# REFERENCES


1. Katsunari, N., Studies on peri-root tissue formation around new type artificial root made of dense hydroxyapatite. Clin. Mat. 12,3 (1993): 159-167.
2. Teraoka, K.; Naomi T.; Doi, Y.; Taoda H.; Nagamura, K.; Yokogana, Y.; Kameyana, T. Hydroxyapatite implantation on the surface of pure titanium for orthopedic implants. Mater. Sci. Eng. C13,1 (2000): 105-107.
3. Lonenstam, H. A.; Weiner, S. On Biomineralization. Oxford: Oxford University Press, 1989.
4. Nancollas, G. H. Biological Mineralization and Domineralization. Springer, Berlin: Dalhem Konferenzen, 1982.
5. Legeroz, R. Z.; Legeroz,J. P. in: J.O. Nriago. Moore, P.B. (Eds.). Phosphate Minerals p.351. Berlin: Springer, 1984.
6. Hench, L. L. Bioceramic: From concept to clinic. J. Am. Ceram. Soc.. 74,7 (1991): 1 487-1510.
7. Bertoni, E.; Bigi, A.; Cojazzi, G.; Gandolfi, M.; Panzavolta, S.; and Roveri N. Nanocrystals of magnesium and fluoride substituted hydroxyapatite. J. Inor. Biochem. 72 (1998): 29-35.
8. Park, J. B. Biomaterials Science and Engineering. New York: Plenum Press, 1987.
9. Suchanek, W.; Yashima, M.; Kakihana, M.; and Yoshimura, M. Hydroxyapatite/ hydroxyapatite-whiskers composites without sintering additives: mechanical properties and microstructural evolution. J. Am. Ceram. Soc. 80,11 (1997): 2805-2813.
10. Ioku, K.; Yoshimura,M.; and Somiya S. Hydrothermal synthesis of Ultrafine hydroxyapatite single crystal. Nippon Kagaku Kaishi. 9 (1998) 1565-1570 in Japanese.
11. Kinoshita, M.; Itatani, K.; Nakamura, S.; and Kishioka, A. Preparation and morphology of carbonate-containing hydroxyapatite by homogeneous precipitation and hydrothermal methods. Gypsum & Lime. 227 (1990): 207-215 in Japanese.

12. Christiansen, N.; and Riman, R.E. Bioceramics-A future through microstructural and chemical design. Proceedings of the 5th Scandinavian symposium on materials science, New materials and processes. (1989): 209-220.

13. Mortier, A.; Lemaitre, J.; Rodrique, L.; and Rouxhet, P.G. Synthesis and thermal behaviour of well-crystallized calcium-deficient apatite. J.Solid State Chem. 78 (1989): 215-219.

14. Kinoshita, M.; Itatani, K.; Nakamura, S.; and Kishioka, A. Preparation and morphology of carbonate-containing hydroxyapatite by homogeneous precipitation and hydrothermal methods. Gypsum & Lime. 227 (1990): 207-215 in Japanese.

15. Yoshimura, M.; Suda, H.; Okamoto, K.; and Ioku, K. Hydrothermal synthesis of needle-like apatite crystal. Nippon Kagaku Kaishi. 10 (1991): 1402-1407 in Japanese.

16. Suda, H.; Asaoka, N.; and Yoshimura, M. Preparation and characterization of 1hydroxyapatite whiskers by hydrothermal method. Bioceramics vol.5. Proceedings of the 5th International symposium on ceramics in medicine, Yamamuro, T.; Kokubo, T.; and Nakamura, T.(eds). Konbunshi Kankokai. Japan: Kyoto (1994): 31-34.

17. Yoshimura, M.; and Suda, H. Hydrothermal processing of hydroxyapatite:Past, present, and future, in Hydroxyapatite and related compounds. Brown, P.W.; and Constantz, B.(eds). CRC press. (1994): 45-72.

18. Yoshimura, M.; Suda, H.; Okamoto, K.; Ioku, K. Hydrothermal synthesis of biocompatible whiskers. J. Mater. Sci. 29 (1994): 3399-3402.

19. Asaoka, N.; Suda, H.; and Yoshimuro, M. Preparation of hydroxyapatite whiskers by hydrothermal method. Nippon Kagaku Kaishi. 1 (1995): 25-29.

20. Suchanek, W.; Suda, H.; Yashima, M.; Kakihana, M.; and Yoshimuro, M. Biocompatible whiskers with controlled morphology and stoichiometry. J. Mater. Res. 10,3 (1995): 521-529.

21. Pongkao D.; and Yoshimura,M. Hydrothermal synthesis of hydroxyapatite from bio-bearing calcium phosphate (BCP). Transaction of the 15th symposium on apatite JAPAN: Tokyo (1999): 27-28.

22. Pongkao, D.; and Yoshimura, M. Optimization for stoichiometric and morphology controlled calcium phosphate crystals from bio-bearing calcium phosphate (BCP) under hydrothermal conditions. Particles 2001, USA: Florida (2001): 92-93.

23. Jiang, G.; and Shi, D. Coating of hydroxyapatite on highly porous $Al_2O_3$ substrate for bone substituates. Wiley (1997): 77-81.

24. Van Dijk, K. H.; Schaeken, G.; J Wolke,. G. G.; and Jansen, J. A. Influence of annealing temperature on RF-magnetron sputtered calcium phosphate coating. Biomaterials, 17 (1998): 159-163.

25. Ohtsuka, Y.; and Matsuara, M. Formation of hydroxyapatite coating on pure titanium substrate by ion-beam dynamic mixing. Surf. Coat. Tech. 65 (1994): 224-230.

26. Baszkiewicz, J.; Krupa, D.; Kosubowski, J. A.; Rajchel, B.; Mitura, M.; Barcz, A.; Slosarczyk, A.; Paskiewicz, Z.; and Puff, Z. Influence of the Ca- and P-enriched oxide layers produced on titanium and the Ti6Al4V alloy by IBAD method upon the corrosion resistance of these materials. VACUUM. 70 (2003): 163-167.

