ECONOMICAL ASPECTS OF DILUTED HF SOLUTIONS ON THE ANTIREFLECTIONS PROPERTIES OF THE FLOAT GLASS SURFACES

Jan Wasylak Prof.
Joanna Zontek-Wilkowska, MSc., UST-AGH
University of Science and Technology, Faculty of Materials Sciences and Ceramics, Department of Glass and Amorphous Coatings Technology, Cracow, Poland

Abstract

This work focuses on the development of a simple and low cost alternative to deposited antireflective coatings (ARC) through the use of a diluted HF wet chemical etching technique to form AR layers of silicon dioxide. Soda-lime-silica, low-iron glasses were used as the glass substrates. The float glass was etched for a variety of time intervals and with four different concentrations of HF solutions (0,5M, 1M, 5M, 10M). The optical properties were determined by UV-VIS spectroscopy. The microstructures were examined by SEM microscopy with EDAX analysis. The surface roughness after the wet chemical-etching was determined using an optical profilmeter.

Keywords: ARC-coating, wet chemical etching technique, reflection losses, polished

Introduction:

Coatings and thin nano-layers have been applied on glass in order to obtain various properties, which render the glass more attractive, such as anti-reflection (AR), anti-static, defogging, anti-abrasion, self-cleaning (SC), solar control and electrical conductivity [1,3]**.**

Reflection of float-glass can be reduced and as well the transmission can be increased by using antireflective coatings (AR). In general there are two different anti-reflective coating technologies. Interference-type multilayer systems produced by sol-gel method [1] and sputtering techniques [2] and single layer nanoporous silica-based systems manufactured by dip-coating techniques [3], and also by printing techniques [4].

AR-coatings have many applications, such as spectacles, cameras, binoculars, lasers, solar cells, planar displays and others optical and optoelectronic devices. AR-technology can be used for windows in residential and commercial buildings in order to enhance the view and the energy performance of the windows. Composites of different inorganic matrixes with inorganic layers have interesting combinations of optical, electrical, thermal, magnetic, and mechanical properties as well as enhanced chemical resistance and flame-retardancy. Nano and sub micrometer particles as well known as nano and submicrometer structured materials represent one of the most rapidly expanding fields in science [5]. Recently, chemical etching process becomes attractive as an alternative of a dielectric AR layer material because of its good transparency, low refractive index and ability to form texture coating via etch processes [12-20].

In this paper the properties of AR thin films obtained by wet-etching process have been reported. Due to the phenomena of chemical etching process for a variety of time intervals and with four different concentrations of HF solutions (0,5M, 1M, 5M, 10M) was proceed.

Experimental Part

Float SLS glass plates (4 mm thick), of dimension 50x50 mm^2, were used as coating substrates. The glass samples were cleaned in the ethanol solution and deionised water for 10 minutes independently to remove all contaminate and degreased.

After cleaning process, all samples were etched for a variety of time intervals (10s, 20s, 90s, 310s) and with four different concentrations of HF solutions (0,5M, 1M, 5M, 10M), then samples were heat treated at 640^0C per 15 min.

The morphology of the AR coating was studied by scanning electron microscopy (Nora Nano-SEM). Transmittance and reflectance of following samples of thin films were measured on UV–VIS spectrometer Jasco V 300 in the spectral range between 300 and 1100 nm.

Results

The antireflection properties of thin layers are dependent on the concentrations of HF solutions for wet-chemical etching system, etching time and also thermal conditions of the heat treatment, respectively.

Optical Properties

All prepared thin fimls have got high transmitance level. The sample which was etched into the 1M HF solution per 90s is charecterized by the highest transmittance level and the lowest reflectace level. Increasing of concentration HF into water solution could cause the micro cracks which could indicate bad imapct into the optical propertis of the glass surface. The lowest (89.9 %) transmittance was observed in case glass samples etched into 10M HF solution via 310s , on the other hand the transimittance of this sample was high enough to be used as an AR layer. The transmittance and reflectance spectra of the thin films are shown in the Fig.1 –Fig 2

