A Study of Thermal Spray Coating on Artificial Knee Joints

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Abstract: Thermal spray coating technology is used worldwide in many industrial applications, but the application of thermal spray biocompatible coatings on the titanium alloy surface in the biomedical field as to improve the knee's functions is a relatively new area. The purpose of this study was to experimentally compare the difference of various pretreatment (ultrasonic vs. high pressure gas cleaning) and to verify the benefit of cryogenic treatment in the post-processing for titanium alloy surface coating in artificial knee joint by thermal spray coating technology. Experimental results exhibit that coating performance with ultrasonic cleaning and cryogenic treatment has been improved and verified.

[J.-C. Hsiung, J Tzeng, K. Kung,H.-S. Chen. **A Study of Thermal Spray Coating on Artificial Knee Joints.** *Life Sci J* 2013;10(2):236-241] (ISSN:1097-8135). http://www.lifesciencesite.com. 37

Keywords: Thermal spray; Artificial knee joint; Titanium alloy; Cryogenic treatment

1. Introduction

Knee joints are the largest and most important joints, which are vulnerable to various injuries and diseases. Currently there are around 20,000 cases per year for the artificial knee joint replacement surgery in Taiwan, and this surgery is fairly common in orthopedic surgery[1-3]. Knee structure is mainly composed of the femur, tibia, patella and meniscus formed and fixed by the external muscles and ligaments. Typical components commonly used in artificial knee joint contain the femoral components, tibial components, patella and tibial bearing component as shown in Figure 1[4]. Because the metallic materials have excellent mechanical properties, easy processing and stability, they have been widely used in artificial knee joint [5-10]. Due to density as close to human bones, low modulus, corrosion resistance and biocompatibility, titanium alloys are better than stainless steel and cobalt alloy and mainly used for the artificial knee joint [11-12].
Thermal spray technology encompasses a group of coating processes that provide functional surfaces to protect or improve the performance of a substrate or component. Many types and forms of materials can be thermal sprayed—which is why thermal spray is used worldwide to provide protection from corrosion, wear, and heat; to restore and repair components; and for a variety of other applications[13-14]. Compared with other industrial applications, biomedical coating is a relatively new class of applications for thermal spray coating [15-18].

In the previous published paper [19], a comprehensive overview of applications and characterization of titanium alloy surface coating in artificial knee joint by thermal spray has been discussed. The purpose of this study was to experimentally explore the thermal spray process on the coating properties of titanium alloy in artificial knee joint, and to compare the benefit of different pre-treatment and cryogenic treatment.

Figure 1. The components of an artificial knee joint.

2. Experimental Procedures

2.1 Experimental Materials

The materials used in this study were bars and plates of titanium alloys (Ti-6Al-4V). The bars were mainly used for bond strength test, and the plates were used for the surface roughness, hardness, microstructure, and abrasion tests. The properties of titanium alloy are shown in Table 1, the sample size of the bar isφ25.4mm × 30mm and the plate is 75 × 25 × 35mm, as shown in Figure 2 (a) and 2 (b).

Table 1. The material properties of titanium alloys tested.

Chemical composition	Al	V	Fe	C	N	H	O	Ti
	5.5-6.75	3.5-4.5	≦0.20	≦0.08	≦0.05	≦0.015	≦0.020	Remainder
Mechanical Properties	Tensile Strengh(MPa)		Yield Strengh(MPa)		Elongation(%)		Bending test	Hardness(HRC)
	≧895		≧825		≧10		≧25°	34°±2

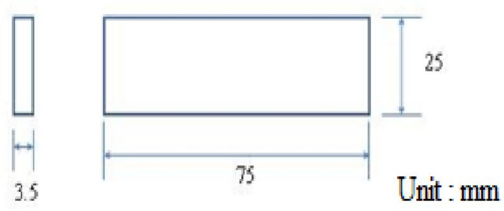

Figure 2(a) Schematic diagram of sample size for the surface roughness, hardness, microstructure and abrasion tests.

