MICROSTRUCTURAL STUDIES OF THERMAL SPRAY COATING

by

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the

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Approved by

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CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

Mohd Muizaddin Bin Mohamed

ABSTRACT

Chromium carbide nickel chrome, Cr_3C_2 -NiCr coatings applied on AISI 340 alloy steel through high velocity oxy-fuel (HVOF). In this microstructural studies, steel sample were prepared to differentiate the thickness of the coating based on the hardness value between coating and substrate. The experimental procedures Cr_3C_2 - NiCr thermal spray coating process, testing procedure and equipment involved were being determined through numbers of literature reviews and availability of equipment inside university laboratory. It is to prove that chromium carbide nickel chrome Cr_3C_2 -25NiCr can produce better protection for AISI 340 alloy steel.

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CHAPTER 1 INTRODUCTION

 Cr_3C_2NiCr is a chromium carbide nickel chrome coating which is used for coating powder for HVOF thermal spray coating. It been used due to high wear resistance and corrosion which can protect material for longer life span. Therefore, this studies conducted to know

the microstructural of the chromium carbide coating which undergo HVOF thermal spray coating process in creating protecting layer for AISI 304 stainless steel substrate.

1.1 Background of Study

Metal is a great interest in industrial applications for its specific strength, stiffness and heat resistance. However due in time it will get rust (corrosion) and wear (erosion) due to contact movement of chemical or physical material on its surface. Today, coating is often been use as an element of protecting the metal from having the effect of wear and rust in a short period of time.

Thermal spray coating is a coating process that use other material or powder that been melted with heat either heated through electrical (plasma or arc) or chemical that is combustion flame and sprayed it on to surface to create protective layer shield on the metal surface. The use of thermal spray coating not only protect the metal surface from corrosion (rust) but it is also enhance properties of coated material such as thermal and wear resistance. It is the most flexible technique.

In thermal spray coating the material that been use metals, alloys ceramic plastics and composites. In this studies, Cr_3C_2 NiCr is been used as a medium in the form of micrometer size particles that will be heated to a molten or semi molten state and accelerated towards substrates. All the particle will accumulate and will be resulting the coating protecting layer of the metal.

1.2 Problem Statement

On each material surface, it have its own wearing thickness at the surface. So the coating hardness is important in order for the thermal spray coating becoming the shield for the material. Therefore the hardness of the coating is important to know in order to protect the material for longer period of time.

1.3 Objective of the Study

With regards to the problem stated, the current studied was carried out with the following objectives:

- To conduct the thermal spray coating (Cr₃C₂-NiCr) process on steel.
- To study the micro hardness of the coating (Cr₃C₂-NiCr) and substrate.
- To study the microstructure of coating (Cr₃C₂-NiCr).
- To study the porosity and oxide stringer of the coating (Cr₃C₂-NiCr).

1.4 Scope Study

This studies is focused on the microstructure of coating which is chromium carbide nickel chrome (Cr_3C_2NiCr) powder is use as a powder and using thermal spray coating (HVOF) high velocity oxy fuel process to create protective coating structure on top the surface layer of AISI 304 steel.

CHAPTER 2 LITERATURE REVIEW

This chapter contains the reviews on previous researches and theories which are relevant with the present study. It mostly cover on description of the material used, processes involves, chemical composition, achieved current results and discussions and also the appropriate procedure of the process. Thus, review on this section will attempt to use the useful information with regards on the issues that may arise.

2.1 Thermal Spray Coating

Thermal spray coating is a process of separating the metallic or the nonmetallic materials which is stored in liquid or semi molten structure. Coating material keep up the state of powder, earthenware rod, wire, or liquid materials. Utilizing liquid and powder metals Dr. M. U. Schoop and his partners created this method in 1900s for delivering coatings [1]. Their deliberations created the very first instrument for spreading with strong metal in wire structure. This basic gadget was focused around the rule that if a wire pole were nourished into an extraordinary, concentrated fire, (the smoldering of a fuel gas with oxygen), it would liquefy and, if the fire were encompassed by a stream of packed gas, the liquid metal would get to be atomized and promptly impelled onto a surface to make a shield. This methodology was at first alluded to as metallizing and been called oxy fuel. Incorporate wire, powder (metallic and earthenware), liquid metal, fired bar, explosion and high speed oxy-fuel (HVOF) is a variety type of oxy-fuel strategies. Ordinarily electrical vitality is utilized to create a high temperature source into which powder, and all the more as of late wires, are bolstered, liquefied/plasticized and passed on onto the top to be covered. The thermal spray coating can be developed based on two [2] high temperature that is flame spray and Electrical (Figure 1).



