The study of mechanical properties and corrosion resistance of Molybdenum coating and Aluminum coating using Thermal Spray method on stainless steel 316L

R Riastuti*, S T Siallagan, Z Rahmat, B A H Bancin

Department of Metallurgy and Materials Engineering, Faculty of Engineering, Universitas Indonesia, Depok, West Java, Indonesia
* rini.riastuti@ui.ac.id

Abstract. Thermal spray is one of the most common coating technologies used in oil and gas industries. This research applied two thermal spray methods, namely High Velocity Oxygen Fuel for Molybdenum coating and Electric Arc Spray for Aluminum coating to compare the coating formed by different methods on stainless steel 316L substrates. The thickness of the Mo coating ranges from 15-20μm, whereas Al coating has a thickness of 90-100μm. Coating characterization was assessed by using several testing methods, i.e. the Positive Material Identification (PMI) to identify the coating composition, the metallographic testing to observe the interface bonding of substrate and coating, hardness test and wear test to characterize the mechanical properties of the coating and salt spray test to identify the corrosion resistance. Base on the PMI testing, the Al coating show 98.15% wt of aluminum, while Mo coating contains only 9.832% wt molybdenum. The SEM/EDX applied in the metallographic test shows that the coating has a mechanical bond with the surface of the substrate, in which the Mo coating has a more effective bond than the Al coating on the substrate. The aluminum coating has a lower hardness value of 96 HB, while the Mo coating is approximately 106 HB. The wear test shows the Mo coating has a better wear resistance than the Al coating because the Mo coating has a smaller slit width of 0.375mm. The salt spray test shows the two types of coating did not affect the scratched area but there is significant discolouring that indicates the formation of uniform corrosion.

1. Introduction
Stainless steel material is used as one of the option to reduced corrosion rate of material. However, for some applications that require high corrosion resistance, stainless steel is coated with metallic coatings such as aluminum, nickel, chromium and molybdenum on the surface of the stainless steel. The coating method applied to stainless steel material is the thermal spray method. The thermal spray is one of the most common technologies used because it can improve wear resistance, corrosion resistance and resistance to aggressive and high-temperature environments [1].

The thermal spray method is a technique using spray gun to fill the melted particles on the substrate surface followed by fast cooling process and solidification [2]. The Thermal spray technology has been used intensively as a heat-resistant coating for space industries, boiler components, offshore rigs and equipment in the oil and gas industries. There are several types of thermal spray commonly used such as High Velocity Oxygen Fuel (HVOF) and Electric Arc Spray. HVOF method uses powder material as a feedstock and oxygen gas as fuel gas.
The spraying process is done by feeding the powder axially into the nozzle then driven by a high-pressure combustion gas. The particles that have been heated in the combustion chamber will be sprayed onto the surface of the substrate. After the particles penetrate onto the surface of the substrate, the particles are cooled and deformed into laurel [3,4]. Meanwhile, the electric arc spray method uses 2 electric wires with DC current. In the spraying process, the currents will generate an electric spark between the two ends of the wire causing the wire to melt. The melted wire is sprayed onto the surface of the substrate that forms the droplets and then fills the pores of the coated substrate [5].

This study used Stainless Steel 316L as a substrate coated by an aluminum coating and molybdenum coating. The molybdenum coating is obtained by using High Velocity Oxygen Fuel method while the aluminum coating is obtained by using the Electric Arc Spray method. The study aims to observe the mechanical properties and corrosion resistance of the coating generated by different methods.

2. Materials and Methods

2.1. Materials

The material used as a substrate in this research was Stainless Steel 316L with a dimension of 50x150x60mm. The chemical composition of Stainless Steel 316L is shown in table 1.

![Stainless Steel 316L substrate](image)

The feedstock coating used in High Velocity Oxygen Fuel process is molybdenum powder with Amperit brand sized of 88μm containing 97.8% Molybdenum, while the Electric Arc Spray method used feedstock in the form of aluminum wire of Metallisation brand sized of 1.6 mm containing 99.5% aluminum.

