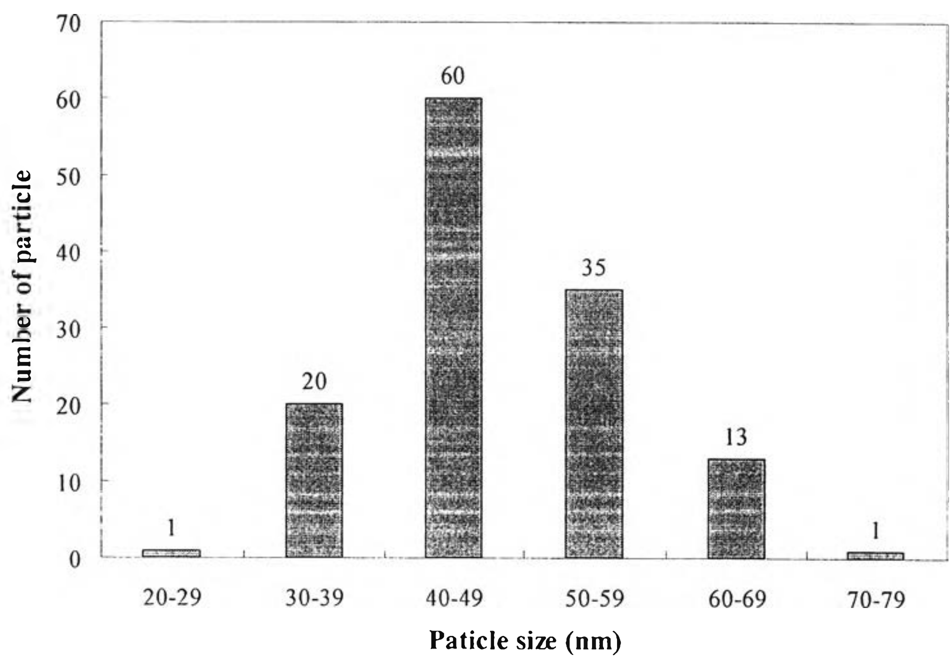

Figure B.1 Size distributions of silver nanoparticles on polyimide film surface prepared by ultraviolet irradiation.

Table B.2 Size distributions of silver nanoparticles on polyimide film surface prepared by water droplet assisted irradiation.

Number of particle	Particle size (nm)	Number of particle	Particle size (nm)
1	28.27	65	50.34
2	28.75	66	50.39
3	30.60	67	50.39
4	30.83	68	50.56
5	32.31	69	50.74
6	32.97	70	50.74
7	33.71	71	51.06
8	33.71	72	51.15
9	34.51	73	51.94
10	35.21	74	52.03
11	35.60	75	52.04
12	35.64	76	52.13
13	35.70	77	52.45
14	36.72	78	52.47
15	36.76	79	52.67
16	37.03	80	52.68
17	37.24	81	52.98
18	38.73	82	52.98
19	39.04	83	53.01
20	39.16	84	53.03
21	39.26	85	53.07
22	39.34	86	53.54
23	39.57	87	54.39
24	40.21	88	55.00
25	40.64	89	55.31
26	40.90	90	56.30
27	41.29	93	56.54

Number of particle	Particle size (nm)	Number of particle	Particle size (nm)
28	41.45	94	56.65
29	41.48	95	56.67
30	41.65	96	57.12
31	41.65	97	57.33
32	41.75	98	57.47
33	41.92	99	57.76
34	42.23	100	57.88
35	42.44	101	58.34
36	43.20	102	58.51
37	43.62	103	58.98
38	43.88	104	59.10
39	44.15	106	61.15
40	44.20	107	61.42
41	44.25	108	62.20
42	44.48	109	65.78
43	44.55	110	66.02
44	44.55	111	66.38
45	44.95	112	66.82
46	45.39	113	67.42
47	45.75	114	67.45
48	46.12	115	67.67
49	46.78	116	67.72
50	46.94	117	67.88
51	46.96	118	70.33
52	46.99	119	70.94
53	47.11	120	71.29
54	47.55	121	71.53
55	47.82	122	71.77
56	48.01	123	73.73
57	48.77	124	74.30