27. Wang, C. K.; and Chern Lin, J. H. Structure characterization of pulse laser deposition hydroxyapatite film on titanium substrate. Biomaterials. 18 (1997): 1331-1338.

28. Damodaram, R.; Moudgil, B. M. Electrophoretic deposition of calcium phosphate from non-aqueous media. Colloid. Surf. 80 (1993): 191-195.

29. Shirkhanzadeh, M. Calcium phosphate coating prepared by electrocrystallization from aqueous electrrolyte. J. Mater. Sci. Med. 6 (1995): 90-93.

30. Lu, Y. P.; Li, M. S.; Li, S. T.; Wang, Z. G.; and Zhu, R. F.; Plasma-sprayed hydroxyapatite + titania composite bond coat for hydroxyapatite coating on titanium substrate. Biomaterials. 25 (2004): 4393-4403.

31. Zhang, J. M.; Lin, C. J.; Feng, Z. D.; and Tian, Z. W. Mechanistic studies of electrodeposition for bioceramic coating of calcium phosphate by an in situ pH, microsensor technique. J. O. Electroanal. Chem. 452 (1998): 235-240.

32. Kuo, M. C.; and Yen, S . K. The process of electrochemical deposited hydroxyapatite coating on biomedical titanium at room temperature. Mat. Sci. Eng. 20 (2002): 153-160.

33. Yen, S. K.; and Lin, C. M. Cathodic reactions of electrolytic hydroxyapatite coating on pure titanium. Mat. Chem. And Phy. 77 (2002): 70-76.

34. Monma, H. Electrochemical deposition of calcium deficient apatite on stainless steel substrate. J. O. Cer. Soc. Jap. 101,7 (1993): 737-739.

35. Bertoni,E.; Bigi, A.; Cojazzi, G.; Gandolfi, M.; Pansavolta, S.; and Roveri, N. Nanocrystal of magnesia and fluoride substituted hydroxyapatite. J. O. Inor. Biochem. 72 (1998): 29-35.

36. Kim, H. M.; Miyazaki, T.; Kokubo, T.; and Nakamura, T. Revised simulated body fluid. Eng. Mat. 192,195 (2001): 47-50.

37. Elliott, J.C. Structurer and chemistry of the apatite and other calcium orthophosphates, ELSEVIER, (1994).

38. Faulkner, J. R. J. Chem. Ecud. 60 (1983): 262.

39. Faulkner, J. R. Physical methods in modern chemical analysis. by Kuwana, T. (eds). pp. 137-248. New York: Academic, 1983.

40. Bard, A. J.; and Faulkner, L. R. Electrochemical methods: fundamentals and applications. 2ND ed. WILEY, 2001.

41. Zhitomirsky, I. Electrophoretic and electrolytic deposition of cermics coating on carbon fibers. J. Europ. Ceram. Soc. 18 (1998): 849-856.

42. Zhitomirsky, I. Ceramic films using cathodic electrodeposition[Online]. Available from: http://www.tms.org/pubs/journals/JOM/0001/Zhitomirsky

43. Zhitomirsky, I.; and Gal-or, L. Electrochemical coating. Intermetallic and ceramic coatings. by Dahotre, N. B.; and Sudarshan, T. S.(eds). pp. 83-145. New York: Marcel Dekker, 1999.

44. Shirkhanzadeh, M. Bioactive calcium phosphate coating prepared by electrodeposition. J. Mater. Sci. Lett. 10 (1991): 1415-1417.

45. Shirkhanzadeh, M.; Azadegan, M.; Stack, V.; and Schreyer, S. Fabrication of pure hydroxyapatie and fluoridated-hydroxyapatite coating by electrocrystallization. Mater. Lett. 18 (1994): 211-214.

46. Cells and electrodes for use with potentiostats[Online]. Available from: http://www.consultrsr.com

47. Vijayaraghavan, T. V.; and Bensalem, A. Electrodepositionof apatite coating on pure titanium and titanium alloys. J. Mater. Sci. Lett. 13 (1994): 1782-1785.

48. Ban, S.; and Maruno, S. Effect of temperature on electrochemical deposition of calcium phosphate coating in a simulated body fluid. Biomaterials. 16,13 (1995): 977-981.

49. Ishizawa, H.; and Ogino, M. Formation and characterization of anodic titanium oxide films containing Ca and P. J. Biomed. Mat. Res. 29 (1995): 65-72.

50. Redepenning, J.; Schlessinger, T.; Burnham, S.; Lippiello, L.; and Miyano, J. Characterization of electrolytically prepared brushite and hydroxyapatite coatings on orthopedic alloys. J. Biomed. Mat. Res. 30 (1996): 287-294.

51. Shidhar, T. M.; Arumugam, T. K.; Rajeswari, S.; and Subbaiyan, M. Electrochemical behavior of hydroxyapatite-coated stainless steel implants. J. Mat. Sci. Let. 16 (1997): 1964-1966.

52. Ban, S.; and Maruno, S. Morphology and microstructure of electrochemical deposited calcium phosphates in a modified simulated body fluid. Biomaterials. 19 (1998): 1245-1253.

53. Han, Y.; Xu, K.; and Lu, J. Morphology and composition of hydroxyapatite coatings prepared by hydrothermal treatment on electrodeposited brushite coatings. J. Mat. Sci: Mat. Med. 10 (1999): 243-248.

54. Manso, M.; Jimenez, C.; Morant, C.; Herrero, P.; and Martinez-Duart, J. M. Electrodeposition of hydroxyapatite coatings in basic conditions. Biomaterials. 21 (2000): 1755-1761.

55. Kannan, S.; Balamurugan, A.; and Rajeswari, S. Development of calcium phosphate coatings on type 316L SS and their *in vitro* response. Trends Biomater. Artif. Organs. 16,1 (2002): 8-11.

56. Sridhar, T. M.; Mudali, U. K.; and Subbaiyan, M. Preparation and characterization of electrophoretically deposited hydroxyapatite coating on type 316L stainless steel. Corro. Sci. 45 (2003): 237-252.

