

A STUDY OF THE MICROSTRUCTURE PROPERTIES OF COLD SPRAYED 6061 AL ON 7075-T6 ALUMINUM



BACHELOR IN MANUFACTURING ENGINEERING TECHNOLOGY (PROCESS AND TECHNOLOGY) WITH HONOURS



Faculty of Mechanical and Manufacturing Engineering Technology



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Bachelor of Manufacturing Engineering Technology (Process and Technology) with Honours

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DECLARATION

I declare that this project entitled "A study of Microstructure Properties Of Cold Spray 6061 AL on 7075-T6 Aluminum" is the result of my own research except as cited in the references. The project report has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.



APPROVAL

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the Bachelor of Manufacturing Engineering Technology (Process and Technology) with Honours.

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DEDICATION

This dissertation is dedicated to my beloved parents, my supervisor Dr. Noor Irinah Binti Omar and to those who are unwavering affection, guidance and encouragement have enriched my soul and driven me to undertake and complete this work.



ABSTRACT

Deposits are created in the cold spray (CS) process by depositing powder particles at high velocity onto a substrate. CS-deposited powders do not melt before or after contacting the substrate. Because of this property, CS is useful for the deposition of a wide range of materials, most notably metallic alloys, but also ceramics and composites. The particles undergo extreme plastic deformation during processing, resulting in a stronger mechanical and less metallurgical connection with the underlying material. An individual particle's deformation behaviour is determined by a number of material and process factors, which are categorised into three broad groups: powder properties, geometric parameters, and processing parameters, each having their own subcategories. Changing any of these factors causes the microstructure to evolve and, as a result, the mechanical characteristics of the deposit to vary. While cold spray technology has advanced over the last decade, the process is intrinsically complicated, thus the effects of deposition parameters on particle deformation, deposit microstructure, and mechanical characteristics are yet unknown. The goal of this research is to investigate the microstructure characteristics of cold sprayed 6061 Al on 7075-T6 aluminium. The resistance, physical, and microstructure characteristics of 7075 Al-T6 are affected by factors such as coating temperature, pressure, coating thickness, and particle size. In this study, there is FIVE Testings have been carried out which are Scanning Electron Microscope analysis (SEM), Energy Dispersive X-Ray Spectroscopy (EDX), Tensile strength testing, Microhardness testing and Transmission Electron Microscopy (TEM) testing are used to assess the effectiveness of the CS coating process.

ABSTRAK

Deposit dicipta dalam proses semburan sejuk (CS) dengan mendepositkan zarah serbuk pada halaju tinggi ke atas substrat. Serbuk yang didepositkan CS tidak cair sebelum atau selepas menyentuh substrat. Disebabkan sifat ini, CS berguna untuk pemendapan pelbagai bahan, terutamanya aloi logam, tetapi juga seramik dan komposit. Zarah mengalami ubah bentuk plastik yang melampau semasa pemprosesan, menghasilkan sambungan mekanikal dan kurang metalurgi yang lebih kuat dengan bahan asas. Tingkah laku ubah bentuk zarah individu ditentukan oleh beberapa faktor bahan dan proses, yang dikategorikan kepada tiga kumpulan luas: sifat serbuk, parameter geometri dan parameter pemprosesan, masingmasing mempunyai subkategori mereka sendiri. Mengubah mana-mana faktor ini menyebabkan struktur mikro berkembang dan, akibatnya, ciri mekanikal deposit berubah. Walaupun teknologi semburan sejuk telah maju sejak sedekad yang lalu, proses ini secara intrinsik rumit, oleh itu kesan parameter pemendapan pada ubah bentuk zarah, struktur mikro deposit, dan ciri mekanikal masih belum diketahui. Matlamat penyelidikan ini adalah untuk menyiasat ciri-ciri mikrostruktur semburan sejuk 6061 Al pada aluminium 7075-T6. Ciri rintangan, fizikal, dan mikrostruktur 7075 Al-T6 dipengaruhi oleh faktor seperti suhu salutan, tekanan, ketebalan salutan, dan saiz zarah. Dalam kajian ini, terdapat LIMA Ujian telah dijalankan iaitu analisis Mikroskop Elektron Imbasan (SEM), Spektroskopi X-Ray Penyebaran Tenaga (EDX), ujian kekuatan tegangan, ujian Microhardness dan ujian Transmission Electron Microscopy (TEM) digunakan untuk menilai keberkesanan proses salutan CS.

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LIST OF SYMBOLS AND ABBREVIATIONS

SEM	- Scanning Electron Microscope Analysis
EDX	- Energy Dispersive X-Ray Spectroscopy
TEM	- Transmission Electron Microscopy
CGDS	- Cold Gas Dynamic Spray
HPCS	- High Pressure Cold Spray
LPCS	- Low Pressure Cold Spray
ASTM	- American Society for Testing and Material
NHT	- Non Heat-Treatable
HT	- Heat-Treatable
kV	Kilo Volt
HV	- Vickers Pyramid Number
PSI	- Pound per Square Inch
kN	- Kilo Newton
GPa	- Gigapasccal
MPa	اونىۋىرىسىتى تىكىنىكى Megapascal مالاك
Al	- Aluminium
С	UNIVECTIONI TEKNIKAL MALAYSIA MELAKA
0	- Oxygen
CNC	- Computer Numerical Control
HVOF	- High Velocity Oxyfuel
CS	- Cold Spray
Ø	- Diameter
%	- Percentage
σ	- Stress
3	- Strain
Κ	- Kelvin
Mg	- Magnesium
Zn	- Zinc

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CHAPTER 1

INTRODUCTION

1.1 Background

The cold gas dynamic spray (CGDS) technique is commonly referred to as 'Cold Spray.' Dr. Anatolii Papyrin and his colleagues in Novosibirsk, Russia, invented it in the mid-1980s (Papyrin et al. 2006). This cold spray procedure is ideal for applications where high-temperature spraying processes such as plasma, arc, flame, and HVOF (High Velocity OxyFuel) spray are ineffective due to concerns such as oxidation, coating porosity, and low adherence (Srikanth & Bolleddu, 2020).

Cold Sprayed Coating comes in two types. The first is high-pressure cold spray coatings, in which powder particles are injected into the throat of the spraying nozzle from a high pressurised gas supply, resulting in particle velocities of 800–1400 m/s. For high-pressure cold spray coatings, helium or nitrogen are the recommended propellant gases. The second type of coating is low-pressure cold spray coatings, which involve injecting powder particles from a lower pressurised gas through the diverging section of the spraying nozzle.

A high-pressure supersonic gas jet is used in the CS process to accelerate fine powder particles to or above a critical velocity (500–1200 m/s) for coating deposition. The kinetic energy released by the particle upon impact with the substrate ruptures any surface oxides and plastically deforms the particle as it approaches the clean surface of the substrate, promoting coating bonding (Singh et al., 2021).

A CS coating is formed in two steps: (i) initial particle-substrate contact, followed by (ii) particle-particle interactions. Bonding/adhesion is achieved at the interface of the substrate and the first layer of particles in the first step, followed by the formation of subsequent layers by particle-particle interactions (Singh et al., 2021). Layer-by-layer formations create thick coatings.

The existence of residual stress in the coating and near the coating/substrate interface can influence the mechanical performance of CS deposits, as it does in all thermal spray processes. In the case of HPCS, SPD creates residual stress in the CS deposition, and microscopic evaluation of the residual stress profile (on a particle size) is difficult and needs understanding of various deposition process features, such as non-uniform local deformation and recrystallization. The particles deposited by cold spraying distort initially on collision, resulting in adhesion to deposited particles, followed by subsequent deformation produced by the impacts of incoming particles, resulting in a tamping action. This mechanism explains why the coatings are dense and compact. (Rokni et al., 2017)

1.2 Problem Statement

Fitting sponson spar is often the main structural member of the wing, running span wise at right angles to the fuselage of Nuri Helicopter. The spar carries flight loads and the weight of the wings while on the ground. Other structural and forming members such as ribs may be attached to the spars, with stressed skin construction also sharing the loads where it is used (Bruce,2006). Fitting sponson spar Nuri helicopter is made from aluminium 7075-T6 but premature failure due to corrosion is one of the main challenges associated with this alloy and the most common effect of corrosion on aluminium alloys is called pitting. It is first noticeable as a white or grey powder deposit, like dust, which blotches the surface (Noor irinah, 2012). Beside corrosion problem, structural restoration that involved mechanical and microstructure properties also another problem to be considered. To date, there has been no detailed investigation of application high pressure cold spray process as dimensional

restoration for Malaysian aging aircraft in term of microstructure properties toward adhesion bonding mechanism.

1.3 Research Objective

The main objectives for this project are:

- To investigate microstructure properties of 7075-T6 cold sprayed 6061Al via Scanning Electron Microscopy (SEM-EDX).
- 2. To study the correlation between microstructure properties and adhesion bonding mechanism of cold sprayed 6061 Al on 7075-T6

Al substrate.