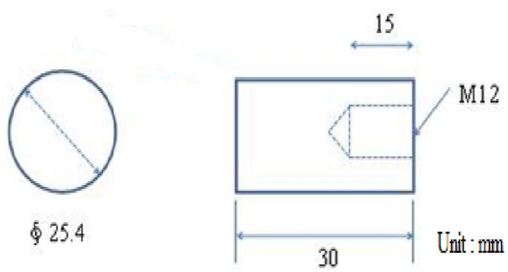

Figure 2(b) Schematic diagram of sample size for the bond test.

2.2 Thermal Spray Process and Parameters

The atmospheric plasma spray (APS) process was used in this study. Three kinds of powder materials such as alumina (Al_2O_3), zirconia (ZrO_2) and hydroxyapatite (HA) are melted respectively, and then molten particles were guided through by air to form a coating on the substrate, as shown in Figure 3. Al_2O_3 and ZrO_2 ceramic materials are biologically inert, while the HA is a bioactive ceramic material. Hydroxyapatite (HA) is calcium phosphate ceramic [$Ca_{10}(PO_4)_6(OH)_2$] that exhibits strong activity for joining to bone tissue. The equipment made by Sulzer Metro Company (Spray Gun Type: 9MB) was utilized. The thermal spray process conditions of the three coating materials were shown in Table 2. Table 2 shows the important parameters affecting the quality of the coating, which includes inert gas composition, current, voltage, powder feeding rate, spraying distance and etc. In addition, the choice of different pre-treatment and post-treatment is important to the bonding strength. The pretreatment is focused to compare the coating properties with high pressure cleaning and ultrasonic cleaning respectively; the post-treatment is to verify the benefit of cryogenic treatment which will affect the bond strength and has been mentioned in the ASTM C633 standard [20].

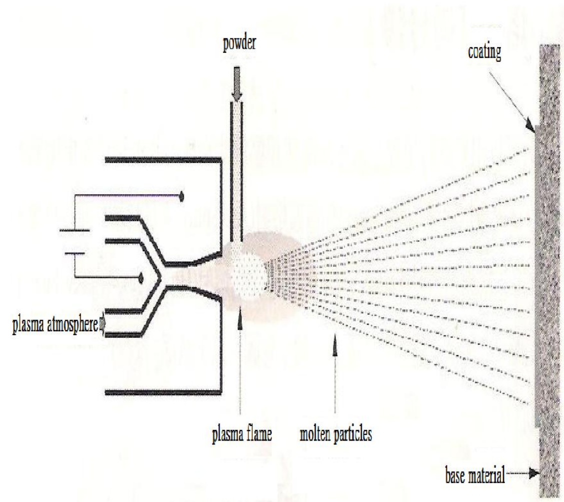

Figure 3 Schematic diagram of thermal spray coating.

Table 2 Thermal spray parameters for HA、Al_2O_3、ZrO_2 powders.

Hydroxyapatite (HA)									
Parameters	Argon (l/min)	Helium (l/min)	Current A	Voltage V	Powder rate (g/min)	Spray distance (mm)	Surface speed (75m/min)	Traverse speed (8mm)	Cooling
Setting	41	60	700	52	30	115	75	8	yes
Al_2O_3									
Parameters	Argon (l/min)	Hydrogen (l/min)	Current A	Voltage V	Powder rate (g/min)	Spray distance (mm)	Surface speed (75m/min)	Traverse speed (8mm)	Cooling
Setting	41	14	600	72	35	110	75	8	yes
ZrO_2									
Parameters	Argon (l/min)	Hydrogen (l/min)	Current A	Voltage V	Powder rate (g/min)	Spray distance (mm)	Surface speed (75m/min)	Traverse speed (8mm)	Cooling
Setting	41	9	600	68	28	120	75	8	yes

2.3 Cryogenic Treatment

Cryogenics is a branch of physics dealing with subzero or deep-cold treatment. Cryogenic treatment is now being recognized as a valuable and crucial tool for industry [21-24]. The LS-100 processor, as shown in Figure 5 was used in this study. Once the specimens were in the freezer, they will start the cooling process and should, to avoid thermal shock, bring the temperature down very slowly over a 6 hour period, to -300°F. The specimens should be held at -300°F for 10 hours, then be heated to room temperature and tempered at 300°F for 4 hours.

Figure 4. LS-100 Cryogenic Processor.