# APPENDICES

# Appendix A

Brushite

## History

- Authors: *Moore*
- Discovery date: *1864*
- Town of origin: *ILE AVES, MER DES CARAIBES.*

## Chemical properties

- Chemical class: *PHOSPHATES*
- Subclass: *Hydrous phosphates, arseniates, vanadates without foreign anion.*
- Chemical formula: $CaHPO_4 \bullet 2H_2O$

## Crystallographical properties

- Crystalline system: *MONOCLINIQUE*
- a: *5.83*
- b: *15.19*
- c: *6.26*
- Beta: *116.47*
- Z : Formula units per unit cell: *4*

## Optical properties

- Optical and misc. Properties: *Transparent – Translucide*
- Refractive index: *from 1.53 to 1.55*
- Axial angel 2V: *86* $^{o}$

**Physical properties**

- Morphology: *PRISMATIQUE; TABULAIRE; FOLIE; TERREUX; PULVERULENT; MASSIF; RENIFORME; FEUILLETE; EFFLORESCENCE; LAMELLAIRE; ACICULAIRE; NODULAIRE.*
- Hardness: *2.50*
- Density: *from 2.32 to 2.33*
- Color: *Colorless; Yellow; Ivory; yellowish white*
- Luster: *Vitreous, Nacreous*
- Streak: *White*
- Cleavage: *OUI*

# Appendix B

## Monetite

### History

- Authors: *SHEPARD*
- Discovery date: *1882*
- Town of origin: *ILES MONETA et MONA, MAYEGUEZ*
- Country of origin: *PORTO-RICO*

### Chemical properties

- Chemical class: *PHOSPHATES*
- Subclass: *Anhydrous phosphates, arseniates, vanadates without foreign anion*
- Chemical formula: $CaHPO_4$

### Crystallographical properties

- Crystalline system: *TRICLINIQUE*
- a: *6.98*
- b: *8.57*
- c: *6.63*
- Alpha: *93.90*
- Beta: *91.50*
- Gamma: *127.63*
- Z: Formula units per unit cell: *4*

### Optical properties

- Optical and misc. properties: *Fragile: cassant – Transparent – Translucide*
- Refractive index: *From 1.58 to 1.64*
- Axial angel 2V: *MODERE A LARGE*

**Physical properties**

- Morphology: *AGGREGATE; MASSIF; RHOMBOEDRIQUE; STALACTITIQUE; CROUTE*
- Hardness: *3.50*
- Density: *From 2.92 to 2.93*
- Color: *Colorless; white; pale yellow; yellowish; pale yellowish white*
- Luster: *Vitreous*
- Streak: *White*
- Break: *Irregular*
- Cleavage: *OUI*

# Appendix C

## Reference Electrode

*The Ag/AgCl Reference Electrode*[46]

The silver/silver chloride or Ag/AgCl reference electrode is many electrochemists' reference electrode of choice. It is easily and cheaply prepared. It is stable, and quite robust. It is sometimes referred to as "SSCE" (Silver/Silver Chloride Electrode) but that abbreviation has been used for sodium saturated calomel electrode also.

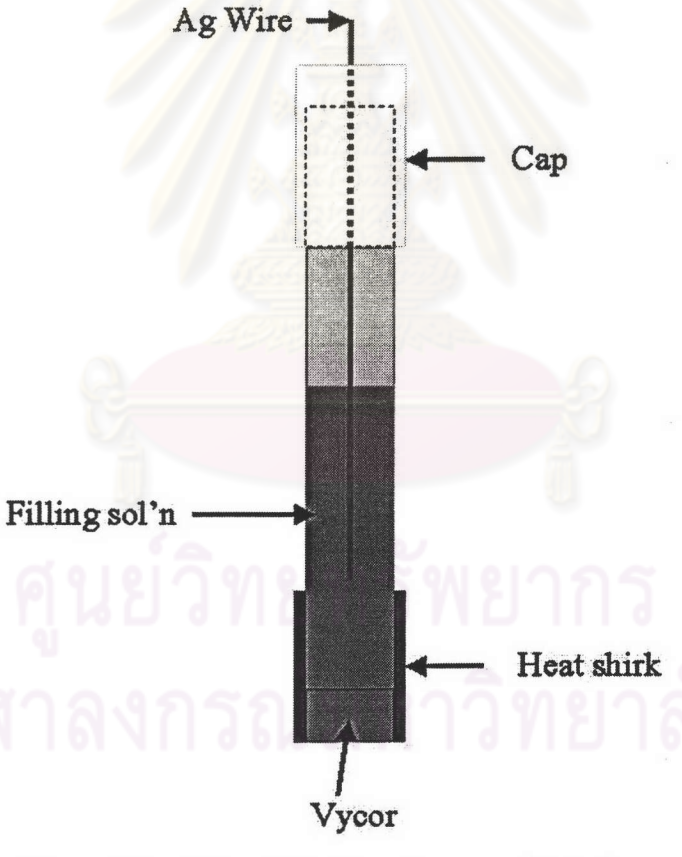



**Figure** Silver/Silver Chloride reference electrode.

The above figure shows an easily constructed Ag/AgCl reference electrode. The body of the electrode is made from 4-mm glass tube. *Vycor*® porous glass is available in 4-mm diameter rod and serves as the ionicly conducting electrical pathway between the inside of the reference electrode and the bulk of your cell. It has low electrical

resistance (under 10 k-ohm for the common filling solutions) and a modest leak rate. The electrical resistance of the reference electrode 'frit' is an important factor in determining the stability and speed of the potentiostat in actual use. The leak rate may be important because of possible contamination of the solution by the reference electrode filling solution and vice versa.

The *Vycor*® frit (about 1/8" long) is attached to the glass tube by 'heat shrink' Teflon tubing. The heat-shrink tubing should be cut flush with the end of the Vycor frit to prevent trapping any air bubbles. The cap is conveniently made out of scrap Teflon or plastic cap or protector made to fit 5/32" OD tubing. It should be snug, but easily removable for replenishing the filling solution.