1.4 Scope of Research

The scope of this research is shown in figure 1 below. The objectives of this research to investigate the microstructure properties of cold sprayed 6061-Al powder on 7075-T6 substrate and to relate the properties with bonding mechanism involved. The scope of this project is to study the properties of sample after undergo cold spray treatment. Aging aircraft part (Fitting sponson spar of Nuri Helicopter) is used as a sample in this project. It is made from Aluminium 7075-T6 but premature failure due to corrosion effect. Besides corrosion, dimensional restroration that involved mechanical and microstructure properties also another problem to be considered. Therefore, the substrate will be coated by using High Pressure Cold Spay (HPCS) process whereby, Aluminium 6061 powder (Valimet 6061 Al) is deposited onto the surface of the substrate. In this process, powder feestock material (Valimet 6061) with nominal particle size 3 microns is injected into the gas stream and accelerated towards the substrate (Al 7075-T6).

In general, this work study is more focused on the characterization and measurement of the sample after the CS process. The evaluation of this research is based on five standard tests which are Scanning Electron Microscope analysis (SEM), Energy Dispersive X-Ray Spectroscopy (EDX), Tensile strength testing, Microhardness testing, Transmission Electron Microscope (TEM). All the tests are a fundamental of material testings. Hence, the result from the test are commonly used for quality control and used to predict how the material will react under these type of many condition such as forces.



Figure 1: Project Work scope

1.5 Conclusion

This chapter covers the research background, problem statement, objectives and scope of the research. The following chapter will consist review of main theories and describe previous works related to this research.



CHAPTER 2

LITERATURE REVIEW

This chapter provides a review of the concept of microstructure properties of 7075-T6 cold sprayed on 6061 aluminium by using high pressure cold spray process. This chapter will also include the information about parameter involves to be exposed to the concepts and theories. The main sources of information are taken from books and journal articles. Each source was selected based on the similarity with the scope of study. The elements will be narrowed down to the analysis of the characterization and measurement of coating specimen.

2.1 Cold spray process

ALAYS!

In this chapter, the basic principle, invention of cold spray technology and advantages of cold spray system will be explained to give some practical information on technologies and equipment's as well as to present the current state of the research and development in this field.

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2.1.1 Introduction

Cold spraying, CS is becoming increasingly important due to its superior qualities over other thermal spraying methods such as flame spray, plasma spray, HVOF, and arc spraying, among others. The fundamental distinction between cold spraying and other methods is that it uses kinetic energy rather than thermal energy for powder deposition, and the operating temperature is always kept below the melting point of the powder particles (Srikanth & Thalib Basha G A, Venkateshwarlu, 2019).

The cold spray procedure is also known as solid-state coating deposition because the powder particles remain solid throughout coating deposition, eliminating the production of defects due to thermal distortion. Temperatures in this method are typically below 8000 °C, although temperatures in other thermal spraying processes can occasionally exceed 20000 °C. The impingement velocity of the powder particles on the substrate, on the other hand, is about more than 1200 m/s. Cold spray is preferred for depositing materials with lower heat capabilities, are softer, and are oxygen sensitive, such as copper, aluminium, and titanium. This approach can reduce oxidation, coating porosity, phase transitions, heat affected zone (HAZ) development, and thermal residual stresses, all of which are significant issues in traditional thermal spray coating procedures. This cold spray method may also deposit highly dense and thick coatings with thicknesses ranging from 100 m to 1500 m (Srikanth & Thalib Basha G A, Venkateshwarlu, 2019).

The cold spray procedure is employed not only for coating deposition, but also for crucial component maintenance. Thus, it is currently establishing itself as an alternative solution for difficult part repair, particularly in the aerospace, defence, and turbine industries. This process has the potential to outperform conventional technologies such as 3D printing, welding, and electro plating. The main benefits of using cold spray as a repairing solution are: (i) improved fatigue properties of the base metal, (ii) residual compressive stresses in the materials, which can prevent cracking from starting, (iii) worn out workpieces can be repaired by same deposition process and placed in the same position, (iv) on-site repairing is possible with the portable cold spray system, and (v) higher coating deposition quality (Srikanth & Thalib Basha G A, Venkateshwarlu, 2019).

As this process continues a uniform layer with little porosity and high bond strength is obtained as the particles continue to impact the substrate and a bond with the material stored has formed. The term 'cold spray' was used to describe this process as relatively low temperature gas flow to grow around the nozzle. In other term, it is referred to the temperature that is always lower than melting point of the material during acceleration of powder particles by the supersonic gas jet. Figure 2.1 shows the schematic diagram of cold spray process.



Figure 2.1: Schematic Diagram of Cold Spray Process (Srikanth & Thalib Basha G A, Venkateshwarlu, 2019)

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Like other materials processing technique, cold spray process also has its own advantages and limitations. The primary advantage of the cold spray process over traditional thermal techniques that rely on a variety of processes like laser, electron beam, plasma, or electric arc) to melt and/or soften feedstock had something to deposition is that consolidation happens in the solid state. The powder particles blasted during the cold spray process remain reasonably cool, below the melting temperature, eliminating oxide contamination, microstructure changes, and tensile stress accumulation.

2.1.2 History of Invention

The cold gas dynamic spray (CGDS) technique is commonly referred to as 'Cold Spray.' Dr. Anatolii Papyrin and his associates in Novosibirsk, Russia, invented it in the mid-1980s (Srikanth & Bolleddu, 2020). The first USA patent on CS technique was issued in 1994, and it concerned the practicality of the CS process for a variety of applications (Alkhimov et al., 1994). Many additional writers later patented the design of various parts of the cold gas dynamics spray system, including de Laval nozzle design, coating materials, and applications (Kumar et al., 2020).

However, this procedure drew the interest of scholars and companies all over the world, and many trademarks were filed in the years that followed. At the beginning of the twenty-first century, there was a real economic development of CS, and the number of studies reported on CS expanded significantly. Furthermore, CS has been applied in a variety of technological applications. It has been used to deposit protective coatings in a variety of industries, including aerospace engineering, automotive, geology, and power production, to prevent component damage and increase component service life (Poza & Garrido-Maneiro, 2022).

Cold spray process development has resulted in positive coating process engineering. As a result, several diagnostic techniques have been created to better understand and optimize the performance of this process. Many academic institutions and firms from various nations have undertaken substantial research on this technique. The cold spray procedure has been shown to be a viable coating process for producing a protective coating, performance boosting layers, ultra-thick coatings, free forms, and near net shapes. Furthermore, current research has focused on optimizing cold spray parameters to generate coatings of various materials with appropriate microstructures.

2.1.3 Types of Cold Spray Process

There are two types of cold spray processes based on patented injection methods: low pressure cold spray (LPCS) and high-pressure cold spray (HPCS). The major difference between the two types of systems is the feed gas pressure and the location of the powder input, which affects the particle velocities obtained. Powder particles are delivered into the diverging region of the nozzle for LPCS, and particle velocities of 300-600 m/s² are attained. In HPCS, the powder particles are injected in the nozzle's converging region, and the acquired velocities can be as high as 800 - 1400 m/s². LPCS systems normally acquire nitrogen or air as the carrier gas, whereas HPCS systems either helium or nitrogen (Evans, 2018).

2.1.3.1 Low Pressure Cold Spray Process

Low-pressure cold spray coatings: In these coatings, powder particles are injected from a lower pressurized gas through the diverging section of the spraying nozzle. The devices used to apply these coatings are usually smaller and portable, with particle velocities ranging from 300 to 600 m/s. Nitrogen or widely obtainable surrounding air are the recommended propellant gases for low-pressure cold spray coatings (Srikanth & Bolleddu, 2020).



Figure 2.2: Low Pressure Cold Spray Schematic (Evans, 2018)

2.1.3.2 High Pressure Cold Spray

High-pressure cold spray coatings: In these coatings, powder particles are injected into the throat of the spraying nozzle from a higher pressurised gas supply, resulting in particle velocities in the 800–1400 m/s range (Srikanth & Bolleddu, 2020). It generates greater particle velocity (1200 m/s or more) by using light weight gases (Nitrogen or Helium), high pressure gas (40 bar), and preheating temperature (800°C). Depending on the process parameters, the temperature of the particle upon impact can reach 1000 K or higher (Kumar et al., 2020).



Figure 2.3: High Pressure Cold Spray Schematic (Evans, 2018)

High pressure cold spray systems can generally operate at the same pressures as lowpressure cold spray systems; however, this is not always the case due to the unique design of low-pressure cold spray systems. The low-pressure system differs from the high-pressure system in two ways: the low-pressure compressed gas is normally replaced by a portable air compressor; and the powder injection point is at the nozzle divergent section, where the local gas pressure is sufficiently low to allow powders from the powder feeder to be released at atmospheric pressure (Yin et al., 2018).

Because of these characteristics, low pressure cold spray systems are more adaptable and far less expensive in terms of both equipment and processing expenses than high pressure cold spray systems. Because of the portability of the simpler low pressure cold spray systems, they are particularly suited to the repairing of the damaged components. However, because particle velocity in low pressure cold spray systems is much less than in high pressure cold spray systems, the use is severely limited, and only a limited range of materials, such as copper and aluminium, may be deposited using a low-pressure cold spray system. As a result, the word "cold spray" usually refers to "high pressure cold spray," while "low pressure cold spray" must be emphasized (Yin et al., 2018).

