Chapter 3

Experimental

The experiment consists of characterization and wear investigation of used off-shore pump plunger. Characterization spray materials were carried out. Preparation and characterization of coating were performed along with in-flight particles and splat investigation. In addition, wear performance of the plunger coatings were tested under simulated condition. The overall step of experiment is illustrated in Fig. 3.1.

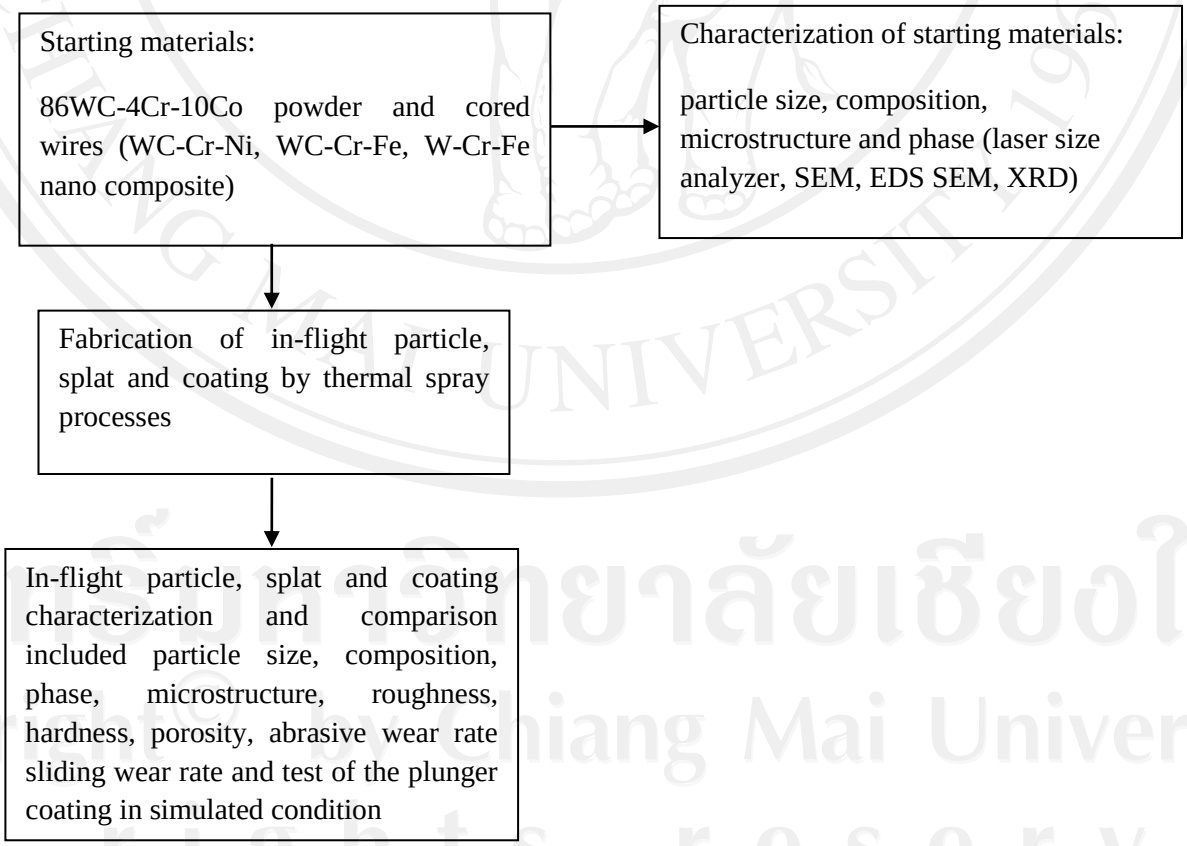

Figure 3.1 Schematic flow chart preparation and characterization of coating.

3.1 Materials and Apparatus

Materials and apparatus employed in this work are as follows:

3.1.1 Materials

1) Original plunger used
2) SUS 304 stainless steel (substrate)
3) 86WC-4Cr-10Co spray powder, DURUM Co. Ltd., Germany
4) WC-Cr-Ni cored wire, DURUM Co. Ltd., Germany
5) WC-Cr-Fe cored wire, Beijing Advance Metal Material Co. Ltd., China
6) WC-Cr-Fe nanocomposite cored wire, Praxair Surface Technology Co. Ltd., USA

3.1.2 Apparatus

1) Arc sprayer, Model TAFA Arcject 9000 and Spray gun holder and speed controller, TAFA Technology Co. Ltd., USA
2) High Velocity Oxygen-fuel sprayer (HVOF), Model JP5000, TAFA Technology Co. Ltd., USA
3) Venier calipers, Mitotoyo, Digimatic calipers, Japan
4) Wire cutting, Model FA 10 S, Japan
5) Metallurgy Cutter, Model Q2, China
6) Hot mounting, Model CitoPress-1, Germany
7) Roughness tester, Model Surtronic 3^{+}, Taylor Hobson, UK
8) Metallographic specimen polishing machine, Model Lapotom 2,Germany
9) Optical Microscope, Model JENGO 233, Germany