Figure 2.1: Thermal spray application method [2].

2.2 Cr₃C₂ NiCr Chromium Carbide Nickel Chrome

To provide high erosive resistance to metal substrates ceramic particle embedded in metallic matrix is called cermet carbide and been used [3]. Cr_3C_2 carbide are refractory compound that combine many favorable properties such as for instance high hardness ,certain plasticity and good wettability with the bounding metal [4]. In order to have quality coating against erosion corrosion and wear this combination of chemicals can make this material a promising coating. [5]. Chromium carbide are use rough, Cr_3C_2 is harder than steel yet gentler than tungsten carbides. Chromium carbide is an amazingly hard-headed ceramic material and demonstrates brilliant strength, anti-disintegration and erosion properties, permanent non magnet ability and surface illustriousness [6].By changing the covering method, preparing parameters and qualities of the feedstock powder, a few endeavors have been made to enhance the hardness and wear property of chromium carbide covering [7]. So chromium carbide is utilized within blend with Ni-Cr composite which enhances the durability and pliability of the coating [8].For giving great consumption wear assurance at high temperature Ni-Cr by is utilized as metallic folio [9].

2.3 HVOF Process

HVOF is a high velocity oxy/fuel coating using high velocity flame spray process [10]. Using metal powders as a medium, Browning and Witfield, utilizing rocket motor advances to create this technique [1]. The procedure was alluded to as High Velocity Oxy-Fuel (HVOF). This methodology can use different fuel gasses such as hydrogen, propylene, propane, and kerosene in burning chamber with high speed. "Stun diamond "will be created leaving the spray, Figure 6. Powders to be used as medium by HVOF are infused pivotally with extending high temperature gasses where impelled forward, heated and quickened into the surface to structure a coating. Mach 1 velocity of gas have been accounted for temperature increasing to 2,300°c (4,172°f). The coupling of inertial determined/exceedingly plasticized particles can attain coatings approaching that of hypothetical density. By implementing more thick coatings and changes in methodologies control counting in- the presentation to oxygen and force levels for HVOF firearms working with gasses is around 100-120 kw, while they can reach 300 kw for firearms working with fluid. All inclusive this procedure, working chiefly with metals, combinations and cermet's (a standout amongst the best applications) has affidavit productivity around 70% powder while stream rates up to 7.2 kg.h-1 for gasfuel gun and up to 12 kg.h-1 for fluid fuel guns. Coming about coating porosities are a couple of percentage with a great grip to substrate (around 60 to 80 Mpa) and low oxygen content (somewhere around 0.5). Noisy sound, dusty with vast amounts of touchy gasses the method will produced [10].



Figure 2.1: HVOF combustion spray [10]



Figure 2.2 :HVOF process [10]

2.4 Vickers Microhardness Test

The Vickers microhardness test is done to evaluate the hardness of the coating, it is generally utilized small sample of experiment work which are been coating. The Vickers strategy is focused around an optical estimation framework. Using standard ASTM E-384, tags a scope of light loads utilizing a diamond indenter to make a space which is measured and changed over to a hardness esteem. It is exceptionally valuable for testing on a wide kind of materials the length of test specimens are painstakingly arranged. As a part of the Vickers scale, a square base pyramid formed diamond is utilized for testing .Even "Macro" Vickers burdens can extend up to 30 kg or more , normally loads are light, going from a couple of grams to one or a few kilograms. Metals, ceramics, and composites very nearly any kind of material utilized from the hardness test. In this experiment, the Vickers hardness test technique comprises of indenting the test material with a diamond indenter, as a right pyramid with a square base and a point of 136 degrees between inverse confronts subjected to a heap of 1 to 100 kgf. The two diagonals of the space left in the surface of the material after

evacuation of the heap are measured utilizing a magnifying lens and their normal figured. The range of the inclining surface of the space is ascertained. By isolating the kgf stack by the square mm of the area the Vickers hardness is the remainder. Test sample arrangement is generally essential with a micro hardness test keeping in mind the end goal to give a little enough example that can fit into the analyzer. The spaces ought to be as vast as would be prudent to expand the estimation determination. The test technique and sample arrangement is liable to issues of administrator impact on the test outcomes.[22]