2.4 Characterization of Surface Coating

The surface coating quality of three kinds of coating materials (Al_2O_3, ZrO_2, HA) with different pre-treatment and cryogenic treatment on Ti-6Al-4V titanium alloy substrate by plasma thermal spraying was tested and characterized. These tests include surface roughness measurement, hardness test, metallographic microstructure analysis, bond strength test (ASTM C633), abrasion test (ASTM G65) [25].

3. Results and Discussion

3.1 Surface Roughness

Surface morphology of the coating can be obtained by surface roughness measurement. The central average roughness (Ra) and the ten-point average roughness (Rz) are commonly used as index of surface roughness. The results indicate that the surface roughness values (Ra, Rz) for Al_2O_3, ZrO_2 and HA coating by ultrasonic cleaning are larger than those by high pressure air cleaning. General speaking, it is more easy to form adhesive joint with higher surface roughness.

Table 3 Surface roughness measurements of Ti-6Al-4V substrate with ZrO_2, Al_2O_3 and HA coating.

	Ti-6Al-4V (unit: μm)		
Powder / Cleaning	Al_2O_3	ZrO_2	HA
High Pressure Air	Ra:5.30 Rz:47.4	Ra:8.30 Rz:67.9	Ra:7.20 Rz:56.9
Ultrasonic	Ra:5.47 Rz:53.4	Ra:8.73 Rz:73.7	Ra:7.97 Rz:66.7

3.2 Hardness

The micro-hardness test method (Vickers Hardness) was used to measure the hardness of the coating layers (at different locations) without and with cryogenic treatment. The results are shown in Tables 4 and 5. These results indicate that the average micro-hardness values of ZrO_2, Al_2O_3 and HA without cryogenic treatment are lower and the hardness values are not uniform through the coating thickness when compared with those hardness values with cryogenic treatment.

Table 4 Micro-hardness test results of three coating materials without cryogenic treatment.

Location / Coating	Outside	Middle	Inside	Average
ZrO_2	472	364.2	382.8	406.3
Al_2O_3	427	447	427	433.7
HA	394	388	311.3	364.4

Table 5 Micro-hardness test results of three coating materials with cryogenic treatment.

Location / Coating	Outside	Middle	Inside	Average
ZrO_2	472	472.2	472.2	472.3
Al_2O_3	467	470	469	468.7
HA	349	347.4	370	355.5

3.3 Metallurgical Microstructure

The thermal spray coating may result in many defects within the coating because of improper process control. Through metallographic microstructure analysis, the quality of the thermal spray coating can be verified. The defects of three different coatings were shown in Table 6. This table shows that three kinds of coatings are free of obvious cracks, interface contamination, not molten particles, clusters of oxides and strip oxides, but with the phenomenon of layer spalling and interface separation. As shown in Figures 7(a) and 7(b), the defects in HA coating by high pressure air cleaning are obvious when compared with those coating by ultrasonic cleaning. In addition, the defects of HA coating with cryogenic treatments are even less as shown in Figure 7(c). There have the same observations for ZrO_2 and Al_2O_3, as shown in Figures 7 (d)~(i).

Table 6 Coating defects of three different coating materials.

Specimen	ZrO_2	Al_2O_3	HA
Crack	N	N	N
Coating spalling	Y	Y	Y
Interface contamination	N	N	N
Interface separation	Y	Y	Y
Not molten particles	N	N	N
Holes	Y	Y	Y
Clusters of oxides	N	N	N
Strip oxides	N	N	N

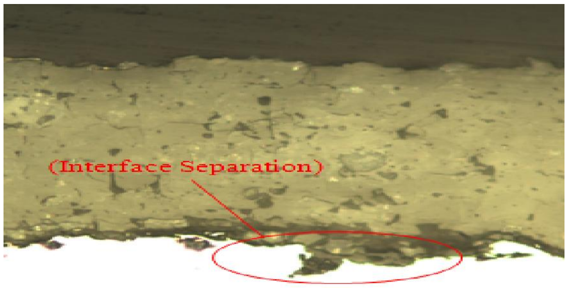

Figure 7(a) HA coating with high pressure air cleaning, magnification 200X.