Filling solution

A variety of filling solution can be used. The most commonly used are saturated KCl or 3.5 M-KCl. KCl has an uncanny ability to 'creep' and form a crusty layer of solid KCl where the solution is exposed to the air. If perchlorate electrolytes are to be studied, $KClO_4$ may precipitate in the pores of the frit, and for these electrolytes NaCl (either saturated or 3M) is preferred. Normally, many chemists routinely uses NaCl filled electrodes, but has used LiCl in special instances.

Saturated solutions of KCl or NaCl have the advantage that the concentration is reproducible even if the temperature changes and are immune to the effects of water evaporation. However, the solid salts harden into an impenetrable block which may lead to ahigh impedance electrode. A "nearly saturated" solution (3.5M KCl or 3M NaCl) can change concentration due to evaporation.

Enemies of the Ag/AgCl electrodes

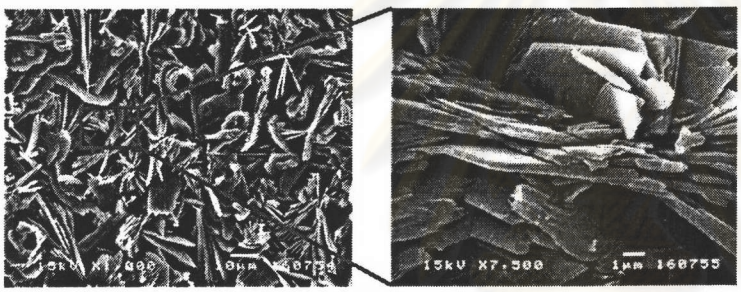
- **Light**. UV light decomposes AgCl to give silver(0) which gives the electrode a black appearance. Normal lab fluorescent lights are OK, but don't store electrodes near the window.

- **Base.** $Ag_2O$ or AgOH will form if the $[OH^-]$ is on the order of 0.1 M and the electrode potential will be a mixed $Ag/AgCl/Ag_2O$ potential and will depend on the pH. $Ag_2O$ will also form in a pores of the frit used.
- **$NH_3$ Buffers.** $NH_3$ will complex silvers and will dissolve AgCl.
- **Sulfide.** Silver sulfide is quite insoluble.

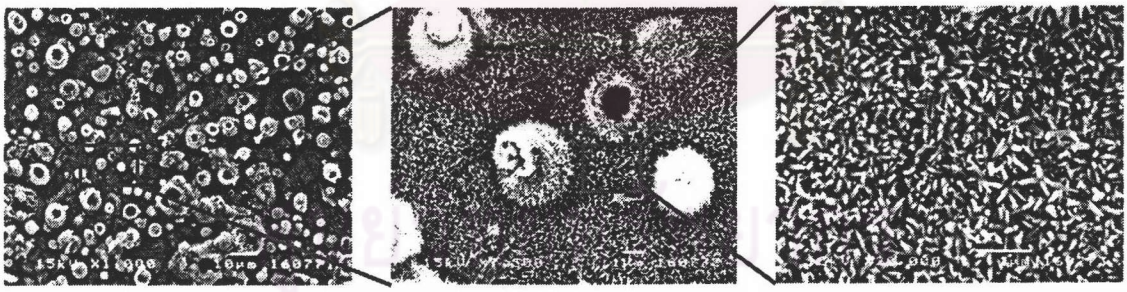
# Appendix D

The 3-min film gained from MCPM based aqueous electrolyte after soaking in R-SBF for 2, 4, and 8 hours respectively.


2 Hour



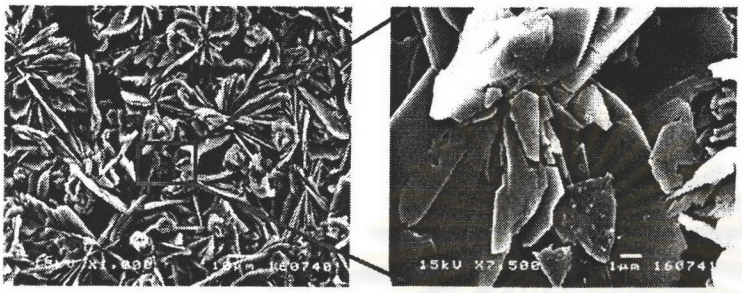

4 Hour



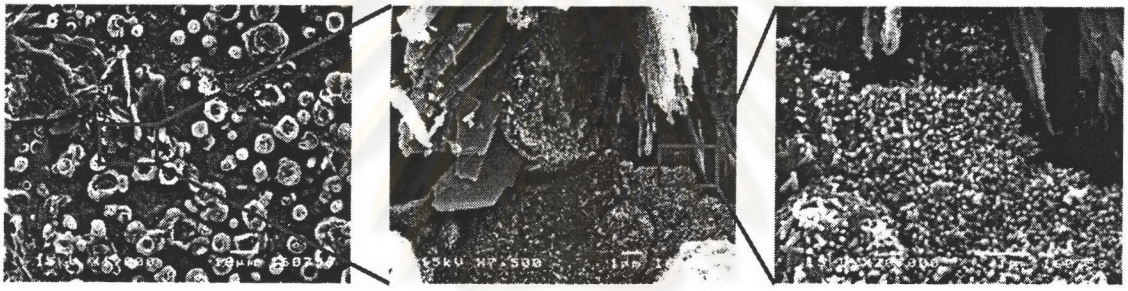
8 Hour

# Appendix E

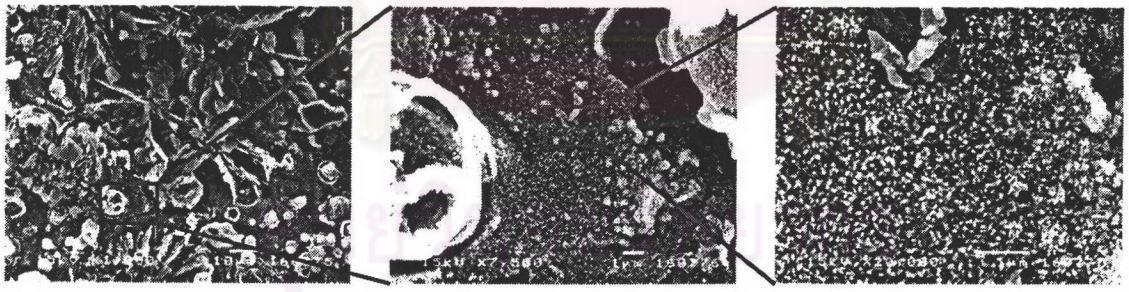
The 5-min film gained from MCPM based aqueous electrolyte after soaking in R-SBF for 2, 4, and 8 hours respectively.