Figure 2.3 :Opposing indenter faces are set at 136 degree angle from one another [20]

The Vickers micro hardness test being carried out on the coating surface sample cross section with the load of 300 g. The expected result for hardness test for Cr3C2-NiCr is about HV0.3= 602 to 782 based on average of 10 running indentations studies that being conducted by Z.Nykiel, LPawlowski, Z Znamirowski, D.Chicot and J.Lesage.[16]



Figure 2.4: HV0.3 for Cr3C2-NiCr[16]

Based on studies that been done by Ruoyu Tan[17],the Vickers hardness test is been done through 3 type of coating that is Cr_3C_2 -NiCr ,WC-CoCr and Al₂O₃.The result of the studies indicate that WC-CoCr have the highest data that is 1215.2Hv.Then 983 Hv is recorded by Cr_3C_2 NiCr and Al₂O₃ 1006Hv which are similarly on the same level.



Figure 2.5: Micro hardness of each coating including each bond coat and substrate [17]

2.5 Scanning electron microscope test (SEM)

Scanning electron microscope (SEM) use to analyze surface profile of the coating before and after undergoing the wear test. Here the sample that have gone thru the coating and wear process will be analyze and scan to get the micro surface data. To produce a mixture of signs at the surface of strong examples the scanning electron microscope instrument (SEM) utilizes a centered light emission vitality electrons. Using the flag that get from electron-test it will uncover the sample data about the specimen including outside morphology (composition), chemical organization, and crystalline structure. A 2-dimensional picture is created that shows spatial varieties in these properties in the sample surface of the experiment. Utilizing routine SEM methods can be imaged in a filtering mode ranges from nearly 1 cm to 5 microns in width (amplification running from 20x to roughly 30,000x, spatial determination of 50 to 100 nm). The two detectors trace the electron signals by an analog or digitizing process captured and stored as a digital image on the computer's hard drive.[21]



Figure 2.6: SEM machine [20]



Figure 2.7: SEM Schematics [20]

After coating austenitic stainless steel AISI 304 with HVOF thermal spray process using Cr3C2-NiCr as medium powder, the reading of the spherical and homogeneous particle using scanning electron microscope (SEM) is well characterized that being conducted by Z.Nykiel, LPawlowski, Z Znamirowski, D.Chicot and J.Lesage.[16]



Figure 2.8: SEM micrograph Cr3C2 NiCr Being Sprayed [16]

The SEM indicate:

- i. In figure 2.9(a) have a alloy matrix which carbide grain (dark colour) were dissolved. Using lover electric power on HVOF coating process the carbide grain have better visible.
- ii. Due to high temperature of the coating process of HVOF, it generated cracks that parallel at coating surface in figure 2.9(b).

2.6 X-Ray Powder Diffraction (XRD)

It's a method that primary used in rapid analytical to indicate and identify crystalline of a material. It is consist of 3 elements that is a sampler holder-ray detector and x-ray tubes. Using cathode ray tubes is how the x-ray are generated when the cathode produce electron and it excel towards sample (target) using voltage and bombarding it with the electrons. Characteristics x-ray produced when all the electron have enough energy to force out the inner shell electron of the sample (target) therefor creating the spectra consisting of K α and K β components which have wavelength. The wavelength is the characteristic of the sample for example (Cr, Ni, Cu, and Fe). As for diffraction, foils or crystal monochromatic needed to be filtered so it can produce monochromatic x-ray. The X-ray will be recorded as the sample and detector being rotated. The computer monitor will indicate the reading that being recorded and process by the x-r ay signal.