2.2 Aluminium

This project study is to investigate the microstructure properties of cold sprayed 6061-Al powder on 7075-T6 substrate and to relate the properties with bonding mechanism involved. Therefore, it is needed to analyse and study the structure of aluminium and its alloy properties.

2.2.1 Introduction

Aluminium and its alloys are now used in a variety of applications. The aerospace industry was among the first to identify and apply the physical qualities of aluminium alloy in its parts. It is generally applied since no other materials can compete with it. Aluminium alloys have been widely and effectively used to manufacture anything from airships to modern aircraft. Aluminium alloys are also implemented in the rail sector, automobile industry, offshore industry, and shipping business.

Aluminium metal composites outperform unreinforced alloys in terms of elastic elasticity, hardness, and tensile strength at room and extreme temperatures, as well as significant weight savings. These features of metal matrix composites based on aluminium are used in aerospace, automotive, and marine applications. To improve metal properties, Al₂O₃, SIC, B₄C, TiC, TiB₂, MgO, TiO₂, FlyAsh, BN, and Graphite are employed as particle reinforcement in metal matrix composites (Vijaya Bhaskar et al., 2018).

Finally, aluminium alloys have limitations such as a high coefficient of thermal expansion and poor tribological properties. Adding material for reinforcing and enhancing univERSITITEKNIKAL MALAYSIA MELAKA and therefore modelling specific aluminium composites results in increased stiffness and toughness, fatigue resistance, and enhancement of tribological qualities (Stojanovic et al., 2018).

2.2.2 Properties of Aluminium

The main properties of aluminium can be identified in Table 2.1 below:

Property	Value	Notes	
Atomic number	13		
Atomic volume	10 cm ³ / g-atom		
Atomic weight	26.98		
-	10		

Table 2.1: Properties of pure aluminium (Alam & Ansari, n.d.)

Coeff of Thermal Expansion	$a = 23.5 \times 10^{-6/0} C$	$a_{200} = 26 \ge 10^{-6/\circ}C$ at 200°C
Density	$P \sim 2.7 \text{ g/cm}^3$	$\sim 2700 \text{ kg/m}^3$ at 20°C
Electrical Resistivity	$R = 2.69 - 2.824 \mu\Omega cm$	At 20°C ambient temperature
Elongation	~ 50%	Maximal before breaking
Hardness	BHN =15 Brinell	
Modulus of Elasticity	$E = 69 kN/mm^2$	$E_{100} = 67 \text{kN/mm}^2 \text{ at } 100^{\circ}\text{C}$
		$E_{200} = 59 \text{kN/mm}^2$ at 200°C
Modulus of Rigidity	$G = 26 kN/mm^2$	G = E/2(1+v)
Point of Melting	$\sim 600^{\circ}C$	
Point of Boiling	$\sim 1800 - 2480^{\circ}C$	
Poisson's Ratio	v = 0.33	Baker and Roderick, 1948
Proof/Yield Stress	$f_{\gamma} = \langle 25N/mm^2 \rangle$	
Thermal Conductivity	$K = 240 \text{ W/m} \circ \text{C}$	At 20°C ambient temperature
Ultimate Tensile Strength	$f_{\gamma ult} = <58 N/mm^2$	
Specific Heat	$c = 22cal/g \ ^{o}C$	
Valency	3	

Based on its physical properties, aluminium can be characterized as lightweight material, high corrosion resistance, ease of fabrication, non-toxic material, non-magnetic, good electric conductivity, and heat conductivity, withstands extreme environmental conditions, high reflective of light and recyclability due to its low melting point.

2.2.3 Types of Aluminium and its alloy

Alloying and subsequent treatment can strengthen pure aluminium. Aluminium alloys are classified into two types: wrought alloys and cast alloys, which are further classified into eight series 1xxxx-8xxxx. Another distinction is the distinction between heat-treatable (HT) and non-heat-treatable (NHT) alloys, as defined by British Standards. As guideline on the aluminium designations, BE EN 515 is used for wrought aluminium and BE EN 1780 is used for cast aluminium alloys. The numerical designation system will be shown in Tables 2.2 and 2.3:

Aluminium Alloy	Principle Alloys	Туре
Designation	Elements	
1XXXX	99% Pure	NHT
2XXXX	Cu	HT
3XXXX	Mn	NHT
4XXXX	Si	NHT
5XXXX	Mg	NHT
6XXXX	Mg and Si	HT
7XXXX	Zn	HT
8XXXX	Others	
HT = heat-treatable	e, NHT = non-heat-treatable	

 Table 2.2: The numerical designation system for wrought aluminium alloys

 (Alam & Ansari, n.d.)

 Table 2.3: Numerical designation system for cast aluminium alloys

 (Verma & Kumar Lila, 2021)

Series	Alloying elements
2 1XXXX	None (aluminium 99% and greater)
E 2XXXX	Copper (Cu)
3XXXX	n/a
4XXXX	Silicon (Si)
5XXXX	Magnesium (Mg)
6XXXX	n/a
TXXXX a	Zinc (Zn)
8XXXX	Tin (Sn)
UNIVE9XXXXTE	KNIKAL MALAYSIA Master alloys
$\mathbf{AC} = \mathbf{C}$	ast Aluminium Alloys

The first digit in the identification refers to the alloy group and yet is associated with the primary alloying element applied. Unless the digit is 0, the second digit identifies a modification from the specific alloy, while the last two digits identify the alloy in its series.

For the current aluminium alloy, five basic temper designations are applied, and all temper names, specifications, and attributes may be found in the British Standard BS EN 515. The letters F, O, H, W, and T represent it. Table 2.4 below contains an explanation of the fundamental temper designation system:

Letter	Description	Meaning
F	As fabricated	Forming process with no special
		control over thermal or strain
		hardening.
0	Annealed	Heat treated to give min, strength
		improving ductility and
		dimensionality.
Н	Strain hardened	Strengthened by cold working
W	Heat treated	Solution heat treated but produces
		an unstable temper.
Т	Heat treated	Thermally heat treated with or
		without additional strain hardening.

 Table 2.4: Basic temper designation (Verma & Kumar Lila, 2021)

The strain hardened alloys (H) and thermally heat-treated alloys (T) groups are further split based on the treatment or treatment combinations used. Table 2.5 contains an explanation for this:

Table 2.5: Temper designation system to current standards(Verma & Kumar Lila, 2021)

Temper destination (Based on guidance given in BS EN 515)				
XXXX	2Fro l	As fabricated	NHT	
	-0	Fully annealed (softened by heating)	NHT	
	-H1	Strain (Work) – hardened only	NHT	
	-H2	Strain (Work) – hardened and partially	NHT	
		annealed		
	-H3	Strain (Work) – hardened and stabilized (by	NHT	
		low temperature treatment)		
	-H4	Strain (Work) – hardened and lacquered or	NHT	
		painted		
	HX2	Quarter-hand	NHT	
	HX4	Half-hard	NHT	
	HX6	Three-quarter-hard	NHT	
	HX8	Fully hard	NHT	
	-T1	Cooled from an elevated temperature shaping	HT	
		process		
	-T2	Cooled from an elevated temperature shaping	HT	
		process, cold worked and naturally aged		
	-T3	Solution heat-treated, cold worked and	HT	
		naturally aged		
	-T4	Solution heat-treated and naturally aged	HT	

-T5	Cooled from an elevated temperature shaping	HT	
	process and artificially aged		
-T6	Solution heat-treated and artificially aged	HT	
-T7	Solution heat-treated and overly aged	HT	
-T8	Solution heat-treated, cold worked and then	HT	
	artificially aged		
-T9	Solution heat-treated, artificially aged and	HT	
	then cold worked		
HT = heat-treatable / NHT = non-heat-treatable			

The aluminium alloy series that involve in this project study is Al 6061 and Al 7075-T6. The 6xxx alloys are heat-treatable magnesium and silicon alloys. It is extremely strong and weldable. The extrudability of Al 6061 alloy, which is ideally suited for architectural and structural elements, good resistance with high strength is a distinguishing quality (Verma & Kumar Lila, 2021). This series of material are weaker compared to mild steel and apparently less ductile.

For 7xxxx series, these aluminium / zinc alloys are (with zinc additions varying from 0.8 to 12.0 percent) and are among the strongest aluminium alloys. These alloys are generally applied in high-performance applications like aircraft, aerospace, and competitive sporting equipment (Verma & Kumar Lila, 2021). Its mechanical qualities are substantially improved over the 6xxxx series alloys, and HAZ softening at the weld is less severe, however it is unsuited for arc welding, has less extrudability than the 6xxxx series, and is difficult to construct. As a result, producing and fabricating it necessitates a high level of knowledge and extensive experience.