2 Hour



4 Hour



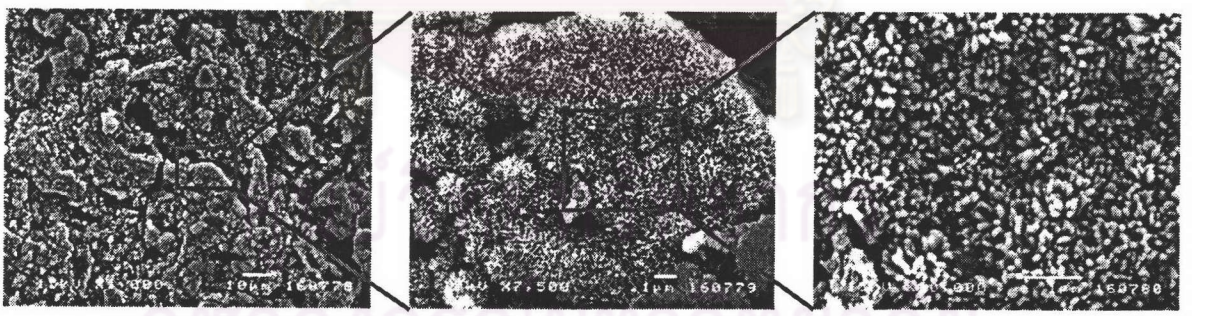
8 Hour

# Appendix F

The 3-min films gained from MCPM based aqueous with ions adding electrolyte after soaking in R-SBF for 2, 4, and 8 hours respectively.

2 Hour

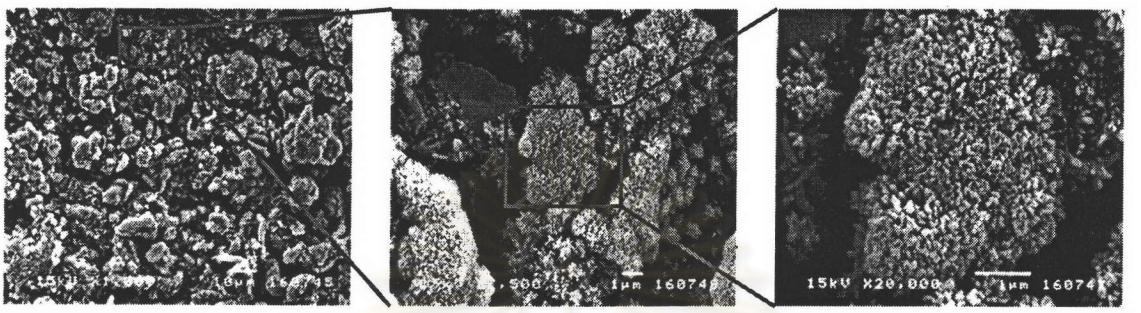
4 Hour



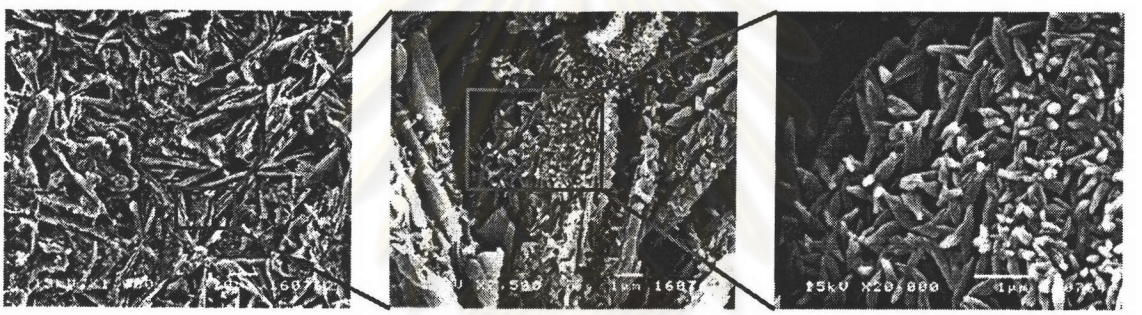
8 Hour

# Appendix G

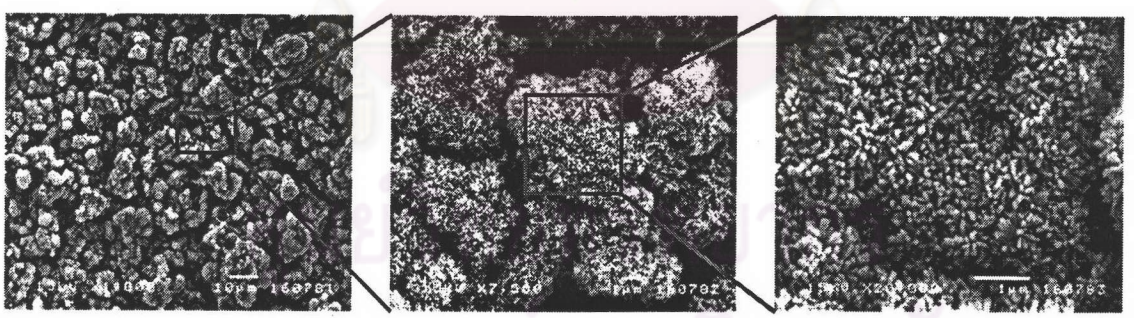
The 5-min films gained from MCPM based aqueous with ions adding electrolyte after soaking in R-SBF for 2, 4, and 8 hours respectively.



