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How to cite this thesis
SURFACE ENGINEERING: LASER METAL DEPOSITION OF TITANIUM ALLOY GRADE 5 AND TUNGSTEN

NDIVHUWO NDOU

Thesis submitted in partial fulfilment for the degree of

Doctor of Philosophy

In

Mechanical Engineering Science

At

The Faculty of Engineering and the Built Environment

University of Johannesburg

Promoter: Prof E.T. Akinlabi

Co-Promoter: Prof S. Pityana

AUGUST, 2017
DECLARATION

“I declare that this research is my own, unaided work. It is submitted in partial fulfilment of the requirements for the DPhil degree at the University of Johannesburg. It is my own original work; and it has not previously been submitted to any other institution of higher learning. I further declare that all sources cited or quoted are indicated and acknowledged by means of a comprehensive list of references”.

Signed by Ndivhuwo Ndou..........on ........the day of........year..........
DEDICATIONS

This thesis is dedicated to my wife, my daughters and the entire Ndou Clan
Ndou khulu dza muvunda ngoma, thavha dzovho ndi vhasidzana, dza ha nwatshisika,
dzinwa madi mulamboni wa tavha dza tamba nga mutavha wa tavha dza rwagwili nga
thoho dzo tinga shango.
ACKNOWLEDGEMENTS

I wish to acknowledge my supervisor for her advice, leadership and knowledge. The opportunity provided by Prof Akinlabi to conduct a research study in advanced manufacturing engineering has proved to be an extraordinary experience; and it will continue to be so for many years beyond the graduate school.

To my research colleagues, Dr Patrick Mubiayi and Dr Erinosho Folorunsho, I wish to thank you for helping me throughout my study work in many ways.

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Lastly, I wish to extend my most profound appreciation to my family, the Ndou clan and friends, for their love and motivation. To my wife, Mrs Elelwani Beauty Ndou, and my two daughters, Mukhethwa Ndou and Mutshidzi Ndou, you are my pillars; and you showed me constant encouragement and moral support. To Mpho Ndou, thanks for the motivation and support.
ABSTRACT

Titanium alloy Grade 5 (Ti6Al4V) has attracted the interest of the engineering community, because of its excellent physical and mechanical properties. Due to its low density, superior quality at high temperature and good corrosion resistance, Ti6Al4V alloy is used in the aerospace industry. The alloy has also been applied in many areas, such as sport, marine, the chemical industry, the automotive industry and the biomedical field – due to its excellent corrosion resistance in a corrosive environment or medium. Because of the poor wear-resistance properties exhibited by the alloy, five weight percent of tungsten (W) was agglomerated with it, this percentage weight addition of tungsten was optimised, in order to improve its surface properties in this research study. The tungsten is selected due to its superior strength, creep resistance, and structural stability at elevated temperatures.

Trial experiments were first conducted with the two powders, Ti6Al4V and W (Ti6Al4V+W). The parameters with good laser deposition process were selected for the preliminary studies. The relationships between the process parameters on the material characterizations were thoroughly investigated. Design Expert 9 software was used to validate the experimental results. In the design of the experiment, the Response Surface Methodology (RSM) was used to determine the required process parameters standard order and the leverage, as well as the response to the input factors. The model was validated to establish the variations between the predicted value and the actual value. The laser deposited Ti6Al4V+W specimens were characterized through the evolving microstructures, dry sliding wear, corrosion, microhardness and x-Ray diffraction.
Excellent coatings with good bonding between the interface of the deposit and the substrate were obtained. It was observed that the microhardness profiles and the wear properties of the primary alloy were improved with the inclusion of the tungsten powder when compared to the uncoated Ti6Al4V alloy. The corrosion tests conducted on the laser deposited Ti6Al4V+W specimens revealed good corrosion resistance that was kinetically active. The improvement in the surface properties of the Ti6Al4V alloy with the agglomeration of W has rendered the composite suitable for the production or repair of turbine blades that are exposed to high temperatures during operation; and to improve the efficiency and life span of components from this combination. Surface degradation of turbine blades during operation can be retarded by the application of a surface coating of Ti6Al4V+W. This study is novel and significant; and it can be applied in other various industrial applications, where Ti6Al4V is required for high temperature applications.

