Serbian Tribology Society

SERBIATRIB '13

13th International Conference on Tribology

Faculty of Engineering in Kragujevac

Kragujevac, Serbia, 15 – 17 May 2013

TRIBOLOGICAL STUDY OF BIOCOMPATIBLE HYBRID ORGANIC MOLECULES FILM WITH ANTIBACTERIAL EFFECT

J.H. Horng*[1], C.C.Wei[1], S. Y. Chern[1], W.H. KAO[2], K.W. Chern[1], Y.S. Chen[1]

[1]Department of Power Mechanical Engineering, National Formosa University, Taiwan
[2]Institute of Mechatronoptic Systems, Chienkuo Technology University, Taiwan
*jhhorng@gmail.com

Abstract: *Optical glass is widely used on bioengineering and various utilities such as public touchscreen display and mobile devices. This work evaluates the feature of anti-bacterial and anti-adhesion on OTS material that mixed with biocompatibility antibacterial agent coated on the optical glass. Test samples were allocated with different bath and drying temperatures as well as reaction time. The result shows that in the contact of angle experiments, pure OTS film and mixed antibacterial films have almost the same contact angle about 105° at the condition of reaction time of 12 hours and reaction temperature of 80°C.The antibacterial test find that the order: antibacterial agent> OTS+ antibacterial agent(50%) > OTS+ antibacterial agent(10%) > OTS. At the same operation condition, OTS mixed with 50% antibacterial agent is able to increase adhesion force between OTS film and lens. It suggests that the surface treatment of optical lenses involving OTS with 50% antibacterial solution is the most to increase the antifouling and antibacterial functions and enhance the adhesion function between films and lens surfaces.*

Keywords: *self-assembled monolayer, adhesion force, friction, terminal group bonding, contact angle, anti-bacterial.*

1. INTRODUCTION

The uses of SAMs in biomedicine utilities are increasing rapidly, such as in biosensors, nonfouling surfaces, bioactive surfaces, and drug delivery [1, 2]. Octadecyltrichlorosilane (OTS) monolayer is one of the most extensively studied self-assembled monolayer [3-5]. Therefore, how to improve the adhesion and anti-bacterial performance of SAMs film becomes an attractive topic in order to enhance device application and reliability. Bierbum [6, 7] noted that the substrate surface water layers are an important factor in the formation of OTS films. Bierbum explained that OTS molecules initially spread vertically on substrate surfaces and been clustered after locating activation positions. Afterwards, other OTS molecules spread to the cluster edges and form islands. The molecules then spread outwards and cause adsorbed molecules to form connections, finally forming tightly connected monolayers. In 1998, Vaillant et al. [8] used atomic force microscopy (AFM) and a Fourier-transform infrared spectrometer (FTIR) to observe the process by which OTS molecules form films on substrate surfaces. The results showed that a larger amount of water in the solutions cause the OTS molecules to undergo a hydrolysis reaction and produce polymerization within the solution. Cloud-shaped or island-shaped molecule films form throughout the solution. In the contrast, solutions with comparatively low proportions of water exhibit point distribution and OTS molecules igrow chaotically into liquid-like form. While the surface diffusion makes OTS molecules absorbing molecules within the solution, the tightly knit, island-shaped structures are formed by messy molecule films. Resch [9] also used AFM and found that OTS molecules initially grow messily and irregularly. With the passage of time, molecules covering the surface spread horizontally and ultimately form tightly arranged molecular films. Carraro et al. [10] examined formation of OTS SAMs under different ambient temperatures. They discovered when the ambient temperature falls below 16°C, OTS first form islands or clouds and then films. When the ambient temperature rises above 40°C, the films grow evenly instead of forming islands. In addition, films form more quickly at lower temperatures. The formation of OTS monolayer on a material surface is highly sensitive to several factors, which include the density of surface hydroxyl groups, reaction temperature, reaction environment, reaction time, solvent used to deposit OTS water content of the solvent concentration of OTS, solution age, roughness of the underlying substrate and cleaning procedures after SAM deposition [11]. Therefore, how to improve the adhesion and anti-bacterial performance of SAMs film becomes an attractive task in order to enhance device application and reliability.