2 Hour



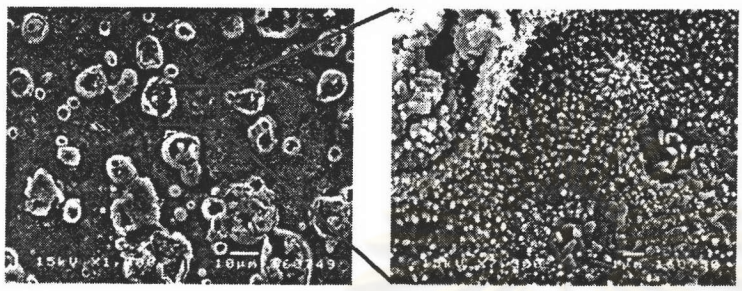
4 Hour



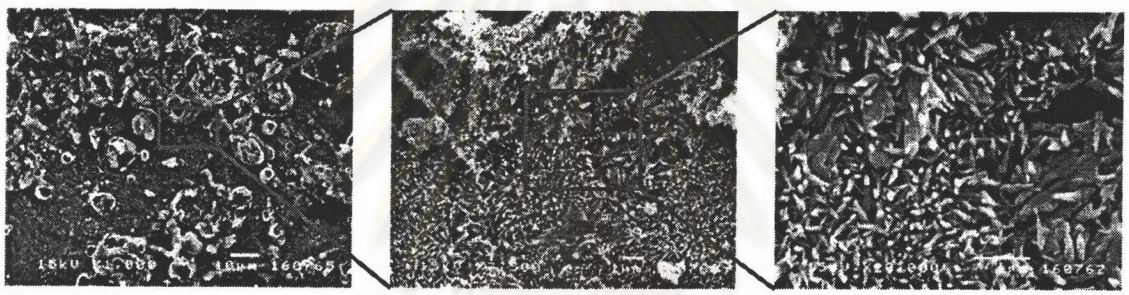
8 Hour

# Appendix H

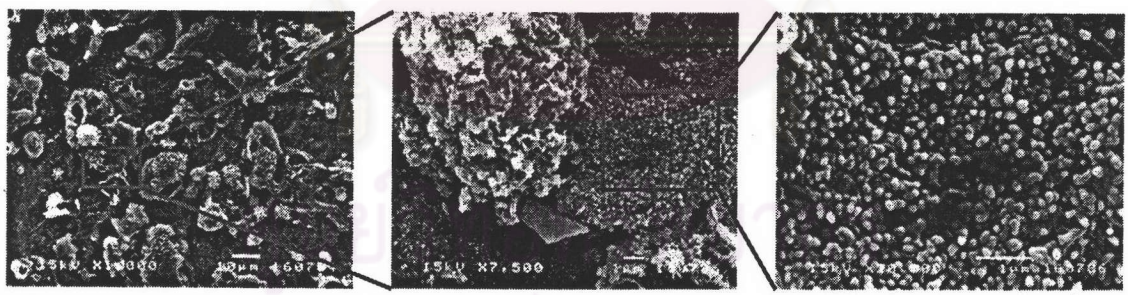
The 3-min films gained from MCPM based 20% V/V ethanol electrolyte after soaking in R-SBF for 2, 4, and 8 hours respectively.



2 Hour



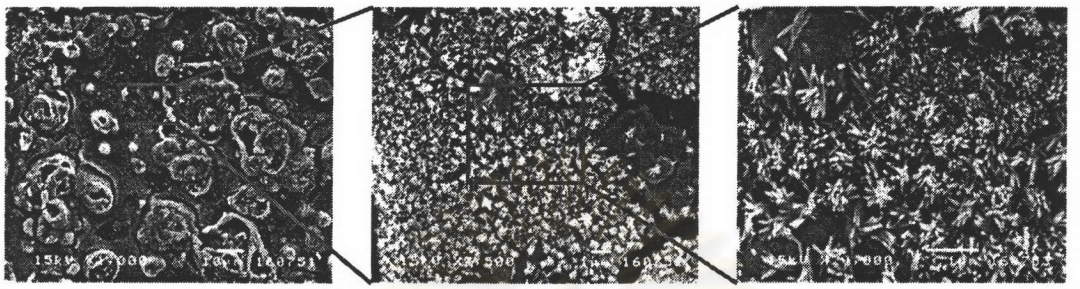
4 Hour



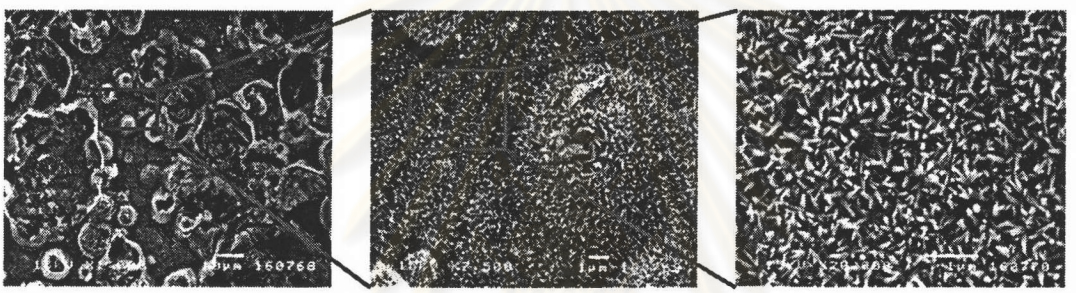
8 Hour

# Appendix I

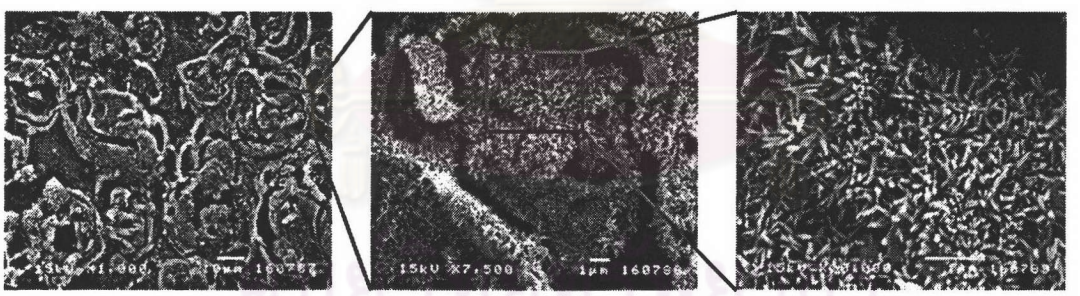
The 5-min films gained from MCPM based 20% V/V ethanol electrolyte after soaking in R-SBF for 2, 4, and 8 hours respectively.