**Keywords:** Design of experiment, Elemental Analysis and X-ray diffraction, Laser metal deposition, Microhardness, Microstructure, Scanning Electron Microscope, Titanium alloy Grade 5, Tungsten powder and Wear properties.
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LIST OF PUBLICATIONS

The following publications have resulted from this thesis:


4. **Ndivhuwo Ndou, Esther T. Akinlabi, and Sisa Pityana** (2017), the microstructure, hardness and corrosion behaviour of Ti64l4V/W deposited by the laser metal deposition procedure, Book chapter accepted for publication. WCECS 2017
GLOSSARY OF TERMS

Additive Manufacturing: is a process by which digital 3D data is used to build up a component in layers by depositing material.

Argon: A rare gas used to protect the powder and the deposit from oxidations.

Beam: An accumulation of beams of light that might be parallel, focalized, or different.

Beam Diameter: The distance between diametrically opposed points in the cross section of a circular beam, where the intensity is reduced by a factor of 1/e (0.368) of the peak level (for safety standards).

Direct Laser Melting: surface-coating techniques that use laser heat to produce a coating from fine powders making use of powder beds.

Energy Dispersive X-ray Spectroscopy (EDS): A SEM component capable of doing chemical mapping analyses on coated and powder materials.

Laser: A laser is a device that produces an intense, collimated, monochromatic and coherent beam of light. The laser is an abbreviation for light amplification.

Laser Direct metal deposition: a powder injection deposition method that uses laser energy.

Laser Power: The energy per second emitted from a laser. Laser power is measured in watts (W) for continuous wave laser operation.

LENS (Laser Engineering Net Shaping): A process that injects metal powder into a melt pool created by a focused laser beam.

Nd: YAG Laser: Neodymium: Yttrium Aluminium Garnet. A synthetic crystal used as a laser medium to produce 1064nm wavelength light.

Scanning Speed: laser speed that interacts with material being processed.
Scanning Electron Microscope: A high-resolution research microscope necessary for surface and microstructural analysis

Titanium Alloy: Titanium offers a good combination of different properties, for example, excellent corrosion, high strength-to-weight ratio, low density, good biocompatibility and the retention of useful mechanical properties at elevated temperatures.

Tungsten: It is a low erosion rate, high wear resistance, good strength and good thermal conductivity, and the low thermal expansion that makes it attractive for use in different ranges of application.

Wear resistance: This is the ability of material to resist the gradual erosion of its surface material that is caused by abrasion and friction.

X-ray Diffraction: A tool that is able to detail the crystal structure of powdered materials.
# ABBREVIATIONS

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Full Form</th>
</tr>
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<tbody>
<tr>
<td>Al</td>
<td>Aluminium</td>
</tr>
<tr>
<td>AM</td>
<td>Additive manufacturing</td>
</tr>
<tr>
<td>ANOVA</td>
<td>Analysis of variance</td>
</tr>
<tr>
<td>ASTM</td>
<td>American Society of Testing</td>
</tr>
<tr>
<td>DF</td>
<td>Degree of Freedom</td>
</tr>
<tr>
<td>DOE</td>
<td>Design of experiments</td>
</tr>
<tr>
<td>EDX</td>
<td>Energy Dispersion X-Ray</td>
</tr>
<tr>
<td>GFR</td>
<td>Gas Flow Rate</td>
</tr>
<tr>
<td>HAZ</td>
<td>Heat affected zone</td>
</tr>
<tr>
<td>HV</td>
<td>Vickers Hardness</td>
</tr>
<tr>
<td>Hz</td>
<td>Hertz</td>
</tr>
<tr>
<td>LASER</td>
<td>Light Amplification by Simulated Emission of Radiation</td>
</tr>
<tr>
<td>LBAM</td>
<td>Laser Based Additive Manufacturing</td>
</tr>
<tr>
<td>LMD</td>
<td>Laser Metal Deposition</td>
</tr>
<tr>
<td>LP</td>
<td>Laser Power</td>
</tr>
<tr>
<td>MMC</td>
<td>Metal Matrix Composite</td>
</tr>
<tr>
<td>Nb</td>
<td>Niobium</td>
</tr>
<tr>
<td>Nd: YAG</td>
<td>Neodymium: Yttrium Aluminium Garnet</td>
</tr>
<tr>
<td>Ni</td>
<td>Nickel</td>
</tr>
<tr>
<td>OM</td>
<td>Optical Microscope</td>
</tr>
<tr>
<td>PFR</td>
<td>Powder Flow Rate</td>
</tr>
<tr>
<td>RSM</td>
<td>Response Surface Method</td>
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<td>SEM</td>
<td>Scanning Electron Microscope</td>
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xix
<table>
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<tr>
<th>Abbreviation</th>
<th>Description</th>
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<tr>
<td>SLM</td>
<td>Selective Laser Melting</td>
</tr>
<tr>
<td>SS</td>
<td>Scanning Speed</td>
</tr>
<tr>
<td>Ti</td>
<td>Titanium</td>
</tr>
<tr>
<td>Ti-6Al-4V</td>
<td>Grade 5, Titanium Alloy Containing 6% Al (Aluminium) and 4% (Vanadium)</td>
</tr>
<tr>
<td>TiMMC</td>
<td>Titanium Metal Matrix Composite</td>
</tr>
<tr>
<td>VHN</td>
<td>Vickers Hardness Number</td>
</tr>
<tr>
<td>W</td>
<td>Watt</td>
</tr>
<tr>
<td>XRD</td>
<td>X-Ray Diffraction</td>
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**NOMENCLATURE**