10) Scanning electron microscope (SEM), Model JEOL JSM-5910, Japan
11) X-ray diffractrometer, Model Rigaku TTRAX III, Japan
12) Focus iron beam (FIB), FEI xT Nova Nanolab 200 dual beam microscope, USA
13) Transmission electron microscope (TEM), Philips CM200, Japan
14) Gold coater, Model JFC 1200 Fine coater, Japan
15) Vickers Hardness tester, Model Wolpert W Group, UK
16) Image analysis program SCENTIS, Germany
17) Pin on disk tester, Model ISC-200 TRIBIMTER by ASTM G99_90 Standard
18) Dry sand rubber wheel tester by ASTM G65 Standard
19) Particle size analyzer Laser diffraction, Model Mastersizer S
20) CMM measuring machine, Mitutoyo, Model Beyond-CrystaC
21) Analytical balance mettle, Toledo, Model AB304-S
22) Lathe machine
23) Grinding and diamond wheel
24) Plunger test unit

3.2 Characterization of original plunger and wear test

3.2.1 Worn surface analysis of used original plunger

The used plunger surface was cleaned by alcohol and blow dried. Worn surface compared with unworn area of the plunger were examined by optical

microscope. Diameter of plunger at five random points were measured by a vernier caliper to compare the difference of plunger size between worn and unworn area.

3.2.2 Plunger characterization

3.2.2.1 Roughness

The plunger surface was clean by alcohol and blown dried before roughness measuring by roughness tester with the transverse speed of 1 mm/sec and transverse length of 2.5 mm The roughness of unworn and worn area was randomly tested along the plunger length for 15 locations and calculated for the average roughness.

3.2.2.2 Microstructure

The cross-section of a plunger was prepared by procedure shown in Fig. 3.2. Step 1, using wire-cut machine for cross-sectional cutting to obtain a piece of 10 mm thickness. Step 2, cutting the outer part of a coin shape at 5mm of depth. Step 3, mounting sample by hot mounting machine. All samples were ground with a wet sand abrasive paper ranging from 350, 500, 600, 800, 1000 and 1200 respectively. Fine polishing was done by using powered wheel with 0.3 μm of alumina slurry. All samples were gold coated using sputtering technique prior to for 30 seconds for microstructural characterization by OM and SEM. Chemical compositions of the original plunger coating was analyzed by EDS-SEM technique.

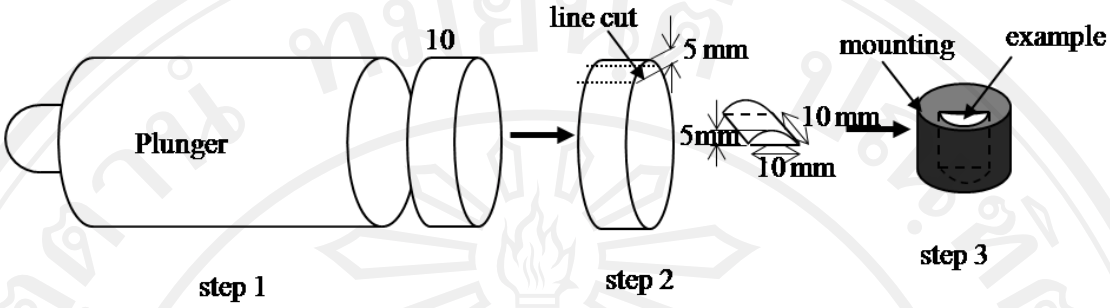

Figure 3.2 Schematic for the cutting and preparation of sample for characterization of microstructure.

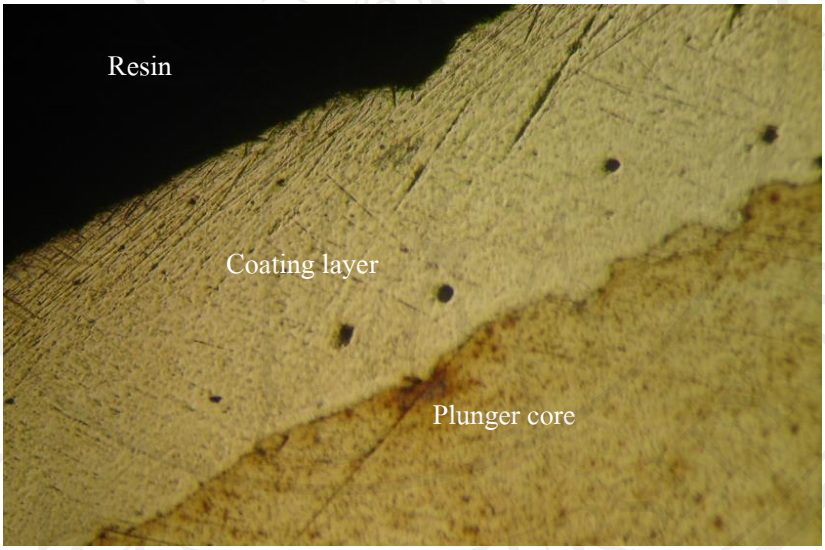

Figure 3.3 OM image of cross-section of plunger at 100X magnification.

3.2.2.3 Thickness

The sample prepared by procedure described in 3.2.2.2 was also characterized by optical microscope. Optical micrograph of the outer coating layer and the core part (inner) were taken to measure the thickness by using SCENTIS Image analysis program (Fig. 3.3). The thickness was randomly measured for 10 points on unworn and worn area and calculate the average thickness value.