Number of particle	Particle size (nm)	Number of particle	Particle size (nm)
58	48.77	125	75.03
59	48.78	126	78.36
60	49.19	127	80.15
61	49.40	128	81.62
62	49.57	129	84.52
63	49.79	130	84.93
64	50.27		

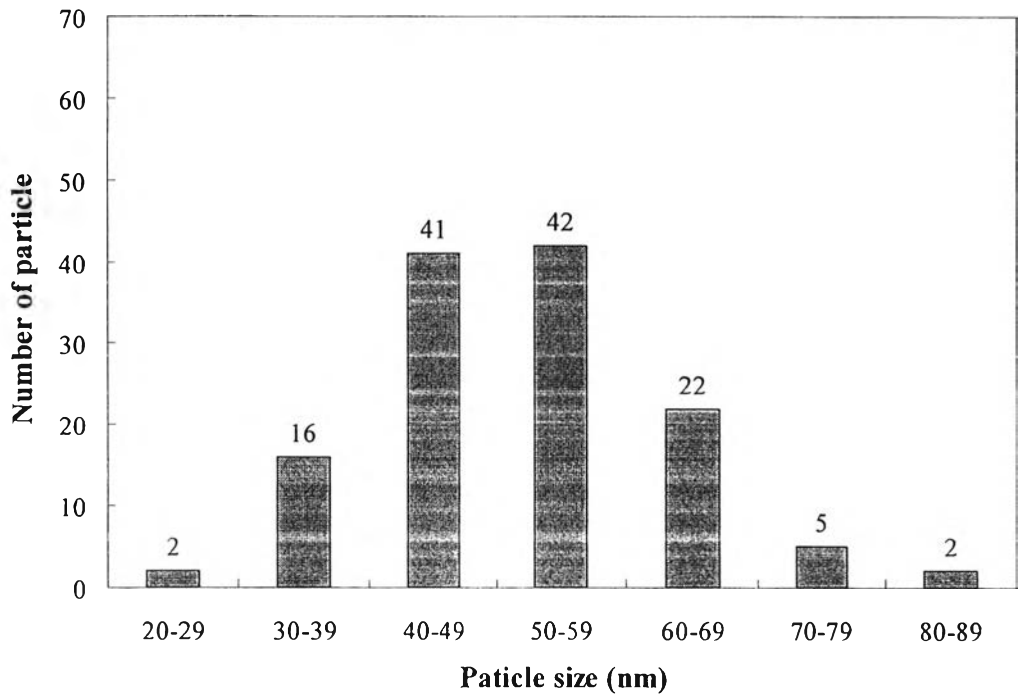

Figure B.2 Size distributions of silver nanoparticles on polyimide film surface prepared by water droplet assisted irradiation.

Table B.3 Size distributions of silver nanoparticles on polyimide film surface prepared by water film assisted irradiation.

Number of particle	Particle size (nm)	Number of particle	Particle size (nm)
1	26.54	65	38.16
2	28.02	66	38.22
3	28.67	67	38.56
4	28.67	68	38.77
5	28.86	69	38.87
6	29	70	38.88
7	29.38	71	38.93
8	29.74	72	39.09
9	29.95	73	39.13
10	30.15	74	39.45
11	30.64	75	39.54
12	31.05	76	39.59
13	31.08	77	39.63
14	31.42	78	39.96
15	31.49	79	39.96
16	31.56	80	40.08
17	31.88	81	40.35
18	32.05	82	40.35
19	32.23	83	40.51
20	32.25	84	40.69
21	32.58	85	40.75
22	33.01	86	41.09
23	33.01	87	41.26
24	33.15	88	41.29
25	33.24	89	41.29
26	33.28	90	41.39
27	33.45	93	41.39
28	34.01	94	41.49