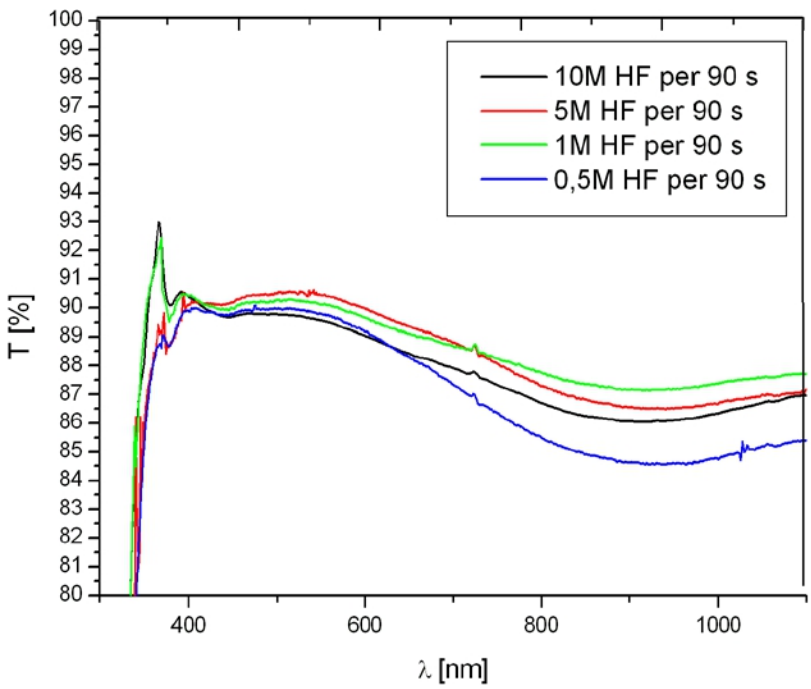

Figure 1 Transmittance spectra for 90 s etching time

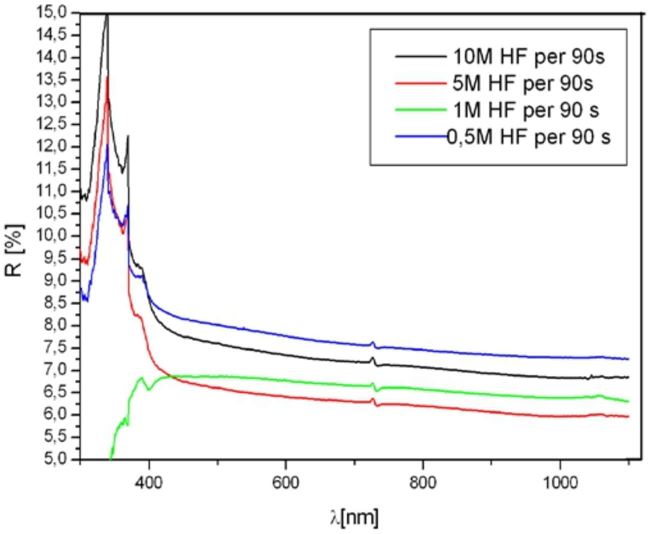

Figure 2 Refrectance spectra for 90 s etching time

Microstructure And Surface Profile

Scanning electron microscopy is a convenient method for studying the microstructure of thin films. The microstructure of etching glass samples are shown at photos 1- 3 presented below. All layers are characterized by hierarchical structured. The EDAX analysis confirmed that the silica concentrations on the glass surface increased above 30,67 at. % for sample etched per 90s into 1M HF solution, compared to the parent-uncoated glass. In the table 1, has been shown the results of elements concentration changes for samples etched via 90 s in different acid solutions.

Table 1 The avarage elements concentration changes into the glass surface etched by different concentrations of HF acid solutions

Element	0,5M HF	1,0M HF	5,0M HF	10,0MHF
O	59,24	53,72	53,33	53,02
Na	9,09	9,04	9,07	9,06
Mg	2,29	2,50	2,55	2,57
Al	0,57	0,58	0,52	0,51
Si	25,87	30,67	30,07	30,53
Ca	2,97	3,49	3,46	3,74