3.2.2.4 Porosity

Porosity of the original plunger coating was measured by optical image analysis at 100X magnification using SCENTIS Image analysis program. Ten spots were measured to obtain the average porosity value.

3.2.2.5 Hardness

The hardness of plunger coating and core part were examined by Vickers micro hardness tester with 300g applied load and 5 seconds dwell time. The hardness was randomly measure at 15 points to obtain the average value.

3.2.2.6 Abrasive wear test

The plunger specimen was prepared for abrasion test as illustrated in Fig 3.4. The plunger was cross sectioned by wire cutting technique to obtain a piece of 50 mm long and then was longitudinal cut at 8 mm thick. The dry sand rubber wheel wear tester according to ASTM G65 standard was employed using brown Al_2O_3 abrasive particle at the flow rate of 210 g/min. The load used for testing was 27.5 N at 100 m sliding distance of each interval followed by cleaning with alcohol, blow-dried and measured weight loss. Weight loss was measured up to 6 internal to obtain a total of 600 m sliding distance. Wear rate was calculated by plotting a graph between accumulated weight loss and sliding distance. Wear rate can be obtained from the slope in a unit of g/m.

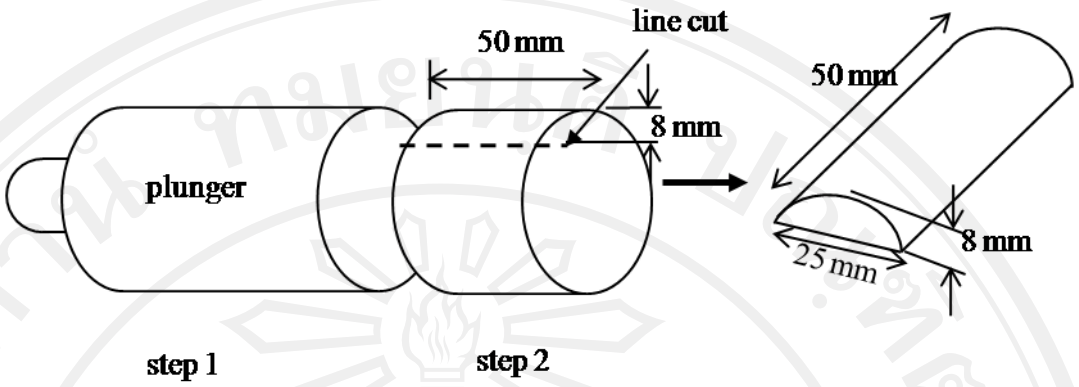

Figure 3.4 Schematic diagram showing cutting procedure of plunger for wear test.

3.3 Characterization of starting materials

Spray materials in this work were 86WC-4Cr-10Co powder and three types of cored wire including WC-Cr-Ni, WC-Cr-Fe, and W-Cr-Fe nanocomposite. Characterization of spray materials included morphology, size, microstructure, chemical compositions, and phase compositions. Nominal compositions of spray materials detailed by supplier were shown in Table 3.1.

3.3.1 Morphology and Size

The as-received 86-WC-4Cr-10Co powder and filler particle taken from all cored wires were prepared by putting particles on carbon tape followed by gold sputtering just prior to SE-SEM and BSE-SEM investigation. Characteristics of cored wires were examined for both outer shell and core filler for all types of cored wire. The cored wires were cross sectional cut into 10 mm long and hot mounted followed by sand paper grinding with 320, 500, 600, 800, 1000 and 1200 respectively. Samples were then polished with 1 µm and 0.3 µm of alumina slurry followed by gold sputtering coat. All

samples were analyzed by SE-SEM and BSE-SEM at 15 kV applied voltage along with EDS-SEM for chemical analysis.

Size distribution of spray powder and filler particles of cored wires were analyzed by laser particle size analyzer.

Table 3.1 Nominal chemical compositions of powder and cored wire by supplier.

Designated code	Chemical Compositions (wt%)													Diameter (mm)
	Si	B	C	W	Mn	Cr	Mo	WC	Nb	TiC	Co	Ni	Fe	
86WC-4Cr-10Co	-	-	-	-	-	4	-	86	-	-	10	-	-	5-22 (powder size)
WC-Cr-Ni	5	2	0.4	-	-	10	-	50	-	-		Bal	-	1.6 (ϕ wire)
WC-Cr-Fe	1	2		-	-	13	-	26	-	6		6	Bal.	2.0 (ϕ wire)
W-Cr-Fe nanocomposite	2	5	4	15	3	25	6	-	12	-		-	Bal.	1.6 (ϕ wire)

3.3.2 Microstructure

Filler particles of cored wires were cross-sectional characterized. Preparation was done by hot mounting, grinding with sand paper using mesh size 320, 500, 600, 800 and 1000, respectively and then polished with 1 µm and 0.3 µm alumina slurry. All samples were gold coated by sputtering technique before SEM investigation

at 15 kV applied voltage. Micrographs were taken by SE-SEM and BSE-SEM techniques.

3.3.3 Chemical Compositions

Chemical compositions of all filler were analyzed by BSE-SEM along with EDS-SEM using area analysis.

3.3.4 Phase Compositions

Phase compositions of the cored wire filler were analyzed by X-ray diffraction technique. The XRD conditions were 50 kV and 30 mA by using Cu K_α. The goniometer was set at a scan rate of 1.54 Å, W= 0.02°, 2.4 degree per second/over a 2 θ range of 20-40°. Data obtained were Bragg's angle and intensity of veering peak diffraction (I). Phase were identified by using JCPDS standard.