Number of particle	Particle size (nm)	Number of particle	Particle size (nm)
29	34.17	95	41.52
30	34.24	96	41.64
31	34.48	97	41.89
32	34.56	98	42.03
33	34.78	99	42.06
34	34.95	100	42.23
35	35.19	101	42.29
36	35.39	102	42.29
37	35.43	103	42.61
38	35.54	104	42.74
39	35.57	106	42.92
40	35.62	107	43.46
41	35.72	108	43.58
42	35.72	109	43.77
43	35.8	110	43.82
44	35.81	111	43.84
45	35.87	112	43.87
46	35.93	113	43.93
47	36.09	114	44.15
48	36.16	115	44.15
49	36.22	116	44.15
50	36.23	117	44.21
51	36.23	118	44.35
52	36.5	119	44.51
53	36.76	120	44.9
54	36.78	121	44.93
55	37.16	122	45.19
56	37.24	123	46.48
57	37.27	124	47.43
58	37.36	125	48.72

Number of particle	Particle size	Number of particle	Particle size
59	37.52	126	48.81
60	37.65	127	49.09
61	37.68	128	50.02
62	38.00	129	50.65
63	38.10	130	50.89
64	38.14		

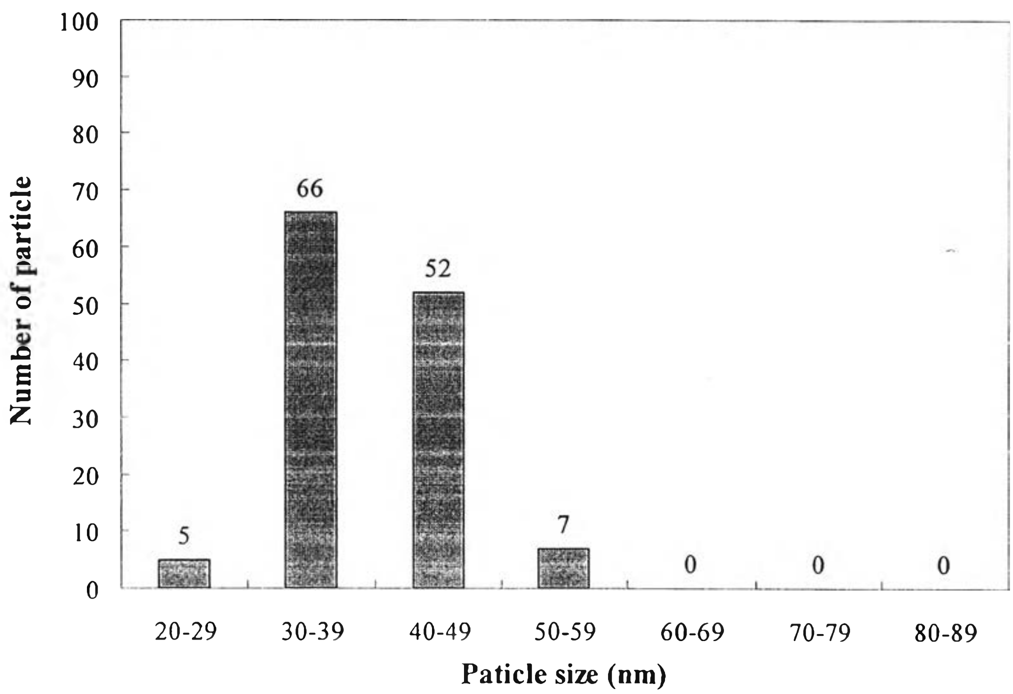

Figure B.3 Size distributions of silver nanoparticles on polyimide film surface prepared by water film assisted irradiation.

Table B.4 Size distributions of silver nanoparticles on polyimide film surface prepared by cycle irradiation (immersed in $AgNO_3$).

Number of particle	Particle size (nm)	Number of particle	Particle size (nm)
1	0.03	65	0.05
2	0.03	66	0.05
3	0.03	67	0.05
4	0.03	68	0.05
5	0.03	69	0.05
6	0.03	70	0.05
7	0.03	71	0.05
8	0.03	72	0.05
9	0.03	73	0.05
10	0.03	74	0.05
11	0.04	75	0.05
12	0.04	76	0.05
13	0.04	77	0.05
14	0.04	78	0.05
15	0.04	79	0.05
16	0.04	80	0.05
17	0.04	81	0.05
18	0.04	82	0.05
19	0.04	83	0.05
20	0.04	84	0.05
21	0.04	85	0.05
22	0.04	86	0.05
23	0.04	87	0.05
24	0.04	88	0.05
25	0.04	89	0.05
26	0.04	90	0.05
27	0.04	93	0.05
28	0.04	94	0.05