2.3 Coating

Coating is one of the strategies that may be used to protect metal surfaces from wear and tear. To examine the application of coating, it is required to analyse and understand the attributes of the complete system, such as the character of the metal substrate, the surface pre-treatment conditions, and the application techniques. This project is chosen and mainly focuses on cold spray coating as the relationship between microstructure qualities and adhesion bonding mechanism.

2.3.1 Bonding Mechanism in Cold Spray

The topic of discussion is the real and sensible method by which solid powder particles change their shape and size permanently and a bonding is created. Several hypotheses have been offered by various researchers. Through a widely accepted model, it is likely that solid particles endure permanent deformation that may damage thin material surface layers like oxides that impart intimate contact under very high local pressure, allowing bonding to occur. In the past, a wide range of materials, including Cu and Al, have been plastically deposited by the CS approach, indicating that interface blending is primarily determined by the hardness of the substrate, density, and powder particle velocity (Kumar et al., 2020).

For metallic materials, the general form of interface bonding can be defined by the contact area, time, temperature, and recoverable resilience under diverse adiabatic shear instability circumstances. [Nikbakht et al. (2018)] investigated the asymmetrical bonding of two different materials in cold gas dynamic spraying (Ni and Ti). The experimental results demonstrated that when the impact velocity increased, the bonding increased from the periphery to the centre of the surfaces (Kumar et al., 2020).

2.3.1.1 Coating Build-up

In the case of the CS process, there are various points of view on coating build-up. First point is: The adhesive bond can be formed when the surface fusing and formation of pure metal surfaces in the metal jet occur in a manner like explosive welding. Second point: An adhesive bond can be formed when melting does not occur but a significant fracture and deformation of oxide coatings on metal particles occurs, allowing pure metals (Cu, Ag, Au, Ni, etc.) to come into touch with each other (Kumar et al., 2020).

Besides for very thin metal coatings, the cold spray technology can be imagined as a process that involves two phases: The coating is generated when the initial layer of solid powder is sprayed on the substrate. The solid powder particles interact with the substrate during the first phase. To improve, grit blasting is frequently performed prior to coating deposition. Sand blasting is avoided in several circumstances, particularly on softer surfaces where grits can contaminate the surfaces. The sprayed metallic powder particles are solid during the CS coating process. Previously, solid sprayed metallic powder particles might be utilised for substrate preparation and pre-treatment, especially when sand blasting is harmful to the surfaces. The cold-spray coating build-up mechanism by splitting the coating growth process into four basic sub-steps (Figure 2.4) (Kumar et al., 2020).



Figure 2.4: Coating growth process in four steps (Kumar et al., 2020).

Step 1: Cratering of substrate and building up of first layer of particles.

Step 2: Multilayer coating build-up.

Step 3: Particle-particle bonding and void reduction.

Step 4: Densification and work hardening of the coating.

As illustrated in Figure 2.4, the volume of displaced material, which is usually metal, is comparable to the difference between the original volume of the particle before deformation and the volume of final powder particle. When the density of particles increases, the material displaces at the point of contact. The contact area expands in dimension, and material flows into the inter-particle spaces, filling the voids. This phenomenon known as "peening" (Kumar et al., 2020).

2.3.1.2 Particle Deformation Behaviour

Particle deformation is a natural feature of CS deposition, influencing particle and substrate interaction, surface topography, and metallurgical processes (such as work hardening) within the deposit. Because deposit formation is based on plastic deformation caused by particle collisions, particle deformation behaviour is critical during the CS deposition process. During CS deposition, the extent of particle deformation is determined by two key parameters: particle velocity and particle temperature. Particle deformation parameters are those that influence particle velocity and temperature (Xie et al., 2019). This can be classified in three categories:

- (1) Powder characteristics (size, shape and state, surface oxide layer)
- (2) Geometric effects (spraying standoff distance, incidence angle, and nozzle geometry)
(3) Process parameters (gas type, temperature and pressure, substrate hardness, temperature, and surface roughness).

2.3.1.3 Bonding mechanism in metallic cold spray that consist of mechanical bonding and metallurgical bonding.

The cold-spraying (CS) approach, a relatively new technology, can construct the sample via solid-state deposition of feedback powders without melting or solidification. Powders are propelled to high velocity in this process by the effect of supersonic flow. Bonding is accomplished through the extensive plastic deformation of solid-state particles caused by high-velocity collision at temperatures below the melting point. Cold spray, because of its distinctive 'cold' feature, can reduce or eliminate significant oxidation, phase transformations, and thermal stress and can be widely employed for the manufacture of functional coatings, metallic matrix composites, manufacturing, and dimensional damage restoration (Xie et al., 2019).

The bonding mechanism is usually a hot issue in cold spray to increase coating quality. Mechanical and metallurgical bonding are currently regarded as the primary bonding mechanisms in cold spray. Metallic bonding is generated as a chemical reaction between deposited particles or the interface between coating and substrate at the oxide-free interface and metal-to-metal contact. Metallurgical bonding is thought to offer relatively high adhesion strength in cold-sprayed coating, as evidenced by the presence of intermetallic, amorphous phases, or dimple-like features at the fracture surface of the coating or a single splat (Xie et al., 2019).

The most plausible mechanism of metallurgical bonding creation is the increase of adiabatic shear instability (ASI) caused by the high strain rate $(10^8 - 10^9 \text{ s}^{-1})$ and localised plastic deformation at the interface. Because adiabatic heating predominates over work

hardening at the ASI region, thermal softening causes the metal to behave like a viscous substance, resulting in the development of an outward jet and extrusion. Because of the severe plastic deformation after impact, the thin oxide surface on the particle or substrate is shattered, breaking the intimate contact between particles and substrate. A dimple-like ductile feature at the fracture surface is generally seen as evidence of metallurgical bonding (Xie et al., 2019).

Peening effect has been introduced to cold spray coating as an effective way to improve material qualities by intensifying plastic deformation and improving densification. In addition to the peening effect produced by mixing big particles in feedstock powders, unbonded particles can create peening effects on already formed coatings. The bonding properties are affected by a stronger peening effect at higher pushing gas pressure (Xie et al., 2019).

However, metallurgical bonding was found at the entire coating deposition due to the presence of diffusion following heat treatment. Thus, the enhanced peening effect of consecutive particles with sequential impact energy could be the deciding element in the establishment of metallurgical bonding. The increased peening impact greatly improved coating quality through improved metallurgical bonding, as evidenced by increased adhesion strength and decreased porosity (Xie et al., 2019).

2.4 Mechanical Testing

The research report will include an evaluation of five standard tests, which are Scanning Electron Microscope analysis (SEM), Energy Dispersive X-Ray Spectroscopy (EDX), Tensile strength testing, Microhardness testing, and Transmission Electron Microscope

(TEM). All the tests are crucial to material testing and adhere to the American Society for Testing and Materials (ASTM) standard.

2.4.1 Introduction

Material testing is the final phase in the manufacturing process in which the materials' mechanical properties are determined. All testing will be done in accordance with the American Society for Testing and Materials (ASTM). The standard is essential for ensuring that the outcomes are precise and true. Furthermore, the materials should be compatible with the applications to avoid defects. As a result, materials testing is essential. The significance of material testing is to provide routine information on a product's quality, to produce new or significantly improved information about materials, and to collect reliable measurements on fundamental properties of materials.

Material testing is divided into two types: destructive testing and non-destructive testing. Destructive testing is performed until the specimen fails. When compared to non-destructive testing, these tests are easier to comprehend, considerably easier to perform, and offer more information. Non-destructive testing refers to testing that does not destroy the specimen. This research project focuses on all of the tests to determine the structural and mechanical properties of Al 7075-T6 coated with Al 6061 powder particles.

2.4.2 Types of Testing

In this project of study, five standard tests that will be carried out are:

2.4.2.1 Scanning Electron Microscope Analysis (SEM)

The human eye is incapable of distinguishing things smaller than 200 m. (0.2 mm). In other words, a human eye has a resolution of 200 m, but a light microscope can often magnify pictures up to 1000 times to see details as small as 0.2 m. The resolution limit is defined as the lowest recognisable distance between two objects, also known as the least resolvable distance. For example, two objects separated by less than 200 m will seem as one to the human eye since the latter is unable to discern features with dimensions less than 200 m. As a result, 200 m might be regarded the human eye's resolution limit.(Ul-Hamid, n.d.) The strengths of SEM are:

- 1. A wide variety of specimens can be examined.
- 2. Relatively easy and quick sample preparation.
- 3. Ease of use due to user-friendly and automated equipment.
- 4. Rapid imaging, quick results, time-efficient analysis, and fast turnaround time.
- 5. Relatively straightforward image interpretation.
- Large depth of field (ability to focus large depths of samples at one time and produce 3-D like images)
- 7. Non-destructive (some beam damage may result).

The materials can be conductive or non-conductive, solid or powder, and evaluated as received or processed (sectioned, ground, polished, etched, coated, etc.). The SEM can study materials in both dry and wet states, as well as extract microchemical information from minute structural features (Ul-Hamid, n.d.).