Bragg's law

$$2d_{hkl}\sin\theta = n\lambda \qquad (3.1)$$

λ = wave length,

n = reflection order

d_{hkl} = spacing between adjacent crystal plane

θ = the angle of scattering

3.4 Preparation of coatings and collection of in-flight particle and splat

3.4.1 Substrate preparation

The SUS 304 stainless steel with a dimension of 25 x 50 x 5 mm were used as substrate for all coatings. All substrates were subjected to sandblasting with 24 mesh size of SiC. After blow clean substrates were attached and welded to the holder wheel, as shown in Fig. 3.5. The wheel was mounted on the lathe machine and rotating at speed of 35 rpm during spraying.

Figure 3.5 Installation of substrates on holder wheel and lathe machine.

3.4.2 Preparation of coating

3.4.2.1 86WC-4Cr-10Co coating

86WC-4Cr-10Co coating was prepared by HVOF spray process using spray parameters shown in Table 3.2.

Table 3.2 Spray parameters of HVOF process.

Parameters	
Oxygen pressure (bar)	14
Kerosene Fuel (l/min)	5.5
Nitrogen pressure (bar)	2
Spraying distance (mm)	350

3.4.2.2 Cored wire coatings

Cored wire coatings were prepared from three types of cored wire including WC-Cr-Ni, WC-Cr-Fe and W-Cr-Fe nanocomposite. The arc spray parameters recommended by manufacturers were employed as shown in Table 3.3. Spraying procedure are as follows.

1) rotate the holding wheel at the speed of 35 rpm

2) feed the spraying gun at the speed rate of 15 m/min, for 10 cm length on each side of the sample

3) repeat 5 passes for each spray at the controlled temperature 90 °C

4) measure thickness of the coating with vernier caliper and wait until the coating temperature was below 50°C before the next spraying

5) repeat steps 3 and 4 until the coating was approximate 500 μm thick

Table 3.3 Recommended arc spray parameters.

Parameters	Wire types		
	WC-Cr-Ni	WC-Cr-Fe	W-Cr-Fe nano-composite
Arc voltage (V)	35	40	35
Arc current (A)	200	250	200
Air pressure (bar)	4.2	4.2	4.2
Wire feed rate (m/min)	8	8	8
Spray distance (mm)	150	150	150

3.4.3 Collection of in-flight particles

The three types of cored wire: WC-Cr-Ni, WC-Cr-Fe, and W-Cr-Fe nano composite were arc sprayed by parameters shown in Table 3.3. The in-flight particles for all types of cored wire were collected in distilled water by spraying through a 3 holes (Ø 3 mm) stainless steel plate into water (Fig. 3.6a). The collected in-flight particles were cleaned by alcohol and air dried.

3.4.4 Collection of Splat

Fig. 3.6b shows method of splat collection for all types of cored wire. Splat collection was done by spraying onto stainless steel plate.

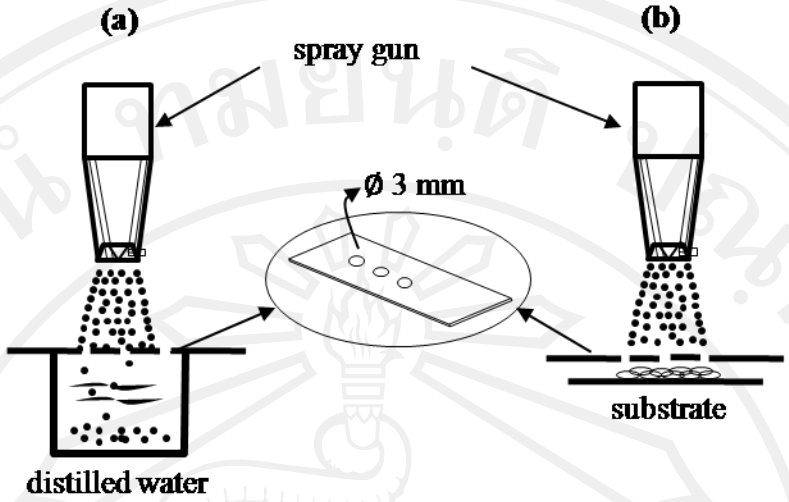

Figure 3.6 Schematic diagram shows collection methods (a) in-flight particle collection and (b) splat collection.

3.5 Characterization of in-flight particle

Characteristics of in-flight particle produced by three types of cored wire including WC-Cr-Ni, WC-Cr-Fe, and W-Cr-Fe nanocomposite were investigated morphology, particle size, microstructure, chemical compositions, and phase compositions are described as follows.

3.5.1 Morphology and size

In-flight particles were prepared as previously described for morphology and size investigation by SE-SEM.

Size distributions of in-flight particles were also analyzed by laser particle size analyzer.

3.5.2 Microstructure

In-flight particles of cored wires were cross-sectional characterized. Preparation was done by hot mounting machine, grinding with sand paper using mesh size 320, 500, 600, 800 and 1000, respectively and then polished with 1 µm and 0.3µm alumina slurry. All samples were gold coated by sputtering technique before SEM investigation at 15 kV applied voltage. Micrographs were taken by SE-SEM and BSE-SEM techniques.