Number of particle	Particle size (nm)	Number of particle	Particle size (nm)
29	0.04	95	0.05
30	0.04	96	0.05
31	0.04	97	0.05
32	0.04	98	0.05
33	0.04	99	0.05
34	0.04	100	0.05
35	0.04	101	0.05
36	0.04	102	0.05
37	0.04	103	0.05
38	0.04	104	0.05
39	0.04	106	0.05
40	0.04	107	0.05
41	0.04	108	0.05
42	0.04	109	0.05
43	0.04	110	0.05
44	0.04	111	0.05
45	0.04	112	0.05
46	0.04	113	0.05
47	0.04	114	0.05
48	0.04	115	0.05
49	0.04	116	0.06
50	0.04	117	0.06
51	0.04	118	0.06
52	0.04	119	0.06
53	0.04	120	0.06
54	0.04	121	0.06
55	0.04	122	0.06
56	0.04	123	0.06
57	0.04	124	0.06
58	0.05	125	0.06

Number of particle	Particle size (nm)	Number of particle	Particle size (nm)
59	0.05	126	0.06
60	0.05	127	0.06
61	0.05	128	0.06
62	0.05	129	0.06
63	0.05	130	0.06
64	0.05		

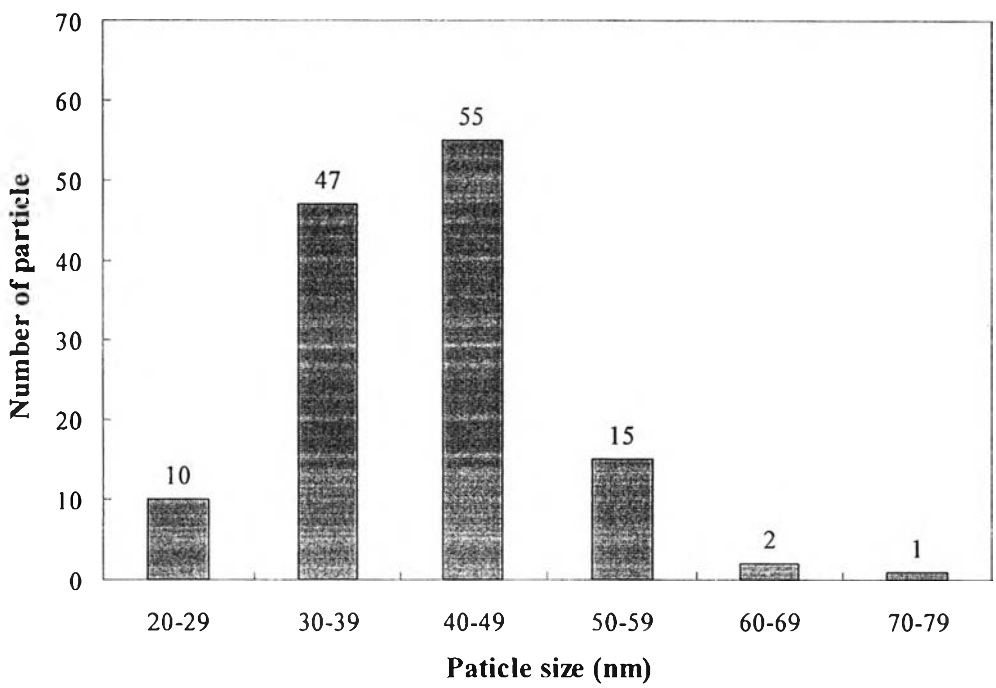

Figure B.4 Size distributions of silver nanoparticles on polyimide film surface prepared by cycle irradiation (immersed in $AgNO_3$).

Table B.5 Size distributions of silver nanoparticles on polyimide film surface prepared by cycle irradiation (immersed KOH).