The SEM is one of the most widely used instruments in academia, research, and industry due to aspects such as high resolution, vast depth of field, compositional information, time-efficient analysis, and relative ease of use and image interpretation in both materials and biological sciences. Although the SEM may yield rich morphological and compositional data for a wide range of materials, it is frequently essential to use several other analytical instruments to complete materials characterization (Ul-Hamid, n.d.).

2.4.2.2 Energy Dispersive X-Ray Spectroscopy (EDX)

EDX spectroscopy is used to determine the elemental makeup of a sample using a scanning electron microscope. EDX can identify elements with atomic numbers more than boron, and these elements can be identified at concentrations of at least 0.1 percent. Material evaluation and identification, contaminant identification, spot detection analysis of regions up to 10 cm in diameter, quality control screening, and other applications are all possible with EDX (Abd Mutalib et al., 2017).

In a conventional SEM, when the samples collide with the electron beam, they interact with the beam and emit distinctive X-rays. Because no other elements have the same X-ray emission spectrum, they may be distinguished and tested for concentration in the sample. The X-ray is produced by the interaction of the primary electron beam with the nucleus of the sample atom. A primary electron beam will excite an electron in an atom's nucleus, ejecting it and forming an electron hole. The extra X-ray is released when an electron from the atom's outer shell (higher energy) replaces the missing expelled electron. The emitted X-ray comprises of X-ray continuum (caused by electron slowdown) and distinctive X-ray (generated because of higher shell electron filling the electron hole in the nucleus shell) (Abd Mutalib et al., 2017) as shown in Figure 2.5:



Figure 2.5: Schematic representation of the types of X-ray spectrum emitted upon bombardment of fast electron (Abd Mutalib et al., 2017)

The X-ray continuum is not essential for identifying items in the sample that must be identified to differentiate them. Factors such as probe current, accelerating voltage provided, and sample atomic number all contribute to the intensity of the X-ray continuum. The characteristic X-ray, on the other hand, will be recorded by the energy dispersive spectrometer for elemental composition analysis in the material.

2.4.2.3 Tensile strength testing

Tensile testing is a destructive test that provides information about the metallic material's tensile strength, yield strength, and ductility. The strength of a material is a widely utilised and recognised material attribute. The tensile test is used to determine the strength or mechanical behaviour of a material. The basic principle behind a tensile test is to clamp a sample of a material between two fixtures called "grips." The length and cross-sectional area of the material are known. We then start applying weight to the material that is grabbed at

one end while the other is fixed. We continue to increase the weight (also known as the load or force) while measuring the change in length of the sample (De et al., 2017).

This is one of the most often used mechanical testing methods. It is used to determine how strong a material is as well as how far it can be stretched before breaking. The yield strength, ultimate tensile strength, ductility, strain hardening properties, Young's modulus, and Poisson's ratio are all determined using this test method. Tensile testing shows results using graphs of stress versus strain (De et al., 2017).

The region on the stress-strain curve where deformation can be reversed by releasing stress which is known as elastic deformation. It is also the location where stress is most strongly correlated to strain. On a stress-strain curve, it is identified as the first linear part of the graph. The elastic modulus, commonly known as Young's modulus, is the constant that links the proportion of stress (σ) to strain (ε) during elastic deformation. It is the initial slope of the linear portion of a stress-strain curve. The equation $\sigma = E \cdot \varepsilon$ represents this relationship. Hooke's Law, which was designed to represent the behaviour of springs, is the name given to this relation.

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Strain above the material's yield point generates strain hardening, which permanently deforms the material and modifies its mechanical characteristics, resulting in plastic deformation. The yield point is the boundary between the elastic and plastic deformation regions. It is distinguished by a steep bend in the stress-strain curve at the elastic region's end. Materials with no clear end to the elastic area do not have a yield point. In those circumstances, the offset approach is used to approximate yield. It can, however, only be evaluated experimentally by loading and unloading and gradually increasing stresses to discover where plastic deformation begins.

2.4.2.4 Microhardness Testing

Vickers hardness testing is also known as microhardness testing and is commonly used for small parts, thin sections, or case depth work. The area of the indentation is measured rather than the depth. An optical measurement system embodies the Vickers method. ASTM-E-384 specifies a range of light loads that are measured and converted into hardness values using a diamond indenter (Zhao et al., 2021).

Microhardness of materials can show the material's local resistance to external invasion (Zhao et al., 2021). The following are examples of common measurement procedures:

1) press the indenter into the tested material with a defined load.

2) measure the local plastic deformation dimension.

3) calculate the hardness.

Based on the abovementioned, the pioneering hardness is measured manually using optical microscopes to measure the diagonal length of the indentation region. To improve testing automation, a semi-automatic method based on image processing and analysis algorithms is provided for detecting the indentation dimension of Brinell and Vickers indentation images. Microhardness is an efficient analyser of manufacturing-affected layers, when the material properties are altered during the manufacturing process. A variety of investigations have also been reported in this regard (Zhao et al., 2021).

A square base pyramid shaped diamond indenter with a 136° angle is gently depressed on the surface of the sample in the Vickers scale. Held for a set amount of time (10–15 seconds). As a result, the hardness is estimated based on the size of the plastic

indentation left on the specimen surface after the indenter is retracted and the load is removed.



Figure 2.6: Principle of Vickers Hardness Test (Zhao et al., 2021).

Despite the availability of the methods, the following bottlenecks can still be identified:

1) it is difficult to obtain accurate indentation depth information based on local micrographs because commercial hardness testers could not stitch the micrographs.

2) the accuracy of indentation dimensions measured by hardness testers cannot be guaranteed, especially when the captured micrographs have noise, texture, or material defects (such as scratches, pits, and cracks during the sample preparation process prior to the observation process), whereas measurement thru human raw eyes are incapable of providing satisfying strength and efficiency. As a result,

understanding the microhardness profile would be going to be difficult (Zhao et al., 2021).

The Vickers hardness is donated by the symbol HV followed by numbers representing the applied test force and the duration time of the test force. Example:



The value of diagonal indent length is measured and converted into hardness number (HV) and in International Standard of units (MPa) as shown in Eq.2.1 and Eq.2.2 respectively.

Hardness (HV) =
$$\frac{1.845 P}{\left(\frac{d1+d2}{2}\right)^2}$$
(Eq.2.1)
Hardness (MPa) = HV x 9.87(Eq.2.2)

-

Where P, is applied load (kgf), d₁ and d₂ are the diagonal indent length in millimetre (mm).

Vickers microhardness testing is useful for a variety of applications, including testing of very thin materials such as foils or measuring the surface of a small part, small parts, or small area, measuring individual microstructures, and evaluating the detail of case hardening by segmenting a part and making a series of indentation to explain a profile of the change hardness. As much as test samples are prepared cautiously, a wide range of materials can be examined. A microhardness test normally requires sample preparation to deliver a small enough specimen to fit inside the tester. Furthermore, sample preparation is required to smooth the specimen's surface, allowing for a regular indentation shape and accurate measurement. Also need to ensure the samples can be held perpendicular to the indenter.

2.4.2.5 Transmission electron microscope, TEM for single particle Al 6061 on 7075-T6.

Transmission electron microscopy (TEM) is an effective method for studying the microstructure of biological tissues, cells, and organisms, including worms. The TEM examines specimens by sending an electron beam through them, revealing comprehensive information about the internal structure of the objects. The most difficult challenge in analysing biological material with an electron microscope is ensuring that the specimens' ultrastructure remains as intact as possible. Furthermore, due to electrons' limited penetrating ability, samples must be thin or split into tiny portions (50 - 100 nm) to allow electrons to pass through. Thin pieces must be dyed with heavy metal salts.

The TEM works in the same way as an optical microscope does (Figure 2.7). It substitutes electrons for light and electron magnetic lenses for glass lenses. The condenser lens focuses electrons accelerated from the electron source into a narrow, homogeneous beam. To exclude electrons travelling at great angles to the optical axis, a tiny condenser aperture is utilised. The beam then passes through the sample at or near normal incidence. Parts of the beam are diffracted depending on the sample's thickness and electron transparency. The objective lens then focuses the transmitted rays to generate a picture. The picture is then magnified by the intermediate and projector lenses and projected onto a phosphor screen or electron camera. An objective aperture can be used to improve picture contrast by allowing just a portion of the transmitted or diffracted beams, or both, to pass through (Zuo & Spence, n.d.).



Early TEMs used a single condenser lens to light a 1 mm sample area. This vast region of light heated the sample. The two condenser lens setup was used to alleviate this problem. This approach was used in the first mass-produced TEM, the Siemens "Elmiskop," giving it "small area radiation" capacity. At a magnification of 100,000, a beam of 1 micron could be created on the material for an image size of 10 cm (Zuo & Spence, n.d.).

2.5 Conclusion

This chapter provides an overview of the element that are narrowed down to the analysis of characterization and measurement of coating specimen in details. A review of the correlation between microstructure properties and adhesion bonding mechanism of cold sprayed 6061 Al on 7075-T6 Al substrate is provided in this chapter. All the information about parameters involve are exposed to the concept and theories.