Figure 2.9: XRD machine [23]

In this test, based on studies that conducted Z. Nykiel, L. Pawlowski, Z. Znamirowski, D. Chicot, J. Lesage [16], they use 2 types of coating powder that is Cr_3C_2 NiCr and Cr_3C_2



Figure 2.10: XRD diagrams of Cr3C2 NiCr coating with HVOF process technique [16]

NiCrAl. Phased structured being analyzed by XRD and the finding are it can determine the characterization of crystalline material that's has within the coating. In this studies it shows that from Cr_3C_2 NiCr there are three crystal phases that present that is Cr_2C_2 NiCr which are Cr_3C_2 , Cr_7C_3 and Cr_2O_3 . For the Cr_3C_2 NiCrAl it also have 3 phases that can identify that is AlNi₃, Cr_7C_3 and Al₂O₃.

CHAPTER 3 METHODOLOGY

This chapter will describe on the method of investigation and studies that will be used to accomplish the wear characterization of Cr_3C_2 –NiCr thermal spray coating, process flowchart and also key milestones that need to be followed.



3.1 Process Flow

3.2 Experiment Procedure

This is the procedure to undergo the studies:

Preparation of cutting the material sample of austenitic stainless steel AISI 304 50mm diameter for 5 each sample. The substrates need to be cut to 50mm diameter round shape to avoid any cracking during coating process



Figure 3.1:AISI 304 been cut and clean

ii. Undergo HVOF coating spray process.

-In order to analyze the microstructural studies of thermal coating, specimen need to be prepared based on the parameters:

	Table	3.1:	Specimen	parameters
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Parameters	Details
Substrate	AISI 304 (stainless steel)
Coating Powder	Cr ₃ C ₂ -25NiCr
Deposition Technique	HVOF
Coating Thickness	190 µm



Figure 3.2: Coating Powder

-After preparing the substrate, the coating powder of Cr_3C_2 -25NiCr need to be dry in an oven for 15minuites with the temperature of 100°c.



Figure 3.3: Cr3C2-25NiCr been dry

-Next sand blasting process will be done towards the sample substrate to create rough surface using high velocity in order for the coating to immerse with subtract during HVOF process.



Figure 3.4: Sandblasting Process

-After sand blasting all the substrate, the coating machine need to be prepared. At the coating machine it has two part that is flow meter unit and powder meter unit. In flow meter unit it contain the parameters of fuel, oxygen and air pressure that will be used which the fuel that is CH₄ ,oxygen and air will be supply toward the flow meter machine. Then it's the powder meter machine where the powder will placed in and an argon gas will supply towards the machine and transport the argon towards the coating gun. Before undergo the HVOF process this is the parameter need to be set towards the flow meter and powder meter machine.

Parameters	Details
Fuel Gas	CH ₄ (Natural Gas)
Oxygen Pressure (bar)	10.3
Fuel Pressure (bar)	7.6
Air Pressure(bar)	6.9
Oxygen Flow	279
Rate,O2(NLPM)	
Fuel Flow Rate,CH4	190
(NLPM)	
Air Flow Rate, (NLPM)	361

Table 3.2: Flow meter machine parameter

Table 3.3: Flow powder machine parameter

Parameters	Details
Argon Flow Rate (NLPM)	12.5
Air Flow Rate, (NLPM)	361

Table 3.4: HVOF Gun Parameter

Parameter	Details
Spray Rate (g/min)	38-75
Spray Distance (mm)	230
Deposit Efficiency	30



Figure 3.5: Flow meter HVOF machine



Figure 3.6: Powder meter HVOF machine

-Coating powder that been dry been put inside the powder meter machine.



Figure 3.7: Coating powder inserted inside the coating furnace

-After preparing the parameters needed to undergo the coating, HVOF process can be proceed. Firstly need to open up the flow meter allowing the natural gas, oxygen and air to flow towards the HVOF gun. Then start up the gun to create flair. Then turn on the powder meter machine allowing gas argon to flow with the coating powder towards the gun and create "Stun diamond" will be created leaving the spray gun .It will infused pivotally with extending high temperature gasses.



Figure 3.8: HVOF coating process



Figure 3.9: HVOF gun

-The coating process is done and the substrate cover with coating needed to be cold down based on room temperature.



Figure 3.10: Coating Substrates

iii .After doing the coating process, a test been conducted for the microstructural study. The test are:

- Vickers micro hardness test
- Scanning electron microscope (SEM)
- X-Ray Powder Diffraction (XRD)

-Vickers micro hardness test.