2 Hour



4 Hour



8 Hour

# Appendix J

Electrolytes pH parameter

| | | | |
|---|---|---|---|
| MCPM solution= | | 2.81 | |
| | - current | = | 2.83 (3 min) |
| | | = | 2.84 (5 min) |
| DCPD solution= | | 1.95 | |
| | - current | = | 1.98 (7 min) |
| | | = | 1.99 (10 min) |

MCPM with Ions

| | | | |
|---|---|---|---|
| Formula I | solution= | | 2.71 |
| | - current | = | 2.73 (3 min) |
| | | = | 2.74 (5 min) |
| Formula II | solution= | | 2.84 |
| | - current | = | 2.86 (3 min) |
| | | = | 2.86 (5 min) |
| Formula III | solution= | | 2.95 |
| | - current | = | 2.97 (3 min) |
| | | = | 2.99 (5 min) |
| MCPM 20% V/V ethanol | | = | 2.62 |
| | - current | = | 2.63 (3 min) |
| | | = | 2.63 (5 min) |

# Appendix K

The chemicals and their purities and amounts to prepare 1000mL of C-SBF, R-SBF, and R-SBF in this study.

| Reagent | Purity | Amount | | |
|---|---|---|---|---|
| | | C-SBF (From Kim et.al) | R-SBF (from Kim et.al.) | R-SBF (in this study) |
| NaCl | 99.5 % | 8.036 g | 5.403 g | 5.4031 g |
| $NaHCO_3$ | 99.7 % | 0.352 g | 0.736 g | 0.7360 g |
| $Na_2CO_3$ | 99.9 % | - | 2.036 g | 2.0359 g |
| KCl | 99.5 % | 0.225 g | 0.225 g | 0.2251 g |
| $K_2HPO_4 \bullet 3H_2O$ | 99.0 % | 0.238 g | 0.238 g | 0.2381 g |
| $MgCl_2 \bullet H_2O$ | 98.0 % | 0.311 g | 0.311 g | 0.3112 g |
| HEPES** | 99.5 % | - | 11.928 g | 11.9281 g |
| $CaCl_2$ | 94.0 % | 0.293 g | 0.293 g | 0.2930 g |
| $Na_2SO_4$ | 99.0 % | 0.072 g | 0.072 g | 0.0720 g |
| 1M-NaOH | - | - | 1.5 mL | 1.5 mL |
| 1 M-HCl | - | 20 mL | - | - |
| TRIS♣ | 99.0 % | 6.118 g | - | - |

The pH parameter of R-SBF in this study was 7.42.



** HEPES = 2-(4-(2-hydroxyethyl-1-piperazinyl)ethane sulfonic acid)

♣ TRIS = tris-hydroxymethyl aminomethan.

# Appendix L

ASTM D-2197

Designation: D 2197 – 98 (Reapproved 2002)

# Standard Test Method for Adhesion of Organic Coatings by Scrape Adhesion[1]

This standard is issued under the fixed designation D 2197; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

*This method has been approved for use by agencies of the Department of Defense.*

## 1. Scope

1.1 This test method covers the determination of the adhesion of organic coatings such as paint, varnish, and lacquer when applied to smooth, flat (planar) panel surfaces.

1.2 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 609 Practice for Preparation of Cold-Rolled Steel Panels for Testing Paint, Varnish, Conversion Coatings, and Related Coating Products[2]

D 823 Practices for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels[2]

D 1005 Test Method for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers[2]

D 1186 Test Methods for Nondestructive Measurement of Dry-Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base[2]

D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base[2]

## 3. Summary of Test Method

3.1 The materials under test are applied at uniform thickness to flat panels, usually sheet metal of uniform surface texture. After drying, the adhesion is determined by pushing the panels beneath a rounded stylus or loop that is loaded in increasing amounts until the coating is removed from the substrate surface.

## 4. Significance and Use

4.1 Coatings to perform satisfactorily must adhere to the substrates on which they are applied. This test method has been found useful in differentiating the degree of adhesion of coatings to substrates. It is most useful in providing relative ratings for a series of coated panels exhibiting significant differences in adhesion.

4.2 Studies performed in a laboratory using the loop stylus specified in the previous edition showed meaningful adhesion data were impossible when loads of 10 to 20 kg were required to break the surface of a solvent based coating. The chrome plated loop stylus chattered and skipped across the coating surface when loads of this magnitude were required. Similar meaningless data were obtained when powder coatings were tested that required more than 10 kg to break the surface. Therefore, testing under these conditions is not applicable.

## 5. Apparatus

5.1 *Application Equipment*, as described in Practices D 823.

5.2 *Film-Thickness Measuring Apparatus*, as described in Test Methods D 1005, D 1186, or D 1400.

5.3 *Balanced Beam, Scrape Adhesion Tester* (Figs. 1 and 2), consisting of a balanced beam to which is secured a platform for supporting weights, and a rod at an angle of 45° that holds the scraping loop. The rod shall be set so that the scraping loop contacts test surfaces directly below the weights. The loop shall be 1/16-in. (1.6-mm) diameter rod, bent into a "U" shape with an outside radius of 0.128 ±0.002 in. (3.25 ±0.05 mm) and hardened to Rockwell HRC 56 to 58, and shall be a smooth finish. The loop can be either chromium plated, nickel plated, or heat treated polished steel, as agreed upon between the purchaser and the supplier. These testers are adjustable to accommodate flat, metallic, and nonmetallic specimens to 0.5-in. (12-mm) thick and 4 to 16 in. (100 to 400 mm) wide and long; the specimen should be at least ½-in. (12-mm) wide.