3.5.3 Chemical Compositions

Chemical compositions of all in-flight particles were analyzed by BSE-SEM along with EDS-SEM using area analysis.

3.5.4 Phase Compositions

Phase compositions of all in-flight particles were analyzed by X-ray diffraction technique. The XRD conditions were 50 kV and 30 mA by using Cu K_α. The goniometer was set at a scan rate of 1.54 Å, W= 0.02^o, 2.4 degree per second/over a 2 θ range of 20-40^o. Data obtained were Bragg's angle and intensity of veering peak diffraction (I). Phases were identified by using JCPDS standard.

3.6 Characterization of splat

Characteristics of splat produced by three types of cored wire including WC-Cr-Ni, WC-Cr-Fe, and W-Cr-Fe nanocomposite were investigated morphology, splat size, microstructure, chemical compositions, and phase compositions are described as follows.

3.6.1 Morphology and size

Splats were prepared as previously described for morphology and size investigation by SE-SEM.

3.6.2 Chemical Compositions

Chemical compositions of all splats were analyzed by BSE-SEM along with EDS-SEM using area analysis.

3.6.3 Splat diameter, degree of flattening and degree of splashing

Measurement of perimeter and area of individual splat were performed by using image analysis with SCENTIS software. The measurement were randomly performed on 100 splats in order to obtain the average values and to further calculate the diameter of the splat, the degree of flattening (DF) and the degree of splashing (DS). Splat diameter d_s (splat size) was calculated by equation (3.2) using measured splat area. The degree of flattening is the ratio of the splat diameter (d_s) to the in-flight particle diameter (d_i) as shown in equation (3.3), while the degree of splashing is the ratio of calculated splat area ($P^2/4\pi$) to actual splat area (A) measured by image analysis as shown in equation (3.4).[13]

$$d_s = (4A/\pi)^{1/2} \quad (3.2)$$

$$DF = d_s/d_i \quad (3.3)$$

$$DS = (P^2/4\pi).(1/A) \quad (3.4)$$

When P is the perimeter of splat, and A is the actual (measured) area of splat.

3.7 Characterization of coatings

Characterization of all coatings including 86WC-4Cr-10Co, WC-Cr-Ni, WC-Cr-Fe and W-Cr-Fe nanocomposite coating were carried out. Coating characteristics included roughness, thickness, microstructure, porosity, hardness, chemical compositions, phase compositions, and wear performances which are detailed below.

3.7.1 Sample preparation

3.7.1.1 Preparation of coating for OM and SEM investigation.

The cross-section of coatings were prepared by procedure shown in Fig. 3.7. Step 1, using cutter machine for cross-sectional cutting to obtain a piece of 10 mm thickness. Step 2, mounting sample by hot mounting machine. All samples were ground with a wet sand abrasive paper ranging from 350, 500, 600, 800, 1000 and 1200 respectively. Fine polishing was done by using powered wheel with 0.3 μm of alumina slurry. All samples were gold coated using sputtering technique prior to for 30 seconds for micro structural characterization by SEM. Chemical compositions of the coating was analyzed by EDS-SEM technique.

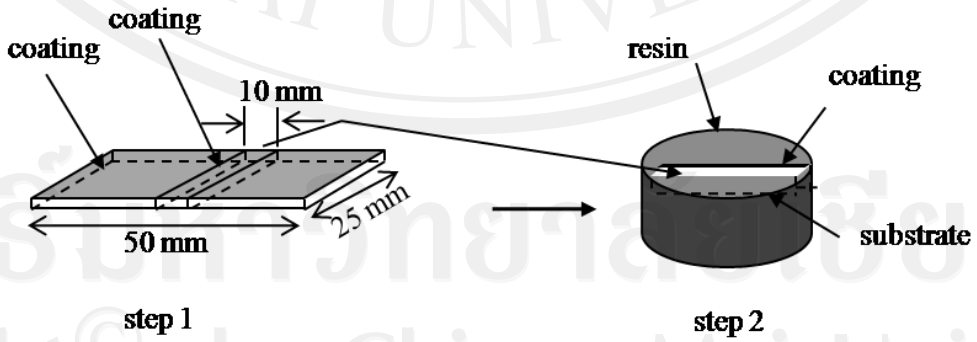

Figure 3.7 Coating sample preparation.

3.7.1.2 Preparation of coatings sample for TEM characterization

FEI xT Nova Nanolab 200 dual beam microscope (FIB) was used to prepare coating cross-sections for TEM observation. It was prepared by using the lift-out method into copper grid of TEM as shown Fig. 3.8. Philips CM200 transmission electron microscope was used to analyse coating matrix along with EDS-TEM.