2. EXPERIMENTAL

The optical lenses were ultrasonicated in acetone and then rinsed with solvent tetrahydrofuran and deionized water and immediately dipped in the OTS solution containing approximately 40 ml. For the preparation of SAMs film, OTS was dissolved in alcohol and prepared to a molar concentration of 10 mM, and then mix in different proportions of antibacterial agent (10%, 50%). The test pieces were placed in the solution at different bath temperatures and duration times and a drying time of 10 min. The test pieces were then removed and set aside for 12 hr before being ultrasonicated in acetone for 5 min to remove loosely bound material and rinsed in deionized water and blown dry with nitrogen gas. The molecular structure of OTS is shown in Table 1. It's hydrophobic properties comes from terminal group (CH_3). Main composition of biocompatibility antibacterial agent is bioflavonoid and citric acid.

For the experimental investigation of hydrophobic properties for the different surface films on the lens, FTA contact angle equipment was used to measure contact angle, as shown in Figure 1. Larger contact angle indicates better hydrophobic and anti-fouling properties of surfaces. Contact angles were measured on both sides of the water drop. Droplet profiles were captured using a video comprising of digital frames over a period of 12 seconds and transferred to a computer for angle measurement. The adhesion force between surface films and substrates were measured using atomic force microscopy (AFM) by scratch mode. AFM also was used to examine topography of samples before and after SAM deposition by non-contact mode.

Table 1. The molecular structure of OTS

SAMs	Molecular formula	Head group	Terminal group
OTS	$CH_3(CH_2)_{17}SiCl_3$	$-SiCl_3$	$-CH_3$

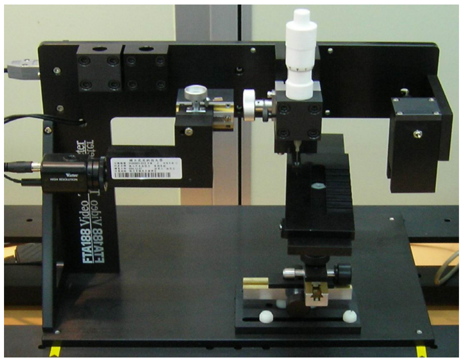
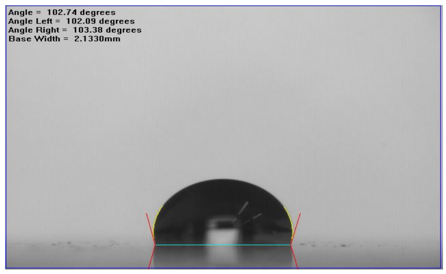


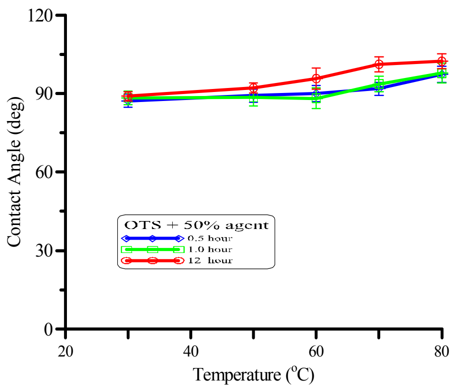
Figure 1. Contact angle equipment

3. RESULTS AND DISCUSSION

In the contact angle analysis of various operation conditions, the measurement data of each test piece was obtained from the mean of five measurements. Figure 2(a) is a photo of the contact angle for OTS material. Figure 2(b) show the contact angle changes with various reaction times and bathe temperatures.



(a)



(b)

Figure 2. Contact angles (a) experimental photo (b) comparison chart for different reaction times and temperatures.

It shows that the higher the bathe temperature, the higher the contact angle. The higher the reaction time, the higher is the contact angle. However, the variation of contact angles of OTS+50% antibacterial agent films under various operation conditions are all quite low. Bathing OTS+50% agent films at a bath temperature of 80 °Cgradually increases the contact angle to approximately 105 degree. The various reaction time and bathe temperature have extremely little influence on the contact angle. In summary, the bath temperature of 80°*C* and duration time of 12 hours was chosen as operation condition in order to investigate the antibacterial characteristics of surface film on lenses.