## 6. Preparation of Specimens

6.1 Apply the materials under test to panels of the composition and surface condition on which it is desired to determine adhesion. The panel material (6.1.1), surface preparation, thickness, and number of coats shall be specified or agreed

---

[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films. Current edition approved Feb. 10, 1998. Published October 1998. Originally published as D 2197 – 63 T. Last previous edition D 2197 – 86 (1991)$^{\epsilon 1}$.

[2] *Annual Book of ASTM Standards*, Vol 06.01.

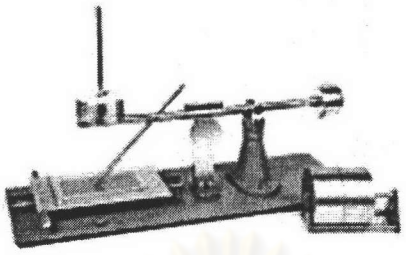


FIG. 1 Balanced-Beam Scrape-Adhesion Tester

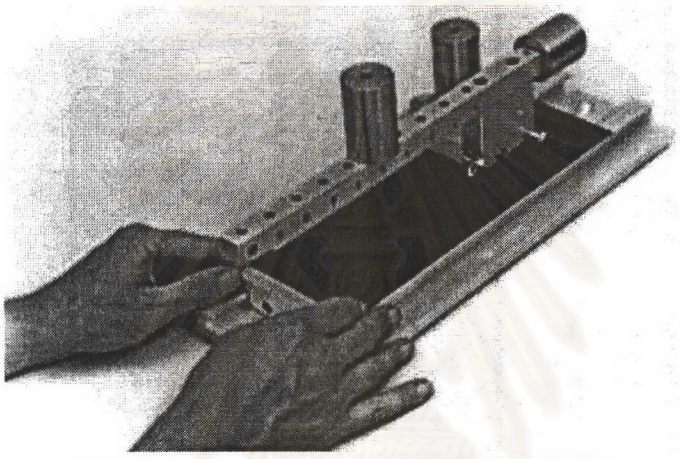


FIG. 2 Balanced Beam Scrape Adhesion and Mar Tester

upon by the seller and the purchaser. Apply uniform coatings and air dry or bake under conditions of humidity and temperature mutually agreeable to the seller and purchaser. Either mask the panel or remove material after application, so that ½ in. (13 mm) at one end of the panel is uncoated.

6.1.1 The surface of the panel must be hard enough that it will not be damaged by the scraping loop. If no panel material is specified, use 0.032-in. (0.8-mm) cold-rolled carbon steel prepared in accordance with Methods B or C of Practice D 609.

## 7. Conditioning and Number of Tests

7.1 Condition the test panels for at least 48 h at 73.5 ±3.5°F (23 ±2°C) and 50 ±5 % relative humidity, and test in the same environment, or immediately on removal therefrom, unless otherwise specified or agreed by the seller and the purchaser. Test at least two replicate specimens of each material.

## 8. Procedure

8.1 When using the instrument shown in Fig. 1, level the base plate of the apparatus and place it so that the weight holder is toward the operator. This places the beam release on the operator's right and allows freedom to move the test specimen manually under the weighted scraping element (loop). Adjust the main bearing support so that the beam is balanced in the horizontal plane when the loop is just touching the specimen surface.

8.2 Raise the beam and lock it. Wipe the loop with clean cloth or chamois. Place a test panel on the sliding platform so that it may be moved away from the operator and the uncoated portion is toward the main beam support. Place weights on the weight support using an initial amount that is estimated to be appropriate for the particular coating. Carefully lower the beam until the loop rests on the uncoated portion of the test specimen and the full load is applied, then slowly (1 to 2 s/in.) push the sliding platform away from the operator for a distance of at least 3 in. (75 mm). If the coating is removed, continue the testing, using successively smaller loads (0.5-kg increments) until the coating is not removed. If the coating is not removed by the initial scrape, continue the testing, using successively larger loads (0.5-kg increments) until the coating is removed or until the maximum load of 10 kg has been applied. Use a new area of the test surface each time a scrape is made.

8.3 When the critical load has been approximately located, repeat the test five times at each of three loadings: above, below, and at the load determined in the first trial. Apply the different loads in random fashion so that all scrapes at one load are not made in succession or on one panel.

8.3.1 Periodically examine the loop to ensure that the original smooth surface is intact. If the contacting surface is worn, reverse the loop. When both sides are worn, replace with a new loop.

8.4 For each applied load, tabulate the number of times the coating was removed or adhered. The load where the scrape results change from mainly adhering to mainly removed, ignoring the first ½ in. (13 mm) of the scratch if the coating

was removed, is the adhesion failure end point.

## 9. Report

9.1 Report the following information:

9.1.1 Load in kilograms at the adhesion failure end point,

9.1.2 Panel material and surface preparation,

9.1.3 "U" shape loop surface finish,

9.1.4 Dry-film thickness, and

9.1.5 Any deviation from the specified procedure.

## 10. Precision

10.1 *Correlation*—This method was developed when correlation with other methods of assessing adhesion was considered to be of equal importance to the agreement between results obtained in the same or different laboratories. It was established that when materials differing widely in hardness and adhesion were evaluated by a number of experienced personnel in several laboratories, the adhesion results obtained using this method correlated well with those obtained with several other methods.

10.2 *Precision*—If sufficient cooperators can be obtained, an interlaboratory study will be conducted to establish precision.

10.3 *Bias*—No information can be provided on the bias of this test method for measuring adhesion because no material having an accepted reference value is available.

## 11. Keywords

11.1 adhesion; balanced-beam scrape adhesion tester; scrape

# BIOGRAPHY

Miss Achariya Rakngarm was born on the 3rd of May 1978, in Chiangmai. After graduateing with a Bachelor's degree in Ceramic Engineering from the Department of Ceramic Engineering, Faculty of Engineer, Suranaree University of Technology in May 2000, she continued a further study in Master's Degree in the field of Ceramic Technology at Chulalongkorn University and graduated in August 2004.