<table>
<thead>
<tr>
<th>Symbol</th>
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<tbody>
<tr>
<td>D</td>
<td>The laser beam diameter (mm)</td>
</tr>
<tr>
<td>E</td>
<td>Laser-energy density</td>
</tr>
<tr>
<td>e</td>
<td>Experimental error</td>
</tr>
<tr>
<td>L</td>
<td>Stroke length</td>
</tr>
<tr>
<td>P</td>
<td>Laser power (W)</td>
</tr>
<tr>
<td>PA</td>
<td>Partially absorbed laser power</td>
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<td>Partially reflected laser power</td>
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<td>r</td>
<td>Ball radius</td>
</tr>
<tr>
<td>R</td>
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</tr>
<tr>
<td>v</td>
<td>Scanning velocity</td>
</tr>
<tr>
<td>V</td>
<td>Wear volume in mm$^3$</td>
</tr>
<tr>
<td>w</td>
<td>Wear track width</td>
</tr>
<tr>
<td>x1</td>
<td>The first factor controlled in the experiments</td>
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</table>
CHAPTER ONE

1 INTRODUCTION

1.1 Background

Titanium and other light-weight materials are focal in the advancement of new aviation, car and organic applications (Sibum, 2003). Alternative manufacturing methods assume an exceptionally basic part in these improvements. Laser beam additive manufacturing (LBAM) is one such option. Near net-shaped titanium components can easily be manufactured at much-reduced cost and with very reduced, or no wastage, of material (Vilar, 1999, Koch and Mazumder, 1994, Liu et al., 2011).

There are various challenges associated with the repairing of the cracked turbine blade during and after repair. The challenges are as follows:

- Preclearing;
- Welding atmosphere;
- Preheating;
- Interpass temperature;
- Filler material composition;
- Welding sequence;
- Cooling rates;
- Heat input rate during welding;
- Post-weld heat treatment.
The designing procedures for a weld repair present many challenges, which are stated as follows:

- Cracking sensitivity of the base material;
- Degenerated structure of the base material in components exposed to service;
- Cracking sensitivity of the weld filler;
- Oxidation of the base material and weld filler material during welding;
- Distortion due to thermal contraction during and after welding;
- Hardness and brittleness of the weld and the Heat Affected Zone (HAZ) after welding;
- High internal stress level of welded components;
- Differences between the structure of the weld material and the base material

The successful application of the laser beam additive manufacturing process in repairing these blades is determined by choosing the proper processing parameters.

The three major factors that restrict the lifespan of a turbine blade are the temperature and the duration of service, the quality of the coating, in addition to the service parameters (Sozańska et al., 2012). It is necessary to protect the super alloys, from which engine parts are made, by diffusion coatings. This coating increases the lifespan, the reliability and the function of the individual engine parts; and eventually, it contributes to the flying safety (Becker et al., 2002). The change of flight mode, the starting or the stopping of aircraft engines creates thermal and mechanical strain, which consequently damages the engine segments (Tamarin, 2002).

The performance of a gas turbine engine relies on the highest temperature in the engine from the inlet to the high-pressure turbine. These parts require periodical substitution, in
order to maintain a strategic distance from the loss of motor power and inevitable breakdown. The blades experience the effects of several damages during operation, which limits the overall lifespan of the component, as well as other issues, such as creep, life cycle fatigue and hot corrosion, in this way, as far as supporting the necessities, fabricating troubles and costs, the blades are the most basic component of the gas turbines (Klimpel et al., 2011). The turbine blade’s damage occurs at the tip of the blade; due to the high-temperature wear that occurs between the tip and the stator line. Likewise, the damage can also be attributed to the thermal expansion of the blades under the high rotating speed and temperature (Klimpel et al., 2011).