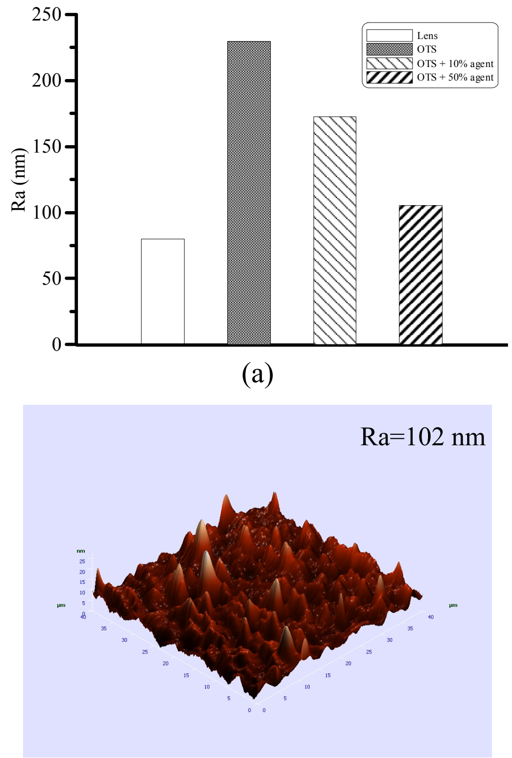


Figure 3. (a)The roughness values of the different surface films (b) 3-D topography image of the OTS + 50% agent film.

The various roughness values of different surface materials are shown in Figure. 3. Roughness test were conducted in air at a relative humidity of about 50% using AFM by non-contact mode. The scanned detection range was 40 µm × 40 µm. The various surface roughness value of different surface materials are shown in Figure 3(a). The comparison chart shows that antibacterial agent can decrease the surface roughness value of pure OTS films. The roughness value of OTS film surface adding 10% antibacterial agent is approximately 175 nm, whereas the OTS film roughness value adding 50% antibacterial agent was decreased to approximately 100 nm. The 3-D topography image for the hybrid organic molecules film (OTS + 50% agent) is shown as Figure 3(b). The island-shaped structures were formed on the surface, as mentioned in Vaillant's work [8]. It shows hybrid organic film exhibit uniform coverage the surface with regular pattern of island formation. It indicates that antibacterial agent absorbed and stored in the topographic valley of OTS film.

The reliability and beauty requirement of the display elements made from company become important in their service life. The light transmittance and film adhesion properties are one of key performances of lens. In order to explore the relation between surface film and light transmittance of lens, Figure 4 shows that transmittance of OTS film and antibacterial agent on the lens. This result indicates that the OTS surface film will decrease the light transmittance of lens. However, the antibacterial agent has extremely little influence on the transmittance of lens. The minimum value of transmittance is 93.6% under the film of OTS + 50% agent. It concludes that all transmittance of surface films is acceptable for industrial applications in our work.

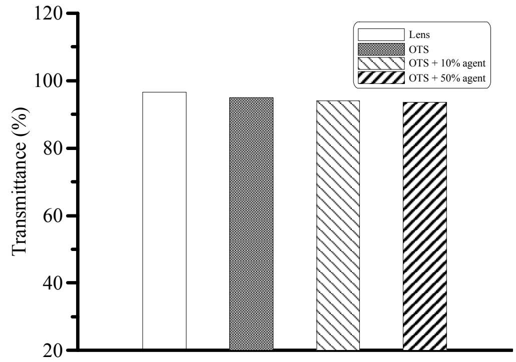


Figure 4 The light transmittance of different surface films on lens

The film adhesion is another one of key performances of lens for reliability. Figure 5 shows the effect of antibacterial agent on the critical load of surface films on the lens. It shows that antibacterial agent increases adhesion force between OTS film and lens. Mixing antibacterial agent (50%) in OTS material increases the critical load to approximately 104μN. In summary, the surface treatment of optical lenses involving OTS+ Agent (50%) is the most capable of effectively increasing anti-adhesion functions.

In the antibacterial tests, staphylococcus aureus were inoculated with different self-assembled film, and then after 24 hours to measure bacteria values (JISZ 2801:2010). Figure 6 is the comparison chart of the number of the bacteria for the different surface films. For the general lens surface, the bacteria number is about 135000 after 24 hours. The pure OTS film also has little antibacterial function. It shows that the bacteria number on OTS with 50% antibacterial agents and pure antibacterial agent surface is less than 10. It is far lower than the bacteria value, 5.3 × 10^4, on the OTS film.