![Figure 1. Stainless Steel 316L substrate](image)

| Elements | C   | Si  | Mn  | P  | S  | Ni  | Cr  | Mo  | N  |
|----------|-----|-----|-----|----|----|-----|-----|-----|----|
| (%)wt    | 0.025 | 0.6 | 0.9 | 0.035 | 0.001 | 10.1 | 16.7 | 2.04 | 0.03 |

2.2. Methods

The surface pre-treatment of the material is done through several stages. The first stage is cleaning the surface of the substrate by using thinner to clean the surface substrate of impurities such as oil and corrosion oxides. Then, the substrate is preheated by using a flame torch to remove water and thinner which may still be present on the surface of the substrate. Furthermore, the sand blasting method is applied to obtain a rough surface using sand
abrasive type of aluminum oxide grade C sized 24#, with spraying range of 10-15 cm in blasting cabinet with an air pressure of 4-5 bar. Subsequently, the Elcometer Surface Profile Gauge is used to measure the surface roughness of the substrate with a desired roughness of not less than 20 microns and hygiene clearance with Sa 3 hygiene (white metal blast cleaning) in accordance with ISO 8501-1: 2007

After the surface cleaning process, the coating process uses the High Velocity Oxygen Fuel (HVOF) method to obtain Mo coating and Electric Arc Spray layer to obtain Al coating. The used of different methods due to differences in physical properties between Al and Mo, where Mo has a higher melting point than Al i.e. 2,896K. So a thermal spray method is required which can generate high flame temperature to dilute the Mo powder i.e. HVOF method. In the High Velocity Oxygen Fuel (HVOF) process, the feed rate used is 20g/min with an oxygen pressure of 150psig. The spraying distance in this method is 250mm with a thickness of 15-20μm. Meanwhile the Electric Arc Spraying method to obtain Al coating uses the OSU Hessler machine and Metallisation aluminum wire. The coating process with this method uses arc voltage of 27-29 V with arc current of 250-280A and the spraying distance of 200-300mm. The Aluminum coating thickness ranges from 90-100μm.

The composition identification is applies the positive material identification (PMI) method using the X-ray beam principle, in which the test sample material will reflect the radiation that will generates energy for each different element. The metallographic test uses the SEM / EDX method to observe the morphology of the surface of the specimen surface before coating and after coating and to identify the chemical composition of the material. The sample dimension in this observation is 1x1cm. The hardness testing was conducted at PT Thermic Coating Industries laboratory using MITECH MH310 Hardness Tester tool with Vickers Hardness method according to ASTM E92 standard.

Wear testing was performed in the laboratory of the Department of Metallurgical and Materials Engineering of Universitas Indonesia using the standard ASTM G99 Ogoshi machine. the samples are tested obtained frictional load from the rotating disk. The salt spray test was performed based on ASTM B 117-03 standard test. The purpose of this test is to observe the performance of the molybdenum coating and aluminum coating in protecting the substrate from corrosion damage in corrosive environments containing Cl ions. The salt spray test was done in a salt spray cabinet with a variable time of 72 hours in NaCl 5% solution. The measurement of the width of the scratched area is performed to determine the ranking of the incremented scratched area which refers to ASTM D1654-79 standard.

3. Result and Discussion
The composition of Molybdenum coating and aluminum coating were determined using Positive Material Identification (PMI) as exhibited in table 2.

| Elements | Stainless steel 316L substrate |
|----------|--------------------------------|
|          | Non-coating | Aluminum coating | Molybdenum coating |
| Al       | 0           | 98.15           | 0                 |
| Mo       | 1.985       | 0.423           | 9.832             |
| Nb       | 0.008       | 0               | 0.016             |
| Cu       | 0.271       | 0.025           | 0.327             |
| Ni       | 9.545       | 0.126           | 7.702             |
Co 0.362       0.098       0.571
Fe 69.347      0.877       63.179
Mn 1.451       0           1.37
Cr 16.848      0.048       16.871
V  0.104       0           0
Ti 0.074       0.252       0

This result shows there is an increase amount of aluminum and molybdenum element in presence of the coating. The stainless steel with Al coating has Al content which almost similar with Al content on the wire that used as feedstock. While, the Mo coating has a significant difference between Mo content in the coating with the Mo powder used as feedstock. This caused by differences of types of tube detectors was used during the process. In accordance with the proposed by Pawlowski (2008) that in the process of thermal spray coating there are formation of mechanical inter locking between the coating and the surface of the substrate[6]. The adhesion between particle and the substrate will be achieved when the particle heat has been sufficient to produced molten particle that will produce microscopy layer [5]).