Number of particle	Particle size (nm)	Number of particle	Particle size (nm)
1	0.03	65	0.04
2	0.03	66	0.04
3	0.03	67	0.04
4	0.03	68	0.04
5	0.03	69	0.04
6	0.03	70	0.04
7	0.03	71	0.04
8	0.03	72	0.04
9	0.03	73	0.04
10	0.03	74	0.04
11	0.03	75	0.04
12	0.03	76	0.04
13	0.03	77	0.04
14	0.03	78	0.04
15	0.03	79	0.04
16	0.03	80	0.04
17	0.03	81	0.04
18	0.03	82	0.04
19	0.03	83	0.04
20	0.03	84	0.04
21	0.03	85	0.04
22	0.03	86	0.04
23	0.03	87	0.04
24	0.03	88	0.04
25	0.03	89	0.04
26	0.03	90	0.04
27	0.03	93	0.04

Number of particle	Particle size (nm)	Number of particle	Particle size (nm)
28	0.03	94	0.04
29	0.03	95	0.04
30	0.04	96	0.04
31	0.04	97	0.04
32	0.04	98	0.04
33	0.04	99	0.04
34	0.04	100	0.04
35	0.04	101	0.04
36	0.04	102	0.04
37	0.04	103	0.04
38	0.04	104	0.04
39	0.04	106	0.04
40	0.04	107	0.04
41	0.04	108	0.04
42	0.04	109	0.04
43	0.04	110	0.04
44	0.04	111	0.04
45	0.04	112	0.05
46	0.04	113	0.05
47	0.04	114	0.05
48	0.04	115	0.05
49	0.04	116	0.05
50	0.04	117	0.05
51	0.04	118	0.05
52	0.04	119	0.05
53	0.04	120	0.05
54	0.04	121	0.05
55	0.04	122	0.05
56	0.04	123	0.05
57	0.04	124	0.05

Number of particle	Particle size (nm)	Number of particle	Particle size (nm)
58	0.04	125	0.05
59	0.04	126	0.05
60	0.04	127	0.05
61	0.04	128	0.05
62	0.04	129	0.05
63	0.04	130	0.05
64	0.04		

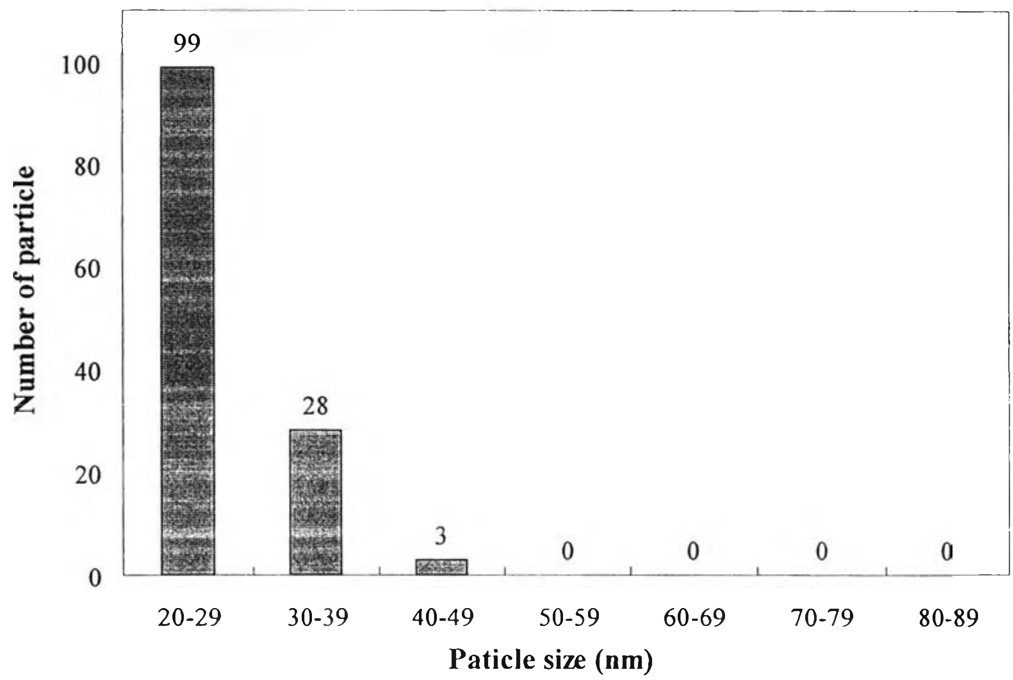

Figure B.5 Size distributions of silver nanoparticles on polyimide film surface prepared by cycle irradiation (immersed KOH).