The parts in the aviation manufacturing industries are considered hard to machine parts, due to their mechanical properties. Therefore, the repairing of parts has been a point of emphasis.

Titanium alloy offers high strength to corrosion, toughness, weight ratio and creep resistance; and this is mainly used in aviation, biomedical application and gas turbines (Guo et al., 2009, M'Saoubi et al., 2008, Wu, 2007). On the other hand, tungsten powder alloys demonstrate the ability of the turbine engine to retain most of their strength after being exposed to extremely high temperature. However, they are regarded as the main material of choice for the turbine of jet engines (Koc and Kodambaka, 2000, Choe et al., 2005, Zhou et al., 2012, Çelik, 2013).

Titanium is considered to be one of the important metals that are used as a basis for many industrial super-lives alloys. Tungsten powder alloys are regularly preferred for high-temperature applications in current advanced industries, because of their superior strength, creep resistance, and structural stability at elevated temperatures.
It requires an amount of time to repair, manufacture or produce complex parts with traditional manufacturing methods; and the overall cost to produce such a component becomes high.

The industry looks for different ways to reduce the production lead time and to produce components at a lower cost (Allen, 2006). Among the various methods adopted for repair, the best is Laser Additive Manufacturing (LAM) that uses computer-aided design (CAD) files to produce solid metallic components (Kobryn and Semiatin, 2001). During LAM operation, the nozzle is used to feed the powders into the melt pool created by the laser beam. The laser beam and the powder from its hopper source build the part layer by layer across the substrate. The LAM easily makes parts that are difficult to produce by traditional manufacturing methods; and it does so with high precision.

Different laser direct rapid manufacturing methods are frequently used for fabrication purposes. These methods include laser engineering, near net shaping, laser solid forming, and direct metal deposition (DMD), as well as Laser metal deposition (LMD). For the purpose of this study and the availability of equipment, the LMD process has been adopted for the study. This LMD is an additive manufacturing technology that offers the additional capability of being able to repair component parts (Mahamood et al., 2013) It plays an important part in the production of the critical part, most especially in the aerospace industry; however, the process of producing parts should be predictable and controllable, in order to achieve a refined surface. At the moment, most of the works in the literature are based on trial and error for optimising process parameters (Obielodan and Stucker, 2013).
This LMD process is especially attractive for titanium aviation components; since it can greatly reduce the buy-to-fly ratio and the lead time for the manufacturing of components (Kobryn and Semiatin, 2001).

In addition, the LMD process is a fairly new technology; and its physical process is still to be understood. However, limited works have been conducted on the relationship between the process parameters and the material defects, such as cracks, voids, inclusions and the lack of fusion.

The challenge facing the research community in this field is a lack of comprehensive physical understanding of the LMD process. The contribution to the body of knowledge will be attributed to optimising the laser-material interaction of Ti6Al4V and W for structural applications that are relevant to the aerospace and other related industries. Ti6Al4V+W composite is used for turbine blade production due to its outstanding strength and oxidation resistance over the high temperatures encountered.

The aerospace industry can be cited as the major driver for the application of superalloys. Figure 1 shows the gas turbine engine of an aircraft. As shown in the figure, air is drawn into the inlet and compressed before being introduced into the combustion chambers.
In the combustion chambers, the air is mixed with fuel and it ignites. The resulting hot air mixture is released through the back, passing over the turbine blades that are connected to a central shaft by the hot mixture; and it transfers this rotation to the compressor blades at the front. The turbine is a rotating part in the hottest section of the engine, and as such, it is considered critical to the advancement of gas turbine engine performance [Aircraft knowledge accessed 2014].

This research includes deposition with process control. The outcome revealed that the LMD procedure is feasible for the repairing of the turbine blade.

1.2 Problem statement

The high cost of blade replacement makes repairing an alternative to extending the life of the turbine blades; as it delays the need for the replacement of the expensive component. The success of LAM in practical application relies on the detailed
understanding of the relationship between the process parameters and the resulting material properties specifically, since the exceptionally high solidification rates caused by the heat input during the process invariably gives rise to highly non-equilibrium microstructures. While these microstructures are mostly beneficial to the mechanical properties of the resultant material, they cannot be predicted from conventional phase diagrams; and they, have to be studied and optimised experimentally.