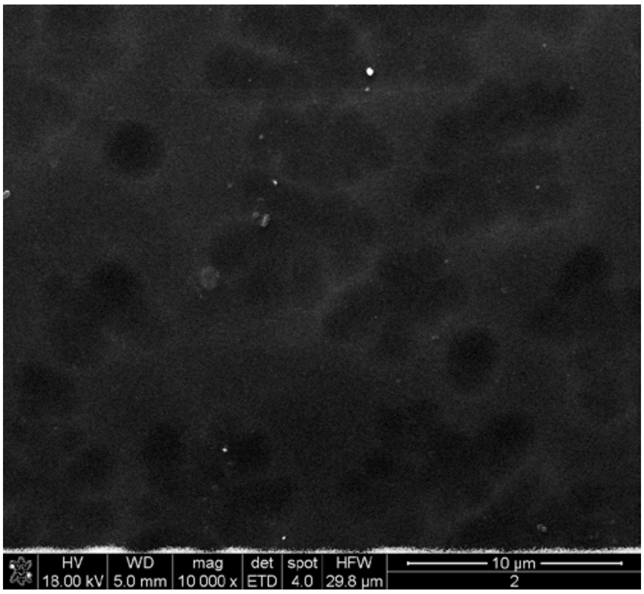

Photo 1 Microstruce of glass surface etched into 10M HF solution for 90 s

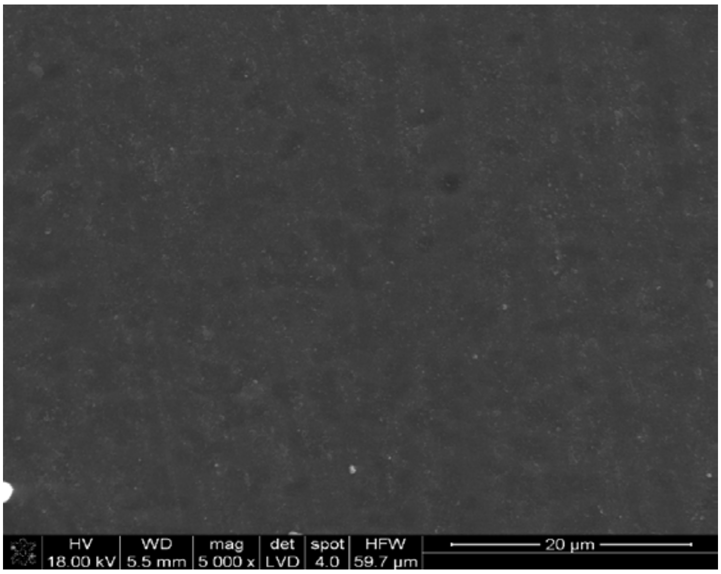

Photo 2 Microstruce of glass surface etched into 5M HF solution for 90 s

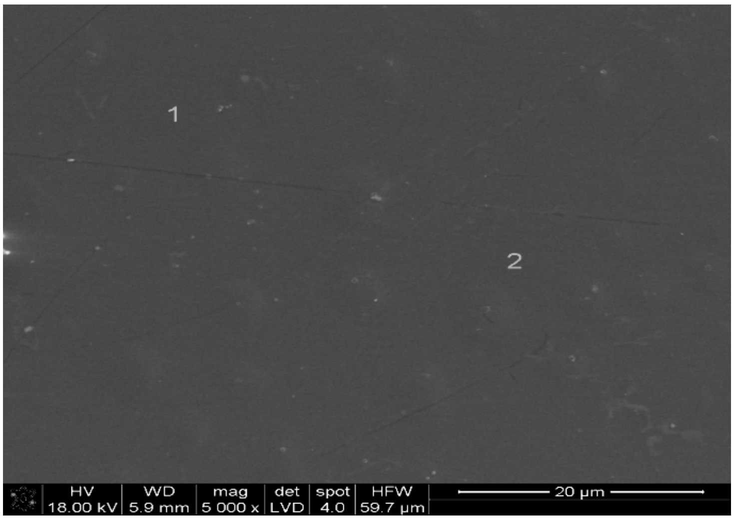

Photo 3 Microstructure of glass surface etched into 1M HF solution for 90 s

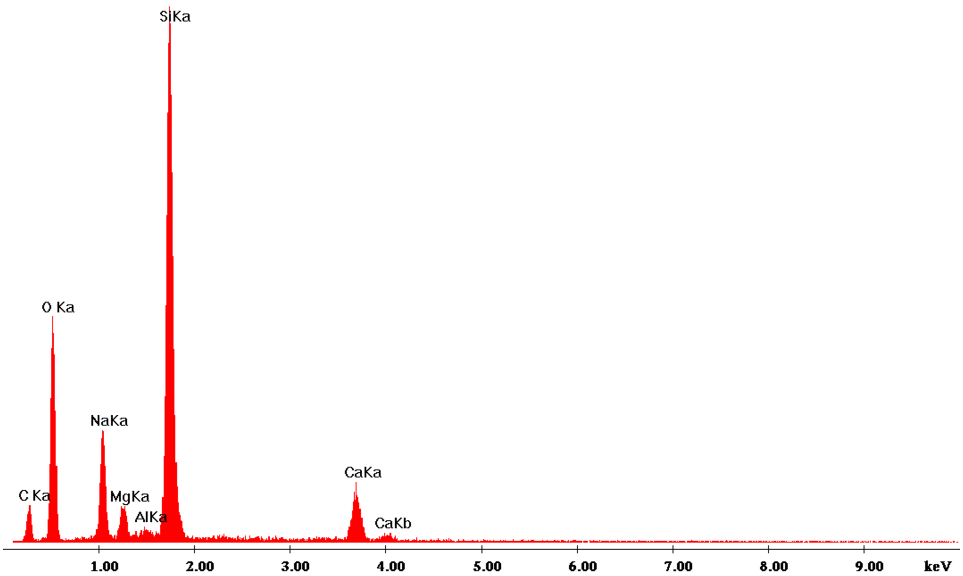

Figure 3 EDAX analysis in point 1

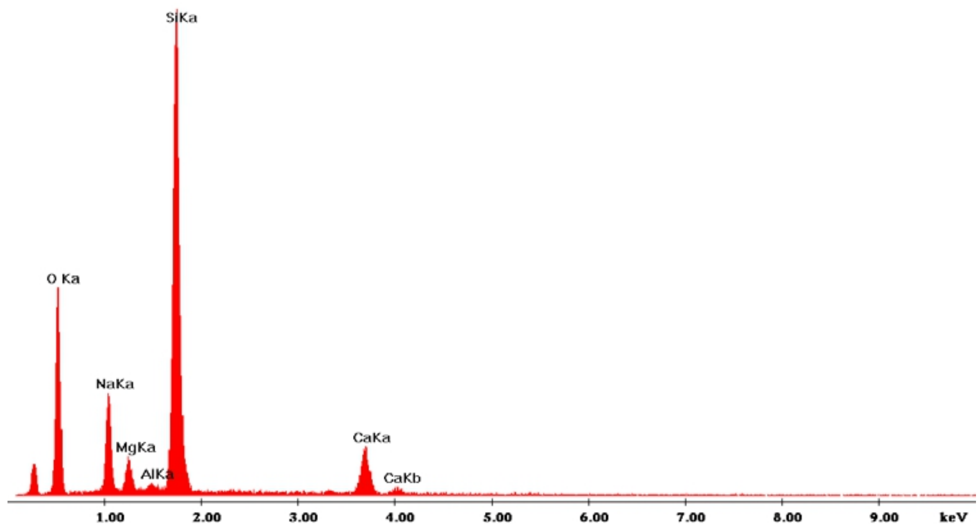

Figure 4 EDAX analysis in point 2

Moreover, there was exanimated the surface roughness, using optical profilometr WYKO NT930. The results show that there is a noticeable, significant increase of the surface roughness, using stronger acid solutions. The highest value of roughness 0,3 µm had been observed for the glass sample etched into 10M HF solutions per 90s. The rest results oscillated into the same value 0,17-0,19µm.

Conclusion:

We have reported the influence of etching process into on the optical properties and microstructure of antireflection thin films. A simple two step procedure has been established and permitted to form the thin films with suitable parameters for ARC-coatings.

1. It was observed that the etching processes conducted into 1M HF gives the best results of transmittance above 94 % and the lowest value of reflectance spectra.
2. It has been seen the noticeable increase of the surface roughness, for the sample etched into stronger acid solutions. The highest value of roughness 0,3 µm had been observed for the glass sample etched into 10M HF solutions per 90s. The rest results oscillated into the same value 0,17-0,19µm.
3. The concentration of silica into the glass surface increase in all cases, but highest value, despite of the optical parameters losses got the sample etched into 10M HF solutions per 90s.