APPENDIX C

ABSORBSNCE OF POLYIMIDE FILM

Table C.1 Absorbance of polyimide film modified with KOH at 50 °C for 1 minute and reduced by conventional irradiation.

Irradiation time (minutes)	Replicate		
	i	ii	iii
30	0.3796	0.3301	0.3458
60	0.3193	0.3854	0.3572
90	0.3922	0.3716	0.3574
120	0.5236	0.5473	0.5403
150	0.5757	0.5930	0.5208
180	0.6236	0.6330	0.5884
360	0.7739	0.7694	0.7723
900	0.9403	0.9883	1.0314

Table C.2 Absorbance of polyimide film modified with KOH at 50 °C for 1 minute and reduced by water droplet assisted irradiation for 3 hours.

Assisted water after irradiation for (minutes)	Replicate		
	i	ii	iii
30	0.961526	1.04799	1.007882
60	1.043906	0.967272	1.037258
90	1.210979	0.996694	1.154119

Table C.3 Absorbance of polyimide film modified with KOH at 50 °C for 1 minute and reduced by water film assisted irradiation for 3 hours.

Assisted water after irradiation for (minutes)	Replicate		
	i	ii	iii
30	0.713296	0.729931	0.783675
60	0.741149	0.724684	0.889094
90	0.908587	0.889094	0.824892

Table C.4 Absorbance of polyimide film modified with KOH at 50 °C for 1 minute and reduced by cycle irradiation for 3 hours.

Immersed KOH after irradiation for (minutes)	Replicate		
	i	ii	iii
30	2.623364	1.965322	2.294343
60	1.934347	2.117519	2.025933
90	1.633521	1.408117	1.30766

APPENDIX D

LIST OF PLUBLICATION

Patinan Chalephol and Varong Pavarajarn, "Growth of Thin Silver Film on Polyimide", the 5th Thailand Materials Science and Technology Conference, Bangkok, September 16-19, 2008.

Growth of Thin Silver Film on Polyimide

Patinan Chalephol and Varong Pavarajarn*
Department of Chemical Engineering, Chulalongkorn University, Bangkok 10330
*Corresponding author: Phone 0-2218-6890, Fax 0-2218-6877, E-mail: fchvpv@eng.chula.ac.th

Abstract

This work investigates the effect of surface hydrolysis of polyimide (PI) on the effectiveness of thin silver film coating. PI surface was modified by 3 different concentration of KOH solution for various period of time, before underwent the coating of silver via the process of silver ions reduction. The statistical method was employed to determine main effect for growth of silver layer. FT-IR spectroscopy was also use for determine change of functional group in PI film

1. Introduction

With an increase in the use of polymers in the microelectronic industry, microscopic understanding of the mechanism responsible for adhesion between metals and polymers has become increasingly important. Among the polymers suitable for microelectronic applications, polyimides (PI) have received increasing attention due to their thermal and chemical stability, low dielectric constant, high electrical resistivity and relative ease of processing into coatings and films. Polyimides have been widely used in microelectronics as dielectric spacing layers, protective coatings and substrates for metal thin films [1].

Various surface treatments and modification methods have been used to enhance adhesion of metal no polyimide. These techniques include the use of ion beam, photografting, plasma, and sputtering. Most of these methods require high vacuum equipment and the productivity is low. Thus, they are not economically feasible. These methods may also introduce undesirable layer of foreign materials into the interfaces, resulting in possibility reliability failure. On the other hand interests in wet-process surface modification of polyimides have increased due to simplicity and low cost. Polyimide films are resistant to most solvents and chemicals, but they react with oxidizing or reducing agents. If the concentration of the chemical reagents, reaction temperature, and reaction time are well controlled, the reactions can be confined to the surface [2].

The study focuses on the surface modification of PI surface by KOH, before ion-exchanged with Ag+ and subsequently reduced silver-ion to form metallic silver film. The effects of time and concentration of KOH in surface modifying step on the thickness of the coated silver film were investigated.