This is the main test for the studies which determined how much the thickness of the coating. This is are the test process:

- i. The sample substrate that cover with coating need to be cut down in each two side of the round shape sample in order to get the cross section of the coating and make it easily stand during the micro hardness test.
- ii. Using polishing machine, the cross section need to be polished using different grade of sand paper starting from low grade (400) which is the rough and to higher grade (1400) which is the smoothest sandpaper.
- iii. Then, set up the substrate with coating below the microscope of the Vickers micro hardness test machine. Use clay to support the substrate to stay still while analyzing it.
- iv. Then set up the machine with different type of load starting from 50 gf and undergo the test with 5 onwards vertical location at the cross section.
- v. The test will repeated using different load of 100gf, 200gf, 300gf, 500gf and 1000gf with the same 5 vertical parallel location at the cross section.
- vi. All the measured data will be obtain and analyze.

-Scanning electron microscope (SEM)

In SEM test there are two type of analysis need to be done that is

- i. Analysis on surface structure of coating and uncoated substrate
- ii. Analysis of cross section coating substrate to detect the porosity and oxide stringer of the coating.

The sample need to be cut down towards the SEM allow test size that is 20mm square size then it can be analyze .All the analyze micro structural pic will be captured and analyze.

- X-Ray Powder Diffraction (XRD)

For XRD test, the same size sample needed to undergo the test that is 20mm square size. After cutting the sample and XRD test can be performed and the result will be analyzed.

Details/Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Preparing Sample														
Coating Sample Process							•							
Progress Report Submission							•							
Project Work Continues														
Pre SEDEX & Poster Exhibition														
Technical Paper & Final Draft Submission												•		
Final Oral Presentation/VIVA														•
 Suggested Milestones Process 														

3.3 Gantt Chart Progress

3.4 Key Milestones Progress



CHAPTER 4 RESULT AND DISCUSSION

This chapter will indicated the result and finding based on the test been conducted

4.1 Vickers Microhardness Test Result

Load	Microhardness Reading (HV)									
(gf)/position	1	2	3	4	5					
1000	409.9	326	313.7	301.2	273.2					
500	384.6	355.2	320.3	303	239					
300	418.3	373.8	288.3	275.2	279.6					
200	549.9	450.3	329.1	265.6	264.2					
100	462.7	451.3	320.6	281.9	281.2					
50	579	493.3	367.2	302.4	265.7					

Table 4.1: Vickers Microhardness Test Reading



Figure 4.1: Vickers Mircrohardness Test Load vs Reading



Figure 4.2: SEM microstructure view of positioning of indentation made across the coating cross section

-In this result microhardness reading been taken across the cross section has been illustrated in figure and microhardness reading were observed and shown on table above. It is shown that based on the different position of the indentation of the diamond shape, the Vickers micro hardness test detect that the subtract cover with coating have high and subtract without coating have low indication of hardness value (HV). Using different load also indicated that using high loads can indicate low value of value when the diamond shape indentation will create large indication resulting low harness value (HV). Based on position, Position 1 indicated that it have highest hardness value (HV) reading as it was the coating covered on the top layer surface which are for 50gf is around 580 HV and for 1000gf is around 400gf. Then we can see decreasing value of hardness value (HV) from position 1 towards position 4 regardless what are the load been used. From position 4 to position 5, it indicated that it is the substrate area where coating no longer exist which would have hardness value reading of 200-300 HV.

500gf and load 200gf also have high reading hardness value compared to load 100gf. This is due to inconsistency of the surface thickness profile along the coating surface which affected by the work of the sand blasting and the HVOF process towards the surface that cause this minor error.

4.2 Scanning Electron Microscope

4.2.1 Surface microstructure



Figure 4.3: Uncoated microstructure surface 50µm



Figure 4.4: Coated microstructure surface 50µm



Figure 4.5: Particle view of coating 10µm

-From figure above, the application of chromium carbide coating Cr_3C_2 -25NiCr that been applied to AISI 304 stainless steel substrate through HVOF process will be creating an uniform, formed and dense protecting layer on top the surface substrate. Based on the coated microstructure surface view also "splats" can be detected while on the uncoated doesn't have. "Splat" occur during the HVOF process the chromium carbide coating Cr_3C_2 -25Nicr when impacted towards the substrate revealing lamellar structure and solidify to another form continuous coating protective layer. The particle size of powder observed for this study respond to the other articles [26] and Ji et al [27].