References:

D. Chen, Anti-reflection (AR) coatings made by sol-gel processes: A review, Solar Energy Materials and Solar Cells, 68, (2000), 313-336

H. K. Pulker, Coatings on glass, (1999), 2ed, Elsevier, Amsterdam

[D. Chen, Anti-reflection (AR) coatings made by sol-gel processes: A review, Solar Energy Materials and Solar Cells, 68, (2000), 313-336, P. Nostell, A. Roos, B. Karlsson, Antireflection of glazings for solar energy applications, Solar Energy Materials and Solar Cells, 54, (1998), 223-233.

M. Fukawa, T. Ikeda, T. Yoneda ans K. Sato, Antireflective coatings with single layer with refractive index of 1.3, Proceedings of the 3rd International Conference on Coatings on Glass (ICCG), (2000), 257-264

Yoshitaka Nakano, Takeshi Morikawa, Takeshi Ohwaki and Yasunori Taga, Appl. Phys. Lett. 88 (2006) 172103.
Y. Kanamori, M. Sasaki, K. Hane, Opt. Lett. 24 (1999) 1422
K. Hadob´as, S. Kirsch, A. Carl, M. Acet, E.F. Wassermann, Nanotechnology 11(2000) 161.
S. Wang, X.Z. Yu, H.T. Fan, Appl. Phys. Lett. 91 (2007) 0611058 3 pages.
G.-R. Lin, Y.-C. Chang, E.-S. Liu, H.-C. Kuo, H.-S. Lin, Appl. Phys. Lett. 90 (2007) 1819238, p.3
C.H. Sun, W.-L. Min, N.C. Linn, P. Jiang, B. Jiang, Appl. Phys. Lett. 91 (2007) 2311058,p.3
C.H. Sun, P. Jiang, B. Jiang, Appl. Phys. Lett. 92 (2008) 0611128 3 pages.
J. Zhu, Z. Yu, G.F. Burkhard, C.-M. Hsu, S.T. Connor, Y. Xu, Q. Wang, M. McGehee, S. Fan, Y. Cui, Nano Lett. 9 (2009) 279.
E. Garnett, P. Yang, Nano Lett. 10 (2010) 1082.
Y. Lu, A. Lal, Nano Lett. 10 (2010) 4651.
M.-L. Kuo, D.J. Poxson, Y.S. Kim, F.W. Mont, J.K. Kim, E.F. Schubert, S.-Y. Lin, Opt. Lett. 33 (2008) 2527.
P. Yu, C.-H. Chang, C.-H. Chiu, C.-S. Yang, J.-C. Yu, H.-C. Kuo, S.-H. Hsu, Y.-C. Chang,Adv. Mater. 21 (2009) 1618.
S.Y. Chou, P.R. Krauss, J. Renstrom, Appl. Phys. Lett. 67 (1995) 31148 3 pages.
V. Boerner, S. Abbott, B. Blasi, A. Gombert, W. Hobfeld, SID. Symp. Digest 34 (2003)68.
C.J. Ting, M.-C. Huang, H.-Y. Tsai, C.-P. Chou, C.-C. Fu, Nanotechnology 19 (2008) 2053018 5 page.
Y. Kanamori, E. Roy, Y. Chen, Microelectron. Eng. 78–79 (2005) 287.
Mitsuhisa Kawakami, A.B. Hartanto, Y. Nakata, T. Okada, Jpn. J. Appl. Phys. 42(2003) L 33.
K.-S. Kim, H.W. Kim, Phys. B 328 (2003) 368.
R. Liu, A.A. Vertegel, E.W. Bohannan, T.A. Sorenson, J.A. Switzer, Chem. Mater. 13(2001) 508.
L.E. Greene, M. Law, D.H. Tan, M. Montano, J. Goldberger, G. Somorjai, P. Yang, Nano Lett.5 (2005) 1231.
Y. Tak, K. Yong, J. Phys. Chem. B 109 (2005) 19263.
Z. Chen, L. Gao, J. Cryst. Growth 293 (2006) 522.
M. Riaz, A. Fulati, G. Amin, N.H. Alvi, O. Nur, M. Willander, J. Appl. Phys. 106 (2009) 034309.
Y.J. Lee, D.S. Ruby, D.W. Peters, B.B. McKenzie, J.W.P. Hsu, Nano Lett. 8 (2008) 1501.
T.H. Ghong, Y.D. Kim, E. Ahn, E. Yoon, S.J. An, G.–.C. Yi, Appl. Surf. Sci. 255 (2008) 746.