1.3 Aim

This project is focused on the laser metal deposition of titanium alloy and up to five weight percent of tungsten for turbine blade repair application in the aerospace industry. The effects of processing parameters on the material characterizations have also been emphasized.

Ti6Al4V+W composites are of interest in many applications, due to their multi-functionality that yields the combinations of properties, such as high specific strength, stiffness, toughness and low coefficient of thermal expansion. The aerospace industry needs the development of materials that can withstand the harsh environment found in a turbine engine. The material works under constant stress; and it experiences deformation, cracks and fatigue.
1.4 Objective

The main objective of this work is to provide a complete physical understanding of the LMD process and its application to the cladding of engineering surfaces that would meet the precise process specifications. The sub-objectives of this study include:

- To laser deposit Ti6Al4V alloy and W powders on a Ti6Al4V alloy substrate.
- To conduct microstructural characterization, microhardness, wear, corrosion and x-Ray diffraction analysis on the laser deposited Ti6Al4V+W composites.
- To optimise the laser-material interaction of Ti6Al4V and W, for structural applications relevant to the aerospace industries.
- To understand the relationship between process parameters and material defects, such as cracks, voids, inclusions and the lack of fusion.

1.5 Hypothesis statement

The development and optimisation of LMD, using tungsten powder to clad turbine blades could result in a more cost effective and increased life extension of blade wear resistance. It is expected that the material characterization techniques employed would lead to the optimisation of the processing parameters to successfully deposit Ti6Al4V and W powders. These optimised parameters (scanning speed, powder flow rate, laser power, and gas rate) would be utilised to develop a model that would enable the optimisation of the process and the commercial application of the cladding of engineering the titanium surfaces that meet precise process specifications.
1.6 Research methods

Titanium alloy and tungsten powders were fabricated by means of the laser metal deposition process. The material properties (microstructure, tensile strength, microhardness, yield strength) of the parent material were determined before the commencement of the laser deposition of the samples. The laser deposited/processed samples were characterised to obtain the optimum process windows. A comprehensive literature survey and background information on the material and manufacturing methods for gas turbine blade was investigated. After the survey of the related literatures, attention was given to the actual experimental set-up of the Design Expert software, using RSM, as well as the results and discussion of the characterisations. The conclusions and the future work were also emphasised in the last chapter of this thesis.

1.7 Project plan

The project plan for the cladding of Ti6Al4V alloy and W is a systematic process that is followed until the completion of the work. The plans are stated as follows:

Materials

- Selection of materials to be deposited (grade 5 Titanium alloy and tungsten powder).
- Material Purchasing.

Parameters optimization

- Calibration of mass flow rates.
- Parametric study.
• Laser beam power.
• Laser scanning speed.
• The design of the gradient.
• Powder particle size distribution.
• Overlapping percentage.
• The rate of compositional change per layer (Flow rate).

Materials characterization

• Optical microscopy
• Scanning electron microscopy
• Microhardness testing
• Wear testing
• Corrosion testing
• XRD testing

1.8 Motivation for research

Titanium and its alloys have gathered much attention; and they have been utilised broadly in the aviation manufacturing industry, because of their superior properties of good strength, better corrosion resistance, good high-temperature, and other properties (Erinosho et al., 2014). Poor wear resistance and high erosion coefficient restrict their potential applications, particularly to the fields where particular surface performance is needed. The joining of a surface metal matrix composite (MMC) layer is a promising answer for enhancing the surface properties of titanium combinations; while their mass properties remain unaffected. The poor wear resistance and the low high-
The LMD method is a good fabricating alternative method for the manufacturing and repairing of titanium alloys parts and components. The portrayal of the materials created utilising this procedure, as for the procedure parameters, is vital for the ideal use of the materials and the procedure itself. However, during the LMD process, the laser beam is utilised to create a melt pool on the metal substrate; and the participating powders are then infused into the melt pool at the same time by gravitational force, in order to form the solidified composite.

In this research, the high quality of five weight percentage of W powder was utilised as strengthening inhibitor to improve the properties of titanium alloy using the LMD process. There is a need to study how different procedure parameters influence the wear resistance execution of the parts created from this procedure, with a specific end goal to have the capacity to effectively assemble a viable practically evaluated material.

1.9 Significance of the research

Within the university:

- Expanding of the research field of the laser metal deposition process.

Generally:
To study the LMD process of Ti6Al4V alloy and W, in order to expand the industrial application of the LMD process within the South African manufacturing industry.