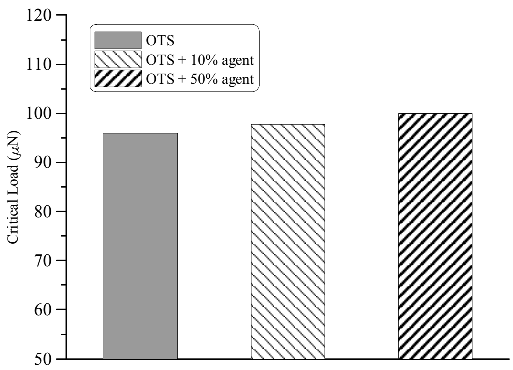


Figure 5. Critical loads between surface films and substrate

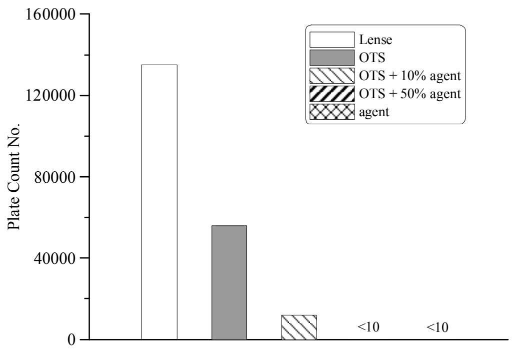


Figure 6. Effect of surface film material on antibacterial

4. CONCLUSION

This work studied the feature of anti-bacterial and anti-adhesion on OTS self-assembled monolayers which mixed with biocompatibility antibacterial agent that coated on optical lens. The results can be concluded as follows:

1. Both OTS and mixed OTS film can effectively increase the contact angle of a lens surface at various bath temperatures as well as duration time and reduces device adhesion force effectively.
2. The adding of antibacterial agent has little effect on the contact angle and light transmittance of pure OTS film.
3. The antibacterial agent can effectively reduce the surface roughness while increase the adhesion force and antibacterial abilities of pure OTS film on lens surfaces (reaction time = 12hr., reaction temperature = 80°C).

ACKNOWLEDGMENTS

The author gratefully acknowledges common Lab. for Micro-Nano Science and Technology at NFU.

REFERENCES

[1] M. Masuko, H. Miyamoto, A. Suzuki: *Tribological characteristics of self-assembled monolayer with siloxane bonding to Si surfac*, Tribology International, Vol. 40, pp. 1587-1596, 2007.

[2] G. Mani, D.M. Johnson, D. Feldman, D. Patel, A.A. Ayon, C.M. Agrawal: *Drug delivery from gold and titanium surfaces using self-assembled monolayers*, Biomaterials, Vol. 29, pp. 4561-4573, 2008.

[3] A.N. Parikh, D.L. Allara, I.B. Azouz, F. Rondelez: *An Intrinsic Relationship between Molecular Structure in Self-Assembled n-Alkylsiloxane Monolayers and Deposition Temperature,* Journal of Physical Chemistry, Vol. 98, pp. 7577-7590, 1994.

[4] C. Carraro, O.W. Yauw, M.M. Sung and R. Maboudian: *Observation of Three Growth Mechanisms in Self-Assembled Monolayers*, Journal of Physical Chemistry B, Vol. 102, pp. 4441-4445, 1998.

[5] R. Brambilla, F. Silveira, J. Santos: *Investigating morphological changes on octadecyl modifiedsilicas by SEM and AFM*, Modern Research and Educational Topics in Microscopy, pp. 626-633, 2007.

[6] P. Silberzan, L. leger, D. Auserre, J.J. Benatter :*Silanation of Silica Surfaces. A New Method of Constructing Pure or Mixed Monolayers*, Langmuir, Vol. 7, pp.1647-1651, 1991.

[7] K. Bierbaum, M. Grunze, A.A. Baski, L.F. Chi, W. Schrepp, H. Fuchs :*Growth of self-assembled n-alkyltrichlorosilane films on Si(100) investigated by atomic force microspcopy*, Langmuir, Vol. 11, pp.2143-2150, 1995.

[8] T.Vallant: *Formation of Self-Assembled Octadecylsiloxane Monolayers on Mica and Silicon Surfaces Studied by Atomic Force Microscopy and Infrared Spectroscopy*, Journal of Physical Chemistry B, Vol.102, pp.7190-7197,1998.

[9] R. Resch, M. Grasserbauer, G. Friedbacher, T.h. Vallant, H. Brunner, U. Mayer, H. Hoffmann: *In situ and ex situ AFM investigation of the formation of octadecylsiloxane monolayer*, Applied Surface Science, Vol. 140, pp.168-175, 1999.

[10]C. Carraro, O.W. Yauw, M.M. Sung, R. Maboudian: *Observation of Three Growth Mechanisms in Self-Assemble Monolayers*, Journal of Physical Chemistry B. Vol. 102, pp. 4441-4445, 1998.

[11] G. Mani, D.M. Feldman, O. Sunho, C.M. Agrawal: *Surface modification of cobalt-chromium-tungsten-nickel alloy using octadecyltrichlorosilanes*, Applied Surface Science, Vol. 255, pp. 5961-5970, 2009.