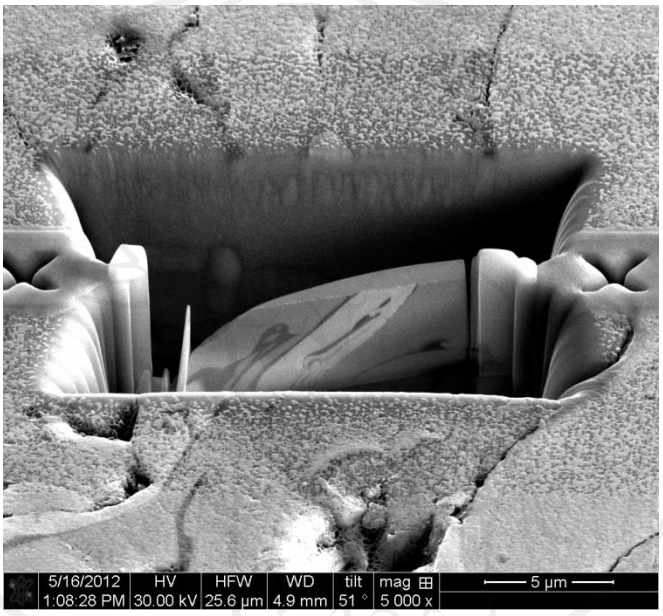

Figure 3.8 FIB image showing cross-section of the coating for preparation sample for TEM.

3.7.2 Roughness

Coatings were clean by alcohol and blow dried before roughness measuring by roughness tester with the transverse speed of 1 mm/sec and transverse length of 8 mm The roughness of coatings were randomly tested for 15 locations and calculated for the average roughness.

3.7.3 Thickness

The coating sample prepared by procedure described in 3.2.2.2 was also characterized by optical microscope. Optical micrographs of coatings layer were taken to measure the thickness by using SCENTIS Image analysis program. The thickness was randomly measured for 10 points to calculate the average thickness value.

3.7.4 Microstructure

The cross-section of coatings sample were prepared by procedure in 3.7.1 as shown in Fig. 3.7. Step 1, using cutter machine for cross-sectional cutting to obtain a piece of 10 mm thickness. Fine polishing was done by using powered wheel with 0.3 μm of alumina slurry. All samples were gold coated using sputtering technique prior to for 30 seconds for microstructural characterization by SEM. Chemical compositions of the coating was analyzed by EDS-SEM technique.

3.7.5 Chemical composition

Chemical compositions of all coatings were analyzed by BSE-SEM along with EDS-SEM using area analysis.

3.7.6 Porosity

Porosity of the coating samples were measured by optical image analysis at 100X magnification using SCENTIS Image analysis program. Ten spots were measured to obtain the average porosity value.

3.7.7 Hardness

The hardness of coating samples were examined by Vickers micro hardness tester with 300g applied load and 5 seconds dwell time. The hardness was randomly measure at 15 points to obtain the average value.

3.7.8 Phase composition

Phase compositions of the coatings sample were analyzed by X-ray diffraction technique. The XRD conditions were 50 kV and 30 mA by using Cu K_{α}. The goniometer was set at a scan rate of 1.54 Å, W= 0.02°, 2.4 degree per second/over a 2 θ range of 20-40°. Data obtained were Bragg's angle and intensity of veering peak diffraction (I). Phases were identified by using JCPDS standard.

3.8 Wear test of coating

3.8.1 Sliding wear test

All coatings specimens of sliding wear were polished to Ra<0.2 μm by grinding and wet sand abrasive ranging from 800, 1000 and 1200 respectively. Pin-on-Disk sliding wear tester was employed with WC-Co ball (3.15 mm-radius) at a load of 1000 g. The sliding radius was set at 4, 6, and 8 mm with sliding distance of 500 m at each radius. The linear speed used was 7.5 cm/min, under normal atmosphere. After each transverse length, width of wear track was measured and calculated for volume loss (V) using equation (3.5) in which wear rate can be calculated by equation (3.6).

$$V = \pi(r_t)(w_t)^3 / 6r_p \quad (3.5)$$

When V = volume loss after (mm^3)

r_t = wear track radius (mm)

w_t = track width (mm)

r_p = sphere radius (mm)

$$Q = V/L \quad (3.6)$$

When Q = wear rate (mm)

V = volume loss (mm^3)

L = distance (m)

3.8.2 Abrasive wear test

Wear performance of all coatings were evaluated by using a dry sand rubber wheel abrasive tester according to ASTM G65 standard. Brown Al_2O_3 with 600 mech size was used as abrasive particle at constant feed rate of 210 g/min. The load used for testing was 27.5 N at 100 m sliding distance of each interval followed by cleaning with alcohol, blow-dried and measured weight loss. Weight loss was measured up to 6 internal to obtain a total of 600 m sliding distance. Wear rate was calculated by plotting a graph between accumulated weight loss and sliding distance. Wear rate can be obtained from the slope in a unit of mg/m. Wear scar were also revealed by SEM image.

3.9 Preparation of pump plunger coating

The plungers used in this research are the courtesy of CHEVRON Company. One 50 mm diameter and three 70 mm diameter plungers along with TXT 6121 model reciprocating pump. All 4 plungers have the same physical characteristics except in the diameters. They are made of the same material from the same manufacturer and designed to work with the reciprocating pump. Without the matching reciprocating pump, the 50.82 diameter plunger is used as a specimen in a destructive test. The three 70 mm diameter plungers are removed a original coating and replaced coatings with three type of cored wires, WC-Cr-Fe, WC-Cr-Fe and W-Cr-Fe nanocomposite. Finally, the plungers are tested in a simulated condition with TXT 6121, 70 mm diameter reciprocating pump.

The coating of three types of cored wires include WC-Cr-Ni, WC-Cr-Fe and W-Cr-Fe nanocomposite were prepared by using arc spraying process. Details of preparing plunger samples, cored wires spraying, and surface finishing are discussed below.