CHAPTER 3

METHODOLOGY

This chapter describes research methodology of this study. The research methodology that is used describes the process required including the preparation of sample and equipment and techniques to analyze the data obtained. It is carried out based on the problem statement and objective structured. Cold spray process was synthesis and characterized by using following chemicals and instruments.





- 1. Preparation of Sample (send the samples to Singapore for Coating)
- 2. Instrument
 - I. High Pressure Cold Spray Machine.
- 3. Powder used
 - I. Valimet Al-6061
- 4. Testing Methods
 - I. Scanning Electron Microscope analysis (SEM)
 - II. Energy Dispersive X-Ray Spectroscopy (EDX)
 - III. Tensile strength testing
 - IV. Microhardness testing
 - V. Transmission Electron Microscopy (TEM)

3.1 Preparation of Sample

Figure 3.2 shows that, after getting the raw sample from supervisor which is the Al 7075-T6

(Cylindrical shaped) to be cut into pieces with the diameter of Ø25mm and with the thickness

of 10mm.



Figure 3.2: Cutting samples using 2+1 CNC turning machine.

Figure 3.3 shows the sample is being cut using the 2+1 CNC turning machine after inserting its diameter and thickness into the NC code of the machine system to be cut precisely according to the requirement given by supervisor.



Figure 3.4 shows the samples after being cut from using the CNC 2+1 turning machine. All the sample were checked for the dimensions and its thickness as per for the requirement.



Figure 3.4: Samples after being cut using CNC 2+1 turning machine.

3.2 Material

As feedstock, we applied 6061 Al powder (Valimet, USA) with an even grain size of 45 μ m as shown in Figure 3.5 below. The material composition of substrates used presented in Table 3.1. The specimens measuring Ø 25 x 10 mm were used to assess the coating adhesion strength.



Figure 3.5: SEM images of the Al-6061 feedstock powder at (a) 50x and (b) 150x.

Table 3.1: Material chemical composition [wt.%]									
Element	Al	Cr	Cu	Fe	Mg	Mn	Si	Ti	Zn
7075-T6 Al	91.4	0.38	2.0	0.5	2.9	0.3	0.4	0.2	6.1
اويومرسيني بيصيصل مليسيا ملاك									

3.3 Deposite Pure Al and Al 6061

High pressure cold spray (HPCS) machine is used as coating technique in this study for preparing samples. In this process, the accelerating Nitrogen gas at high pressure with the range of 500 psi/3.44738 MPa. It is not to increase the particles temperature but it is needed to optimize its aerodynamics properties and forced thru a nozzle. At the nozzle, enthalpy conversion to kinetic energy is produced by gas expansion. It accelerates the flow of the gas to supersonic regime while reducing the temperature. The powder type Valimet Al-6061 is used for this particular process especially for this research. Where, the Gun temperature used is at 500°C and the powder heater temperature used was 280°C. The powder feed rate for 3-

cylinder samples and 2 square to be full coated was 5% whereas, for the single plat particle collection the powder feed rate used was 2%. Figure 3.6 showed a schematic diagram of coating experiment.

All the parameters are summarized as in the table 3.2 below:

Spray Parameters					
Cs System	Centreline EPX				
Test Substrate	Provided by UTeM (Al 7075-T6)				
Powder Type	Valimet Al-6061				
Gas Type	Nitrogen				
Gun Pressure	3.44738 MPa				
Gun Temperature		5	00°C		
Powder Heater Temperature	280°C				
Powder Feed Rate	3 cy	lindrical sample & 2	S	Single splat particle	
WALAYS/A	sq	uares (full coating)		collection	
	5%			2%	
Nozzle Transverse Speed	Se .	50 m/s		2000 m/s	
Stand-off Distance	5	2	0mm		
Spray distance 20mm June ERSTIN Nozzle		يىتى تيكنيد KAL MALAYSIA		اود KA	
Substrate					

Table 3.2: Shows coating experiment conditions
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Figure 3.6: Schematic diagram of coating experiment





3.4.1 Scanning Electron Microscope analysis (SEM)

Scanning electron microscopy (SEM), often known as SEM analysis or SEM method, is widely utilised in a variety of fields across the world. It is a useful approach for analysing organic and inorganic materials on a nanoscale to micrometre (m) scale. SEMs operate at high magnifications of up to 300,000x and even 1000000 (in some current versions) to provide extremely exact pictures of a wide range of materials. SEM scans the specimen's surface using an electron beam. The reflected (back scattered) electron beam is collected and shown on a cathode ray at the same scanning rate. The specimen's most essential criteria are that it be electrically conductive. Before entering the vacuum chamber, all samples must be mounted and cleaned. Figure 3.7 describes the mounting of samples via mounting metallography press equipment.



Figure 3.7: Mounting Metallography Press

Scanning electron microscope (SEM: JEOL6010PLUS) was used to observe crosssectional microstructures of the coating. The observation sample of the 6061Al coating was prepared by embedding the 25mm x 10mmx 5mm sample into a harden-able resin. The hardened sample in hardened resin was grinded with silica papers to a #3000 grit size and was finally polished with 1µm and 0.3µm alumina suspension.

3.4.2 Energy Dispersive X-Ray Spectroscopy (EDX)

Energy dispersive X-ray spectroscopy (EDX) is an analytical technique used to characterise materials analytically or chemically. EDX systems are typically used in conjunction with an electron microscopy instrument such as transmission electron microscopy (TEM) or scanning electron microscopy (SEM). The emission of a specimen's distinctive Xrays is the core for EDX. A beam of high-energy charged particles (electrons or protons) is focused on the sample under investigation. When an electron from a higher binding energy electron level falls into the core hole, it emits an X-ray with the energy of the difference between the electron level binding energies. The elemental composition of the analysed sample is associated with the peaks in the EDX spectrum. Figure 3.8 shows the sample preparation using an EDX machine. Figure 3.9 showed an EDX will be conducted on fracture coating surface.



Figure 3.8: EDX machine (Ikumapayi & Akinlabi, 2018)



Figure 3.9: EDX on fracture coating substrate surface

3.4.3 Tensile Strength Testing

Tensile testing is a method of determining the mechanical strength of a material by applying tensional force to a specimen until fracturing occurs. It is a fundamental materials science study that seeks to comprehend material behaviour under load, involves stress and strain measurements, which necessitates knowledge of the initial cross-sectional area of the sample being evaluated. N/mm2, MPa, PSI, and % are common units of measurement. As shown in Figure 3.10, the test results are typically shown as a graph of stress vs strain. The shape of the sample varies during a tensile test when load is applied. Understanding the change in the sample's dimension at various or specified forces helps determine the material's performance and suitability for a given application or product.



Figure 3.10: Typical stress strain curve for tensile test result (Bałon et al., 2018)

The load is placed along only one axis in this test, and the rate of loading is constant. This test is performed on a universal mechanical testing machine that is either screw-driven or hydraulically operated. The machine may be computer operated in some circumstances. The major data produced is load vs. elongation, which will be transformed into stress vs. strain data. Tensile testing can be done at the most basic level with a handheld force gauge, which measures the pull force applied to a sample, product, or component to determine the maximum force. Advanced tensile testing systems with advanced testing software and accompanying devices, such as extensometers. These testing systems can pull the sample under test at a very precise velocity to a very precise target. Large data sampling helps produce high resolution data for both force and distance or stress and strain, so that very accurate measurements can be taken, analysed, and reported (Bałon et al., 2018).

Load is measured using a load cell in current tensile testers; however, in older or simpler testing machines, the load may be measured using a purely mechanical or hydraulic mechanism. Strain can be determined directly from the specimen or via the displacement of the crosshead. Mechanical dial indications, electrically resistive strain gauges attached to the specimen, or extensometers that use an optical device, a strain gauge, or an inductive or capacitive transducer are common strain measuring devices. The advantage of strain transducers is that they only measure displacement in the gauge length of the specimen. This reduces inaccuracy caused by specimen deformation, slack in the load train, and the stiffness of the testing machine. Figure 3.11 and 3.12 shows the typical Universal Tensile Machine at Material Testing Lab, FTK UTeM.



Figure 3.11: Instron 5960 Series Dual Column 50kN Universal Testing Machine



Figure 3.12: Autograph AGS-X Series - Shimadzu 100kN Universal Testing Machine

International testing standard for tensile test includes from ASTM, ISO, DIN, or other organizations. ASTM standards for common tensile tests are ASTM E8 (metals), ASTM D638 (plastics), ASTM D2343 (fibres), ASTM D897 (adhesives), ASTM D987 (paper), ASTM D3039 (laminate) and ASTM D412 (rubber).

In accordance with ASTM C633, specimens of dimensions of Ø 25 mm x 10 mm as shown by Figure 3.13, assessed the adhesion strength of the coating given as the fracture load value as measured by the universal testing machine (Auto graph AGS-J 10kN Shimadzu) and subsequently divided by the cylindrical coating area. The FM 1000 adhesive glue film was used to bond the pin and coating as shown by Figure 3.14. The strength test was performed after the glue was cured in oven 170 ± 6 °C for 90 min \pm 10 min as per AMS2750. We measured the adhesion strength over an average of three specimens, as minimum requirement of ASTM C633 is 3 samples.