Figure 2. Metallographic observation using optical microscope a) non-coated, b) Mo coating and c) Al coating

The results of macro observation using OM with magnification of 100x, there is a porosity in aluminum coating (see figure 2.c). The cause of the existence of the porosity in the coating porosity due to thermal spray machine parameters, compressor, wire feed and spray angle [7,8]. The other cause of this porosity is due to the presence of aluminum wire material that is not completely melted and the presence of particles that have been solidified and trapped in the coating so that cause a blank space or porosity. The porosity will decrease of corrosion resistance and mechanical properties of the material. This porosity belongs to a coating defect that often occurs in the thermal spray process and as well as oxidized inclusions and non-melt feedstock particles [5]. SEM was used to observe the morphology of coating with greater magnification can be seen in the figure 3. The Al coating and Mo coating were formed on the substrate have good mechanical interlocking. This can be seen in the density levels between substrates and coatings in figure 2. However, in the Al coating there is porosity that indicates can decrease the mechanical properties of Al coating.
Table 3. The hardness test results

| Indentation | Hardness (H_B) |
|-------------|----------------|
|             | Al coating     | Mo coating    |
| 1           | 84             | 91            |
| 2           | 85             | 82            |
| 3           | 83             | 87            |
| 4           | 109            | 112           |
| 5           | 132            | 123           |
| 6           | 83             | 142           |

Hardness and wear tests are performed to compare the mechanical properties between the molybdenum coating and aluminum coating. The measurements hardness of the Mo coating and Al coating were carried out at 6 indentation points. The result shows the aluminum coating has a lower hardness compared to the coating Mo. It is because Al metal does have a lower hardness value than metal Mo and the formation of porosity in Al coating which can the decrease of hardness value. In addition, the value of hardness is influenced by the presence of oxygen capable of forming a very hard coating of oxide [9, 10]. The higher value hardness of Mo coating than Al coating is indicated the formation of Mo oxide in interface of Mo coating [11]. While, the results of wear test are known that the wider the gap indicates the more vulnerable material to wear. The test results show that SS 316L steel with aluminum coating has a greater volume of abrasive that is 0.0132mm\(^3\) compared to SS 316L steel with Mo coating that is 0.0043mm\(^3\). This confirm that SS 316L with Mo coating has better wear resistance than SS 316L with Al coating.
Figure 3. Metallographic observation using SEM a) Mo coating and b) Al coating

| SS 316L | Slit width (mm) | Volume of abrasive (mm³) |
|---------|----------------|-------------------------|
| Non-coated | 0.455 | 0.0077 |
| Al coating | 0.545 | 0.0132 |
| Mo coating | 0.375 | 0.0043 |

The visual observations after salt spray test of Mo coating and Al coating as described in figure 4, shows significant discoloration of the Al coating specimen. The surface of the Al coating specimen has a considerable corrosion, whereas the scratched area does not show any corrosion or widening of the scratches. While the substrate coated with molybdenum, did not show significant change of colour and no corrosion and widening on the scratched area were observed after the test. This shows that the surface of the substrate is protected by the presence of a coating in which the coating functioning as a barrier (isolation) [12]. Based on the observation of the scratched area, the aluminum coating and Mo coating have a ranking of 10 on ASTM D1654-79 which explains the absence of dilation of scratched area.
4. Conclusions
Based on this research, it can be concluded that the Molybdenum coating and Aluminum coating on 316L stainless steel has a function as an insulation (barrier) that will protect the substrate. The presence of Mo coating and Al coating can increase the hardness and wear resistance of specimens. The Mo coating has a higher hardness value than the Al layer that influenced by the presence of Mo oxide on the surface of Mo coating. Stainless steel 316L coated by molybdenum has a higher wear resistance than 316L stainless steel coated by aluminum because the Mo coating has smaller volume of abrasive than Al coating of 0.0043 mm³. The presence of Mo coating and Al coating on SS 316L can increasing the corrosion resistance of specimens whereas no corrosion and widening of scratched area were observed after salt spray test.

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