Figure 4.6: Uncoated substrate composition



Figure 4.7: Coated substrate composition



Figure 4.8: Microstructure image mapping of mixed composition at $40 \mu m$



Figure 4.9: Microstructure image mapping of chromium composition at $40\mu m$



Figure 4.10: Microstructure image mapping of carbon composition at $40\mu m$



Figure 4.11: Microstructure image mapping of oxygen composition at $40\mu m$



Figure 4.12: Microstructure image mapping of nickel composition at 40µm



Figure 4.13: Microstructure image mapping of iron composition at 40µm

-Figure above show the different between the composition of uncoated and coated substrate. Based on the reading before been coated carbon was the major composition. Then after the substrate been coated, chromium become the major composition follow by carbon, iron and nickel.

4.2.2 Porosity and oxide stringer of the coating.



Figure 4.14: Cross Section view of coating obtained by SEM at 5000x magnification



Figure 4.15: Standard images for Porosity rating on Metallographic specimens [24]



Figure 4.16: Cross section view of Cr3C2-25-NiCr coating obtain by SEM at 1000x

-Four images as in figure above were obtained by SEM, and the images are compared to the standard images from ASTM E2109 as illustrated in figure 29.Based on the images obtained, the porosity of the coating average about 3.0%.This is similar from

portray result from Sidhu[25] in were author have higher percentage of porosity from 2.5% to 3.5%. The low porosity of coating is needed as the thermal spray process allow the coating to be dense and become better protective layer. In the coating also there also have the presence of oxide stringers this occur during the collect and harden of the coating compound after undergo the HVOF process.



4.3 X-Ray Powder Diffraction (XRD)

Figure 4.17: XRD analysis 1



-In this test, phased structured being analyzed by XRD and the finding can determine the characterization of crystalline material that's has within the coating. In this test, proves the coating chromium carbide nickel chrome have high reading of Cr_3C_2 and NiCr lower. It shows that chromium carbide is the major and nickel chrome is the minor crystalline material and the combination both prove can create chromium carbide nickel chrome Cr_3C_2 -25NiCr as a protective layer.

CHAPTER 5 CONCLUSION & RECOMMENDATION

This chapter will conclude everything that had been done throughout the process from doing the literature reviews, preparing experimental procedure and making the proposal report.

5.1 Conclusion

Based on the microstructural study been conducted, when undergo HVOF thermal spray process and used chromium carbide nickel chrome as the coating powder Cr_3C_2 -25NiCr it can produce better protecting layer. This are the conclusion obtained based on the study:

- The vickers micro hardness test show that the Cr3C2 NiCr coating have high value of hardness which mean the coating can be consider hardest and is valuable as protective shield.
- Using high load of 1000gf hardness value of the coating is about 400HV and using low load of 50gf the harness value is high 579HV.Here the difference of hardness value in not that far as the use of different load is huge and show the coating of chromium carbide using HVOF thermal spray process can produce hard protective coating.
- HVOF thermal spray chromium carbide nickel chrome coating (Cr3c2-25NiCr) produce dense and lamellar structure of coating.
- The coating also have low percentage of porosity that is 3% to help increasing the hardness by creating denser protective layer with small pores.
- HVOF thermal spray process produce coating with low concentrated oxide stringers and reduce brittleness of the coating that been created.

5.2 Recommendation

Then in terms of the studies that can be carried out in the future, student can do comparison of microstructural studies between different types of coating for example Cr_3C_2 -NiCr vs Al₂O₃.Student also can do a comparison study using the same coating powder with different type of thermal spray process for example HVOF vs plasma coating. In order to safe time during undergo the project, the sample material and the coating powder need to be prepared and purchased early to ensure it is available. This is to ensure the equipment that needed are available such as for cutting the sample and coat it within the target time. The availability of the machinery need to be booked one month early to avoid any held up on the experiment studies. This is because the university equipment often been used for other experiment studies in the university and the quantity of the equipment itself is limited.

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