3.9.1 Coating procedure for pump plunger

Pump plunger used was from gas/pneumatic driven injector pump model 6100 with diameter of 69.86 mm (2 ¾ inch.) and length of 300 mm The plunger was made from 17.4 SS high alloy steel. Coating procedure is detailed as follows.

1) clean and measure plunger with micrometer by measuring diameter from the end of plunger at 300 mm interval and sketch a drawing of the plunger.
2) remove 1 mm of plunger skin to get rid of old coating layer by lathe machine.

3) all plungers surface were grit – blasted clean with 24 mesh size SiC and to create a roughness of about 8 μm:Ra.

4) install the prepared plunger on lathe machine as shown in Fig. 3.8. and rotate the plunger at the speed of 35 rpm while spraying.

5) assemble the arc spray gun on gun holder as shown in Fig. 3.9. Spraying distance was set at 150 mm with a gun speed of 15 m/min.

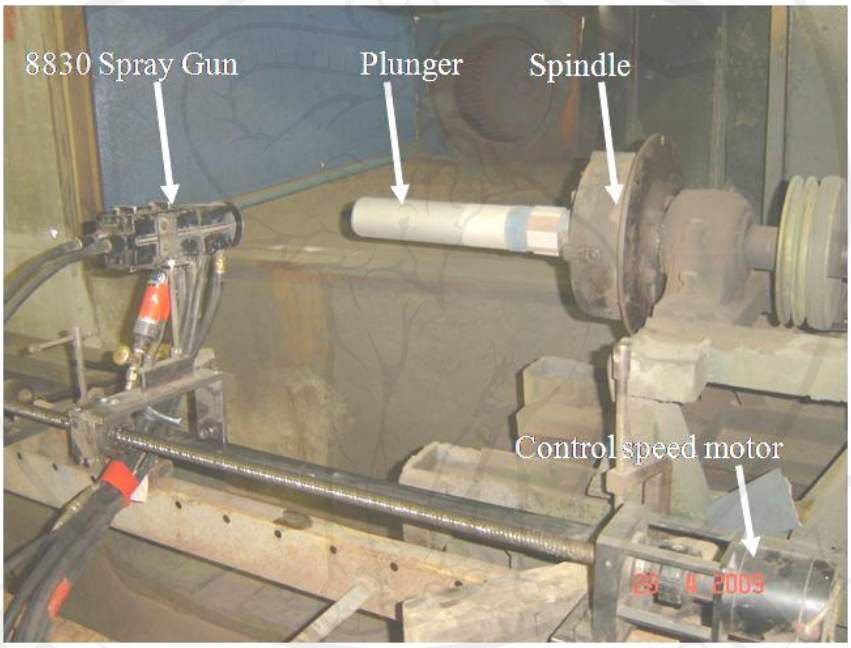

Figure 3.9 Spraying procedures for pump plunger.

3.9.2 Coating spray by cored wires

The plungers were coated with three types of cored wire including WC-Cr-Ni, WC-Cr-Fe and W-Cr-Fe nanocomposite. All plungers were sprayed with TAFA Arcject 9000 sprayer and TAFA 8830 spray gun, using parameters shown in Table 3.2. Coating thickness was set at 800μm.

3.9.3 Finishing method for plunger coating

The coated plunger was resized to its original diameter of 69.860 mm by grinding with diamond wheel. Then, the plunger was polished by woolen fabric using lath machine to achieve the surface smoothness of 0.2 μm.

3.10 Testing of coated pump plunger under simulated conditions

Plunger coated with difference types of spray materials were performed wear testing under simulations. Detail of the test is as follows.

3.10.1 Construction of the plunger test unit and data collection program

The plunger test unit was designed to simulate pump conditions. Perpendicular steel plate with a dimension of 75 x 75 x 6 mm used as a unit structure. The test unit was 80 cm wide, 120 cm long and 80 cm high. The gas/pneumatic driven injector pump model 6121 was installed on the unit test with 5 Hp gear power motor (220 V, 3-phase). The adjustable speed was in the range of 0 - 255 rpm. A 25 meters - long steel pipe with diameter of 25mm (1 inch) was installed as a coil form to minimize the test unit size. Electronics sensors installed to measure pressure, temperature, and stroke, as shown in Fig. 3.10.

Data collection program was created by Visual Basic and installed in a DATA logger which received various signals from multiple sensors. Real-time data were recorded as digital files with .tex extension. Graphs of transverse length, pump pressure and distance of plunger moment were shown on computer monitor.