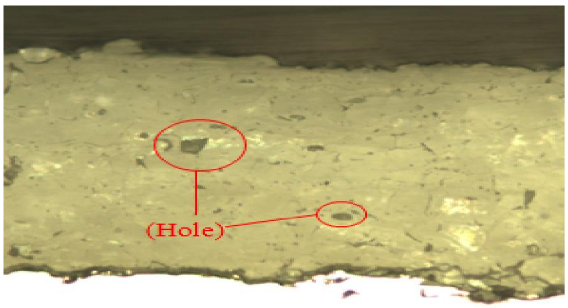

Figure 7(b) HA coating with ultrasonic cleaning, magnification 200X.

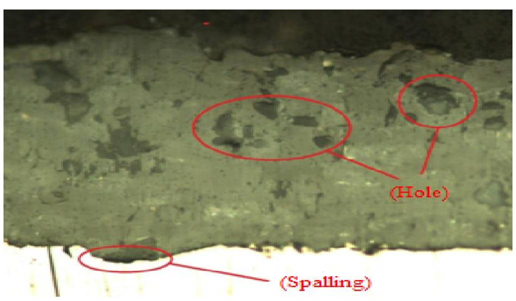

Figure 7(c) HA coating with cryogenic treatment, magnification 200X.

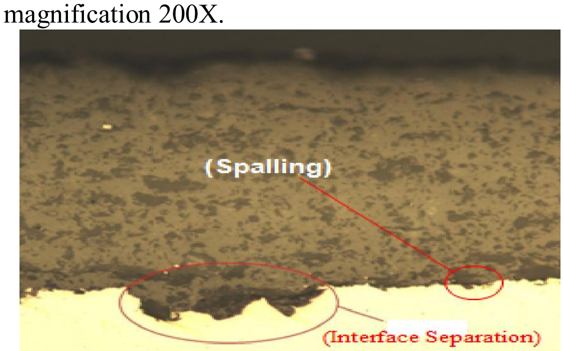

Figure 7(d) Al_2O_3 coating with high pressure air cleaning, magnification 200X.

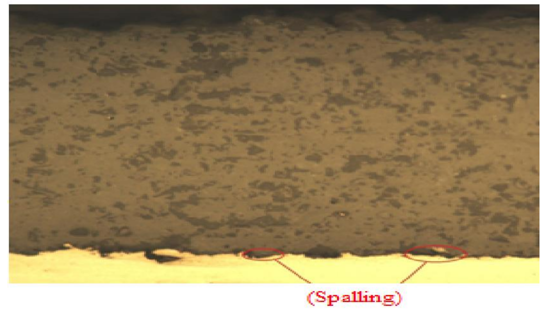

Figure 7(e) Al_2O_3 coating with ultrasonic cleaning, magnification 200X.

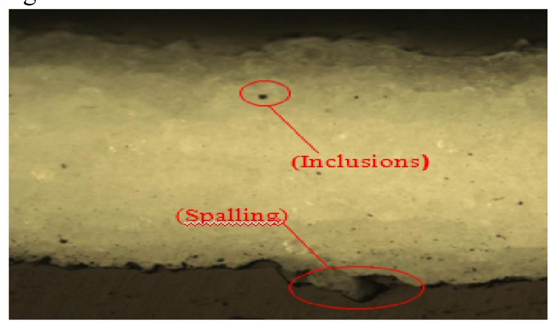

Figure 7(f) Al_2O_3 coating with cryogenic treatment, magnification 200X.

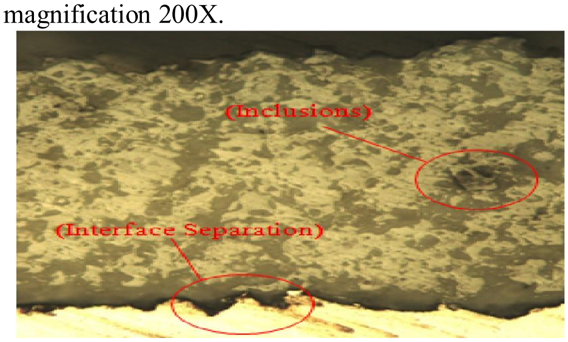

Figure 7(g) ZrO_2 coating with high pressure air cleaning, magnification 200X.