Figure 3.13: Specimen with 6061 Al coating



Figure 3.14: Sample preparation for tensile strength testing

3.4.4 Microhardness Testing

In microhardness testing, a diamond indenter is used to make an indentation on the specimen by applying a load P. (Figure 3.15). A calibrated optical microscope is used to determine the size d of the resulting indentation, and the hardness is calculated as the mean stress applied beneath the indenter. Hardness measurements with a microscope attachment, which includes an indenter and means for applying modest stresses, date back more than 50 years. Microhardness testing was originally used for small components (watch gears, thin wire, foils), but it has since been extended to research studies of individual phases, orientation effects in single crystals, diffusion gradients, ageing phenomena, and other phenomena in metallic and ceramic materials. Testing at temperatures of up to 1000 degrees Celsius is now conceivable. In Europe, the pyramidal Vickers-type (interfacial angle 136 degree) indenter, which produces a square impression, is generally favoured (Wang & Wang, 2019).



Figure 3.15: Application of microhardness testing (Wang & Wang, 2019)

The sample is done using micro-Vickers hardness test machine in figure 3.16.



Figure 3.16: Micro-Vickers Hardness Test Testing Machine

3.4.5 Transmission Electron Microscope, TEM

The construction of these early TEMs (Figure 3.17), which used a series of horizontal lenses in a column, differed from that of current TEMs, which use a series of vertically aligned lenses in a column. Due to the poor resolution, the TEMs with lenses in horizontal series were quickly discarded. Alternatively, vertically aligned lenses in columns may be able to maintain good alignment for a longer period of time due to increased gravity pressures parallel to the optic axis resulting in less mechanical creep. In 1938, the Siemens business in Germany developed the first commercial TEM, which had a resolution of 10 nm at an accelerating voltage of 80 kV (Tang & Yang, 2017).



Figure 3.17: Transmission Electron Microscope, TEM (Tang & Yang, 2017).

The technique of electron focusing is based on the wavelike nature of electrons as negatively charged particles that are deflected by magnetic or electric fields. This property of electrons has also been widely employed in modern electrical devices like as computer screens, TV display tubes, and cathode-ray tubes. During this method, electrons with a narrow energy range may pass through, resulting in a well-defined energy electron beam. The transmitted electrons are then delivered to the specimen in the TEM column, which is put onto the sample holder (or TEM grid, which consists of a metal frame and carbon-based sheet) equipped with a mechanical arm for regulating the position and retaining the specimen (Tang & Yang, 2017).

For electrons to flow through, the thickness of a TEM specimen should be less than 100 nm. Many characteristics of the specimens, such as density or composition, might influence electron beam transmission. For example, more electrons would move through porous metal than through considerably denser material. The paralleled electron beams might also gather the crystal structure information of a specimen by using a condenser lens in a TEM. After passing through the specimen, the transmitted electrons are refocused and enlarged by an electromagnetic lens system comprised of two lenses, and the electron picture is converted on a phosphor screen information to a viewable form. Electron emission directions in the entire imaging system might be modified using electromagnetic lenses such as condenser, objective, and projective lenses. It should be observed that when electrons strike the phosphorescent plate during the TEM imaging process, it lights. A TEM's complete imaging mechanism is likewise akin to photography (Tang & Yang, 2017).

Because of a TEM's excellent resolution, even the fine structure of crystals may be examined by employing electrons with short wavelengths (Figure 3.18). Furthermore, to improve the mean free path of electrons in a TEM column, the column should maintain an exceptionally high vacuum since electrons cannot flow in atmosphere (Tang & Yang, 2017).



Figure 3.18: Types of signals produced by the hitting of a specimen with a high-energy electron beam (Tang & Yang, 2017).

3.5 Conclusion

This chapter describes the research methodology for this study. This chapter describes the processes needed for this research project, including preparation and the equipment that will be used to help analyse the data obtained based on the problem statements and objectives. Except for the coating parameters, which are the actual data for this research project, all of the application details and some numerical data for all tests are based on previous research obtained from books and journals.



CHAPTER 4

RESULT

4.1 Scanning Electron Microscope analysis (SEM) and EDX analysis

Figures 4.1 and 4.2 showed the surface morphology of the cold sprayed 6061 Al powder on 7075-T6 Al substrate. The images show that dense coatings with a thickness around 300 μ m could be obtained on 7075-T6 substrate, meaning that a critical velocity was achieved for Al 6061 powder. The cold spraying can be divided into two phases, adhesion, and cohesion bonding. The first stage is the deposition of an interlayer between the substrate and the particle. This can affect the deposition behaviour of the first layer, although the deposition of the coating is mainly affected by cohesion between the particles.



Figure 4.1 SEM image



Figure 4.2 SEM of Al 7075-T6 coated with Al 6061 powder (a) 30x, (b) 50x, (c) 100x, (d) 150x.

SEM-EDX show that the elements present are Aluminium (Al), Magnesium (Mg),

Carbon (c) for cold sprayed 6061Al coating as shown by figure 4.3 and table 4.1.



Figure 4.3 EDX elemental mapping of cold sprayed 6061 coating: (a) SEM (b) map sum spectrum.

Element	Weight %	Atomic %		
С	14.75	27.96		
Mg	0.89	0.83		
Al	84.36	71.21		

 Table 4.1: Composition of cold sprayed 6061 Al coating

SEM-EDX show that the elements present are Aluminium (Al), Magnesium (Mg),

Carbon (c) and Zinc (Zn) for 7075-T6 substrate as shown by figure 4.4 and table 4.2.



Figure 4.4: EDX elemental mapping of 7075-T6 substrate: (a) SEM (b) map sum spectrum.

Element	Weight %	Atomic %
С	17.31	32.75
Mσ	2.63	2.46
A1	74.69	62.02
	74.09	02.92
Zn	5.37	1.87

Table 4 2.	Composition	of 7075-T6	substrate
I AUIC 4.4.	Composition	01/0/3-10	วนมวน ลเ ต

4.2 Strength of Adhesion and EDX analysis



Figure 4.5 showed samples before and after cold spray coatings.

Figure 4.5: Samples (a) before cold spray (b) after cold spray

The adhesion strength of the cold sprayed 6061-Al on 7075-T6 Al is shown in Table

4.3 below. The mean value of coating adhesion strength is 10 MPa.

Cold sprayed 6061 Al on 7075-T6 substrate	Bond strength (MPa)
Sample 1	10
Sample 2	10
Sample 3	10

Figure 4.6 shows the fracture coating of 6061-Al on 7075-T6 substrate after adhesion strength testing. The interface fracture occurred between the coating and substrate for 7075-T6 substrates, as shown in Figure 4.6.



Figure 4.6: Fracture surface substrate and 6061 Al coating after tensile strength testing

Figure 4.7 shows SEM images of fracture coatings of 6061 Al on 7075-T6 substrate. The fracture coatings images showed 6061-Al particle uniformly dispersed within the coatings. EDX elemental mappings and SEM images are shown below in (Figure 4.8) with elements present are aluminium (Al) and magnesium (Mg).



Figure 4.7: SEM images of fracture coatings 6061 Al on 7075-T6 substrate



4.3 Microhardness Test

Table 4.5 shows the microhardness measurement test results on the coated substrate. The results shows that the 6061 Al powder coating has an appropriate level of adhesion strength based on inspection under the microscope at 5x magnification (HF2 and HF3) from figure 4.9.
	/0/5-10 AI	
Material	6061 Al coating	7075-T6 Al
Hardness (HRB)	24.27	89.4

Table 4.5: Microhardness test measurements obtained on coated substrate and7075-T6 Al



Figure 4.9: Microscopic results (5x magnification) on 6061 Al coating

Table 4.5 also shows the results of the 7075-T6 Al substrate which is the average hardness value of 7075-T6 Al. The test shows that it is 89.4 HRB for the 7075-T6 substrate.



Figure 4.10: Microscopic results (5x magnification) on Al 7075-T6

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Microhardness measurements throughout the deposits and substrates revealed that the Al7075 deposits were twice as hard as pure Al (see Fig. 4.11). According to (Rokni a et al., 2017) the substrate surface hardness, which is coated with pure Al, on the other hand, showed a decreased microhardness compared to the previous series due to increased deformation in the coating/substrate region. (Rokni a et al., 2017) also claimed that the four unique textures depict the indentation shapes that enable strong interfacial interactions between the coating and the substrate (HF1, HF2, HF3 and HF4) in (Figure 4.12). However, the delimitation (HF5 and HF6) near to the indentation demonstrates poor interfacial adhesion. (HF is an abbreviation for adhesion strength in German). Furthermore, each layer of the graded coating may be

investigated in terms of its unique characteristics, in principle, the coating acts as a diffusion barrier.



Figure 4.11: * specimen coated by Al7075 • specimen coated by pure Al (Rokni a et al., 2017)



Figure 4.12: Classification of the acceptable level of interfacial adhesion of thin film based on typical indentation results (Rokni a et al., 2017).