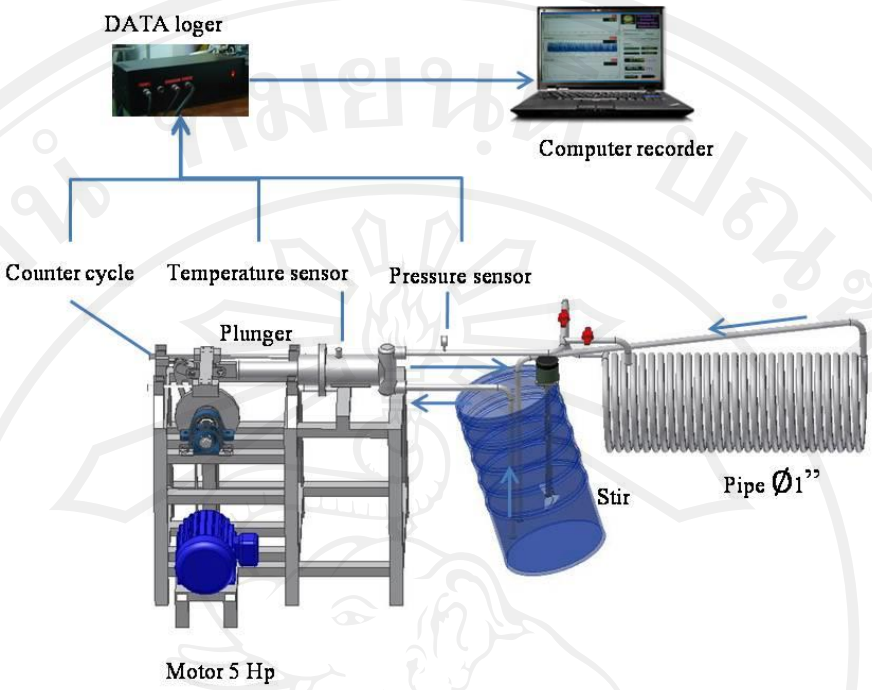

Figure 3.10 Schematic diagram of test unit.

3.10.2 Test conditions and operation

Test condition and operation steps are described as follows.

1) Prepare 210 liter of used engine oil put into 3 buckets (70 liter each) Then, add 20 g of SiO_2 sand into each bucket.
2) Attach a stirrer in each bucket. Tilt the buckets for 15° to facilitate sand particle to settle down at the end of exit tube in order to continuously stir the mixture.
3) Set the test conditions as

- Speed of plunger stroke at 50 rpm
- Stroke length of 16.5 cm
- Pressure of 6 bars

- Data collection and record every 1 min

- Total length of 100,000 m from 5 intervals (20,000 m each)

measure diameter and wear scar observation.

3.10.3 Analysis of worn surface

3.10.3.1 Measurement of plunger diameter

After each test of 20,000 m plunger sliding distance, diameter of the plunger was measured with CMM (coordinate measuring machine) by vertically in stalled the plunger as shown in Fig.3.11. Plunger diameter was measured at 10 mm apart of each point for 18 points to obtain the average value.

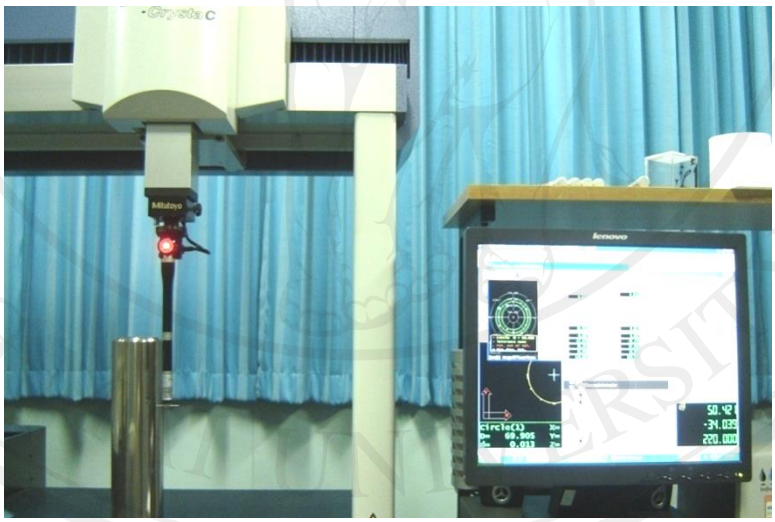

Figure 3.11 Measurement of plunger diameter by CMM technique.

3.10.3.2 Analysis of plunger worn surface by optical microscope

Following the test of each interval, the plunger worn sure face was observed at 1, 5, 10, 15 and 16.5 cm from the end side as shown in Fig. 3.12. Worn surface at each specific length of the plunger were observed by optical microscope.

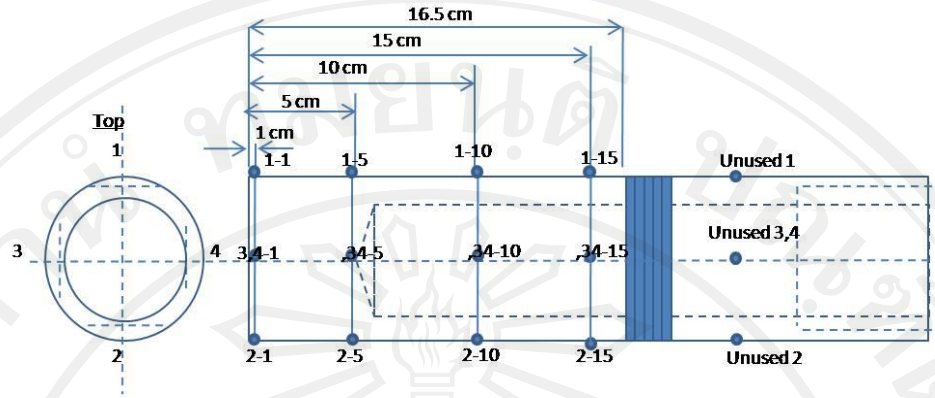

Figure 3.12 Schematic diagram for measurement of plunger worn surface.