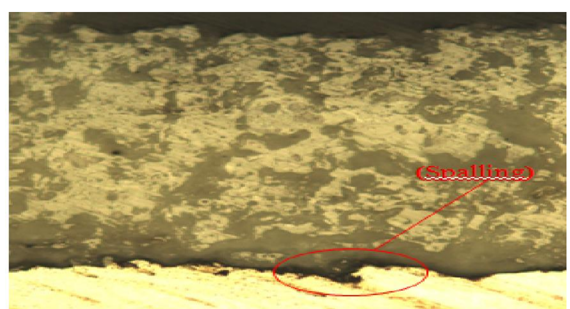

Figure 7(h) ZrO_2 coating with ultrasonic cleaning, magnification 200X.

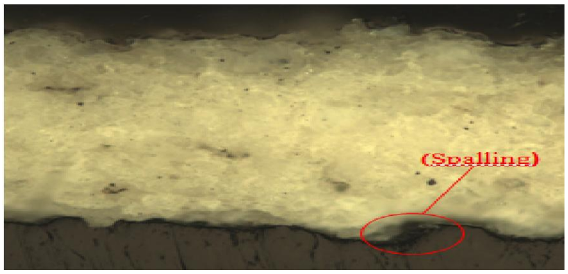

Figure 7(i) ZrO_2 coating with high pressure air cleaning, magnification 200X.

3.4 Bond Strength

The main purpose of the test is to understand the adhesion between coating and substrate. The tensile test is commonly used to evaluate the bond strength per ASTM C633 standard method. As shown in Table 7, the bond strengths with high pressure air cleaning are lower when compared with those with ultrasonic cleaning, and the bond strengths with cryogenic treatment are better than those without cryogenic treatment.

Table 7. Bond strength test results with different pre-treatment and cryogenic treatment.

Coating	Bond Strength (MPa)			
	Without Cryogenic Treatment		With Cryogenic Treatment	
	Ultrasonic	High Pressure Air	Ultrasonic	High Pressure Air
HA	26.56	18.91	36.65	29.30
Al_2O_3	45.96	37.83	38.12	49.10
ZrO_2	40.57	37.93	44.79	41.94

3.5 Abrasion Resistance

This test is a way of abrasive particle wear with rubber wheels and round quartz. As shown in Table 8, the abrasion resistances with high pressure air cleaning are lower when compared with those with ultrasonic cleaning, and the abrasion resistances with cryogenic treatment are better than those without cryogenic treatment.

Table 8 (a) Abrasion test results of three different coating materials without cryogenic treatment.

Coating	HA (Density:3.08 g/cm^3)		Al_2O_3 (Density:3.3 g/cm^3)		ZrO_2 (Density:4.48 g/cm^3)	
	A	S	A	S	A	S
Weight before test (g)	30.93	27.77	24.56	27.54	29.43	26.55
Weight after test (g)	30.87	27.72	24.51	27.50	29.13	26.25
Volume Loss (mm^3)	19.48	16.23	12.73	11.51	67.19	64.96

Table 8 (b) Abrasion test results of three different coating materials with cryogenic treatment.

Coating	HA (Density:3.08 g/cm^3)		Al2O3 (Density:3.3 g/cm^3)		ZrO2 (Density:4.48 g/cm^3)	
	A	S	A	S	A	S
Weight before test (g)	26.59	28.45	31.12	31.14	27.12	29.76
Weight after test (g)	26.54	28.42	31.09	31.01	26.82	29.49
Volume Loss (mm3)	13.64	10.39	9.09	8.49	66.96	60.04

$$* \text{Volume Loss}(mm^3) = \frac{mass\ loss(g)}{density(g/cm^3)} \times 1000$$

Remark: A: High pressure air cleaning
S: Ultrasonic cleaning

4. Conclusions

In this study, experimental results display that the coating properties are very different depending on the choice of pre-treatment and post-treatment. From the test results of surface roughness, hardness, metallographic microstructure, bond strength, and abrasion resistance, the choice of ultrasonic cleaning and cryogenic treatment can effectively improve the coating properties.

Acknowledgements

The authors would like to thank the financial support of National Science Council, Taiwan (Project: Engineering Analysis and Manufacturing of Human Knee Joints and its Prosthesis, NSC 99-2632-E-230-001-MY3).

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3/14/2013