4.4 Transmission Electron Microscopy (TEM)

Figure 4.13 depicts the TEM characterizations of (a) and (b) in the 1-0-0 direction. The crystal dislocations may be seen at magnifications of 6000 and 10,000. Dislocation lines are those having a darker tone and a crack form. The order of dislocation density from high to low, according to Fig. 4.13, is (c), (a), and (b), specifically from the coated region with 6061 Al and 7075-T6 substrate. In general, when the superposition of initial residual stress and cyclic dynamic stress exceeds the material's yield limit, microplastic deformation and dislocation occur in the crystal, accompanied by residual stress alleviation. While heat reduces dislocation density. The dislocation changes detected by TEM in the current work are compatible with the results below.



Figure 4.13: TEM images of the coatings (a) 2µm, (b) 2µm and (c) 2µm 6061 Al on 7075-T6 substrate

Figure 12 shows TEM pictures of the contact between a single 6061 Al powder particle and a 400°C annealed 7075-T6 substrate. After the high-velocity cold-sprayed 6061 Al struck the substrates, these results show the presence of a residual interlayer oxide with a thickness of about 2 μ m for 6061 Al and 7075-T6 Al. From the investigation of the effect of cold-sprayed 6061 Al bond strength on 7075-T6 substrate characteristics, The study's finding is summarised below:

1. The principal bonding process is metallurgical bonding of cold-sprayed 6061 Al particles and 7075-T6 substrate surfaces. Metallic bonding occurs because of a chemical reaction between deposited particles or the interface between coating and substrate at the oxide-free interface, as well as metal-to-metal contact. The presence of intermetallic, amorphous phases, or dimple-like structures at the fracture surface of the coating or a single splat is assumed to indicate that metallurgical bonding provides relatively high adhesion strength in cold-sprayed coating.

4.5 Conclusion

This chapter describes the result for this study. In this study, all these above testing are necessary for this project. All these data provided in this chapter are based on the tests that have been carried out. This results of all the tests shows the actual data of this project.

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CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

High Pressure Cold Spray (HPCS) process was successfully used as a corrosion protection treatment method for pitting corrosion that present on Al 7075-T6 surface. Furthermore, HPCS is the best technique to be applied which can eliminate fatigue phenomena because of no heat applied during this process. In this process, the accelerating nitrogen gas at high pressure range which is 500 psi is preheated up to certain temperature range 280 °C. 45 µm Al 6061 powder feedstock particles with efficiency up to 38% and porosity is less than 0.5% mix with the propellant gas in the prechamber zone are the axially fed into the gas stream. The accelerated Al 6061 powder particles with standoff distance 20mm, feed rate 12-15 grams per minute, gun traverse speed at 40 mm per second-, and 36-38-mm coating per pass impact the al 7075-t6 substrate with enough kinetic energy to induce mechanical bonding by 4mm thickness. Based on all the parameters used, the Al 6061 powder particles were successfully deposited onto the Al 7075-T6 substrate surface. The effectiveness of HPCS process has been proved by undergoing a total number of 5 standard tests.

Scanning Electron Microscope Analysis (SEM) is carried out to study and analyse the organic and inorganic materials on a nanoscale to micrometre (m) scale of the coating onto the substrate. SEMs operate at high magnifications of up to 300,000x and even 1000000 (in some current versions) to provide extremely exact pictures of a wide range of materials. The low density and irregular particle morphology of the particles may have promoted the formation of pores as the gas was entrapped in the coating during the deposition process as the level of porosity was lower when elliptical particles were employed. The SEM is also one of the most

widely used instruments all industry due to aspects such as high resolution, vast depth of field, compositional information, time-efficient analysis, and relative ease of use and image interpretation in both materials and biological sciences. Although the SEM may yield rich morphological and compositional data for a wide range of materials, it is frequently essential to use several other analytical instruments to complete materials characterization. The mechanical and metallurgical bonding are currently regarded as the primary bonding mechanisms in cold spray. Metallic bonding is generated as a chemical reaction between deposited particles or the interface between coating and substrate at the oxide-free interface and metal-to-metal contact. Metallurgical bonding is thought to offer relatively high adhesion strength in cold-sprayed coating, as evidenced by the presence of intermetallic, amorphous phases, or dimple-like features at the fracture surface of the coating or a single splat. The metallurgical bonding was found at the entire coating deposition due to the presence of diffusion following heat treatment. Thus, the enhanced peening effect of consecutive particles with sequential impact energy could be the deciding element in the establishment of metallurgical bonding. The increased peening impact greatly improved coating quality through improved metallurgical bonding, as evidenced by increased adhesion strength and decreased porosity.

Herein, we investigated the bonding mechanism of cold sprayed 6061-Al powder on 7075-T6 Al substrate. The summary of the study's finding are:

- 1. When the high-velocity cold sprayed 6061-Al particle impacted the soft metals substrate surface like 7075-T6 Al, plastic deformation of the particle and substrate are present, which promote metallurgical bonding from occurring in between the 6061 Al coating and 7075-T6 substrate.
- The primary bonding mechanism of 6061-Al particle and soft metal substrate 7075-T6
 Al is metallurgical bonding which required free oxide surface

5.2 **Recommendation for the Future Research**

alunda,

The current work exposed several areas that requires further evaluations. The suggestions for further research works include:

- 1. Evaluate the effectiveness of HPCS process by undergo salt spray test for testing inorganic and organic coating that can cause failure and pin-on-disc test for analyse wear properties.
- 2. Analysing the appropriate effective parameter and adjusting the shape of viscosity during HPCS on coated parts. Therefore, it is possible to generate the coated substrate with fewer defects and good interfacial adhesion.
- 3. Pre heating the 7075-T6 sample to study the influence of annealed toward bonding mechanism. The annealed process, releases the stress on substrate surface which will result in the increase of ductility. This way it is good for its bonding mechanism.

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APPENDICES

APPENDIX A: GANTT CHART PSM1

Project Gantt Chart, ALAYSIA									
et la		14		_	Period Highlight:	1 Plan Duration	Actual Start 🖉 % Complete	Actual (beyond plan)	% Complete (beyond plan
ACTIVITY	PLAN START	PLAN DURATION	ACTUAL START	ACTUAL DURATION	PERCENT COMPLETE	PERIODS	9 10 11 12 13 14 15 16 17 18	19 20 21 22 23 24 25 26	27 28 29 30 31 32 33 34 35
PSM briefing by Prof. Madya Ts. Dr. Lau Kok Tee	1	1	1	1	5%				
PSM meeting with supervisor, Madam Noor Irinah Binti Omar	2	1	2	1	6%				
Reading article/journal which suggested by PSM supervisor, Madam Noor Irinah Binti Omar	3	3	1	3	8%				
Suggestion for New title by PSM supervisor, Madam Noor Irinah Binti Omar.	Wn 4	1	4	1	10%				
Cutting specimen. With the help of En. Azimin from the lab. (at Makmal Pemesinan Termaju dan Studio CNC) Reading article/journal which is related to the project title.	5	3.0	5	3	25% 10%	عيد الم	اونبوس		
Started with the progress of the report by beginning with Chapter 1 – Introduction.	9	3	q	3	20%				
Started with the progress of the report by beginning with Chapter 2 – Literature review			EKN	IIKA	30%	LAYSIA	MELAKA		
Started with the progress of the report by beginning with Chapter - Methodology Started with the progress of the report by beginning with Chapter -	12	7	12	7	20%				
Preliminary Result	13	5	13	7	20%				
Presentation for PSM 1	14	1	14	1	0%				

APPENDIX B: GANTT CHART PSM 2

Project Gantt Chart						
					Period Highlight:	1 Plan Duration Actual Start 🖉 % Complete Mctual (beyond plan) % Complete (beyond plan)
ACTIVITY	PLAN START	DURATION	ACTUAL	ACTUAL DURATION	PERCENT COMPLETE	PERIODS
PSM briefing by Prof. Madya Ts. Dr. Lau Kok Tee	1	1	4 1	1	5%	
PSM meeting with supervisor, Dr Noor Irinah Binti Omar	2	1	2	1	6%	
Sample preparation for SEM observation testing in Material Science Lab.	3	2	3	2	8%	
Sample preparation for SEM observation testing in Material Science Lab.	4	1	4	1	10%	
Reading article on how to conduct adhesion bond strength, based on ASTM standard.	5	3	5	3	25%	
Reading article on how to conduct adhesion bond strength, based on ASTM standard.	6	4	6	4	10%	
SEM testing for the prepared sample at Material Testing Lab in FKM	7	0.3 A	7	3	20%	le up , mand , us
Sample preparation to carry out the tensile strength test.	8	7	8	6	30%	
Preparing sample no.2 for tensile testing to be carried out	9	7	9	5	20%	
SEM/EDX testing for the samples after tensile testing.	10	5	10	4	20%	
Meeting thru video call and face to face with supervisor regarding the report and discussed on how to write chapter 4 and 5.	11	2	11	2	10%	
Completing 1st draft of report including chapter 4 and 5	12	2	12	2	70%	
Presentation for PSM 2	14	1	14	1	0%	
			•	•		