2. Materials and Method

Commercialized PI substrate film (Kapton 100HN) was used in this study. The processing steps for the direct metallization are schematically presented in Figure 1. Polyimide films were first immersed into 30 ml of KOH solution at 40 °C for 1 min in order to cleave imide ring to form carboxylic acid groups (PAA) and amide bonds on its surface and then washed with deionized water. After that silver ions were impregnated into the surface-modified film by immersing the film into 30 ml of AgNO3 solution (0.01 M) for 1 min at room temperature and rinsed with deionized water. To reduce silver ion to form metallic silver, silver ion doped film was immersed into 20 ml of aqueous $NaBH_4$ solution (0.1 g/100 ml of deionized water) at room temperature for 20 seconds. Finally, the film was heat-treated at 110°C for 30 mins in air to convert PAA to PI. In this work, the concentration of KOH solution was changed from 0.1, 1 and 2 M, while the KOH treatment time was changed from 1, 30 and 60 mins

Functional group of the modified PI film was characterized by Fourier Transforms Infrared spectroscopy (FT-IR). The thickness of the silver film was monitored via the transparency of the film, which was characterized by UV/Visible spectroscopy. Finally, the resistivity of coated silver film was measured by LCR meter.

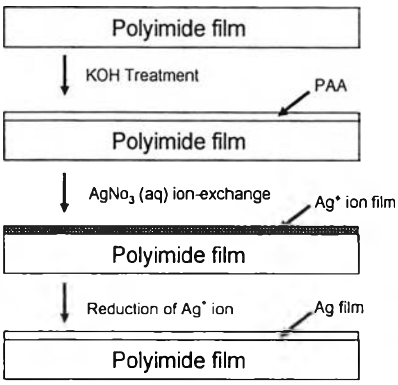

Figure 1. Process of silver coating onto polyimide film.

3. Results and Discussion

Although PI is chemically inert, PI undergoes hydrolysis reaction with strong base such as KOH, which results in cleavage of imide rings and the formation of carboxylic groups. At the same time, KOH treatment gives rise to the formation of the potassium salts of carboxylic acid, as shown in Figure 2. This potassium ion can be later exchanged with silver ions. This behavior was confirmed by FTIR results. The FTIR spectra (not show) revealed that bare PI has absorption bands at around 1720, 1780, 1500, 1380 and 1230 cm^{-1}, corresponding to symmetric C=O stretching, asymmetric C=O stretching, C-C stretching of benzene rings, C–N–C stretching in imide rings and aromatic ether (Ar–O–Ar) stretching, respectively. After KOH treatment, the intensity of the imide peak at wave number around 1720 and 1780 cm^{-1} was decreased as shown in Table 1. Relative intensity of the absorption band at 1720 cm^{-1} decreases from 0.4295 to 0.3591, while the band at 1780 cm^{-1} drops from 2.7432 to 2.3869, which indicates the cleavage of the imide ring to form carboxylate (-COOK) and amide (-CONH-) groups. It should be noted that the absorption band at 1015 cm^{-1} was used as the internal standard for the calculation of the relative FTIR intensities. It should also be noted that there was a slight increase in intensity of the absorption bands at 1780 and 1720 cm^{-1} as shown in Table 1. The relative intensity of the band at 1780 cm^{-1} increases from 2.3869 to 2.5518, while the band at 1720 cm^{-1} increases from 0.3591 to 0.4122, after the coating with silver. This was the result from the reformation of imide ring [3]

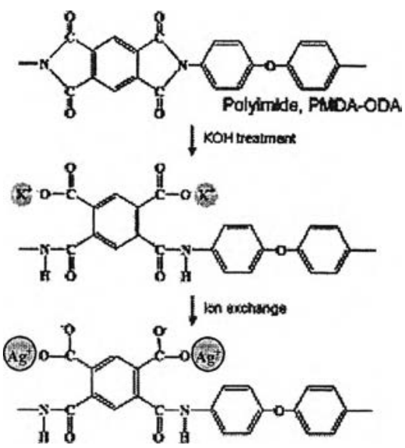

Figure 2. Surface modification of PI with KOH and $AgNO_3$

Although the actual thickness of the silver film was not measured in this work, but the transparency of the film could be used to represent the silver film thickness, since the PI film is transparent, while the silver coating is opaque. Figure 4 shows the transmittance of the light at wavelength of 650 nm through the PI film modified at different conditions and subsequently coated with silver film under the same condition.

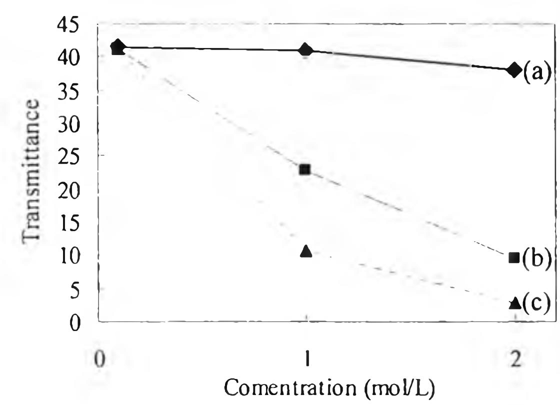

Figure 4. Transmittance of the light through PI film modified the KOH at different concentrations for: (a) 1 min, (b) 30 mins and (c) 60 mins. The films were subsequently coated with silver film.

Table 1. Intensities of FTIR absorption bands for samples prepared at different steps.

Sample	Intensity of absorption band			Relative intensity	
	At 1780 (cm^{-1})	At 1720 (cm^{-1})	At 1015 (cm^{-1})	1780 / 1015	1720 / 1015
Bare PI films	0.6419	0.1005	0.2340	2.7432	0.4295
KOH Modified film	0.6089	0.0916	0.2551	2.3869	0.3591
Silver Coated film	0.6101	0.0983	0.2385	2.5581	0.4122

Table 2. Intensities of FTIR absorption bands for PI modified with KOH at various concentrations.

Concentration of KOH (M)	Intensity of absorption band			Relative intensity	
	At 1780 (cm^{-1})	At 1720 (cm^{-1})	At 1015 (cm^{-1})	1780 / 1015	1720 / 1015
0.1	0.6003	0.1296	0.2367	2.5361	0.5475
1.0	0.6197	0.1228	0.2507	2.4719	0.4898
2.0	0.6389	0.1212	0.2590	2.4668	0.4680

It is shown that, when either KOH concentration on the modification time was increased, the transmittance was significantly decreased. It means that these conditions results in the cleavage of imide rings in greater extent, as witnessed from the results in Table 2, in which the relative height at wave number 1720 cm^{-1} drops from 2.5361 to 2.4468 and at 1780 cm^{-1} drops from 0.5475 to 0.4680. The more imide rings was cleaved, the more silver ions could be incorporated with the carboxylate formed. This makes silver layer thicker this is confirmed by the fact that the resistance of the film was decreased from 389.33 Ω/cm^2 to 0.40 Ω/cm^2, when the concentration of KOH was changed from 0.1 M to 2 M. When the modification time was changed from 1 min to 60 mins, resistance of silver coated film decrease from 389.33 Ω/cm^2 to 176.44 Ω/cm^2.

According to the 2^k factorial design of experiments [4], it was found that the modification time with KOH had the more immense effect on the thickness of the silver film coated on PI than the concentration of KOH (with the statistical parameters of 61.55 versus 10.20). This result also indicated that the diffusion of KOH into PI played an important role during the modification step

4. Conclusion

This work demonstrates that thickness of silver layer on PI surface can be controlled by concentration of KOH solution and length time during the PI surface modification step. It is also proofed that time has much more effect than a concentration of KOH solution for modifying PI surface and growth of silver layer

5. Acknowledgement

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VITA

Mr. Patinan Chalephol was born on 30th January, 1985, in Bangkok, Thailand. He received his Bachelor degree of Engineering with a major in Chemical Engineering from Srinakharinwirot, Bangkok, Thailand in May 2007. He continued his Master study in the major in Chemical Engineering at Chulalongkorn University, Bangkok, Thailand in May 2007.