1.10 Project layout

The thesis is organised as follows:

Chapter One introduces the objectives, the significance of the project, the problem statement, the aim and the research method.

Chapter Two will present a review of the related literature; and it is focused on the LMD process, the titanium based materials and the microstructural requirement, the manufacturing methods and the design of the experimental method.

Chapter Three, the experimental set-up is defined, along with the pertinent criteria for evaluating the results of experimental trials and RSM of the experiment is presented, the sample preparation, and the equipment used during the experiment.

Chapter Four presents the experimental results and a discussion. This includes general preliminary observation, results and the analysis from the parameter optimisation and the investigation into the interfaces of the microstructures.

Chapter Five rounds off the research work with a conclusion and some possible future work.
CHAPTER 2

2 LITERATURE REVIEW

2.1 Introduction

This chapter presents the literature review related to the LMD process of titanium alloy and tungsten composites and other similar deposition processes in the realm of additive manufacturing. Furthermore, the following areas will also be covered:

- Laser technology
- Laser based additive manufacturing
- Design of experiments
- Laser metal deposition (LMD)
- Laser cladding
- Material systems of titanium alloy and tungsten
- Wear resistance
- Process parameters
- Properties of titanium alloys and tungsten
- Surface modification of titanium and its alloys, as well as tungsten.

2.2 Titanium and its alloys
The practice of using different alloys in the industry has increased – because of their superior properties and improvements in machining ability. The titanium alloy Ti6Al4V is used mostly in aviation; and it is largely produced in the industry. The main reason for the high demand of Ti6Al4V alloy is because it provides a reduction in aeroplane fuel consumption. According to Ulutan and Ozel (2011) revealed that it reduces fuel consumption because of its low density, high strength at elevated temperatures, high corrosion resistance and creep resistance. High wear resistance promotes their possible application. Protecting Ti6Al4V alloy with a particularly strengthened metal matrix composite (MMC) is an encouraging answer to advancing their surface properties, while maintaining the advantageous bulk properties undisturbed. Titanium alloy has a low density, good yield strength, high corrosion and erosion resistance and moderate strength at high temperatures, making it appealing for manufacturing application.

According to (Boyer, 1996), there are primary reasons why the aerospace industry uses titanium alloy. The reasons are stated as follows:

- **Weight saving**: The high quality to weight proportion of titanium combinations permits them to supplant steel in numerous applications.

- **Space limitation**: Titanium alloys are utilised for landing gear components on business aircraft; where the measure of aluminium components would not fit inside of the landing gear space envelope.

- **Working temperature** (Al, Ni, steel alloys replacement).

- **Corrosion resistance**: Titanium alloy corrosion resistance is superior to both aluminium and steel alloys.

- **Composite compatibility** (it replaces Al alloys).

- **Fatigue strength**: Titanium alloys have much-preferred strength compared to aluminium alloys.
In addition, the weight saving and high strength are also among the reasons for their industrial acceptance. As indicated by (Boyer, 1996), titanium has a high melting point of 1678°C; and it is twice as solid as aluminium; consequently, it contributes to around 60% of the total worth in the titanium industry. The alloy of titanium has 60% lower density than steel or nickel (Donachie, 2000). With all these good properties and high melting point, (Barksdale, 1966) shows that the alloy loses strength when the temperature is above 430°C.

2.3 Applications of titanium and its alloy

Titanium alloy has been widely used in aerospace manufacturing, biomedical industry, chemical industry, petrochemical industry, automobile industry and sports industries because of its high strength and good corrosion resistance (Zhou et al., 2012). It is widely used as implant biomaterials, due to its excellent combination of strength and mechanical properties, such as good corrosion resistance (Kokubo et al., 2004). The outstanding strength-to-weight ratio of Ti6Al4V alloy offers a reduction of aeroplane weight; and as such, this results in a decrease in fuel utilisation and consumption (Armendia et al., 2010). It is also used in jewellery manufacturing to produce coloured surface jewelleries and other decorative properties (Bartlett, 2006).

Titanium alloys are used to increase orthopaedic implant lifetime; and thus, high fatigue strength, good workability, good corrosion resistance and good mechanical properties make the alloy suitable in the orthopaedic implant industries (Guillemot, 2005). Ti6Al4V alloy is mostly utilised in the aerospace industry; and its high strength to weight ratio has led to its introduction into aircraft construction. The turbines have benefited
continuously in the use of titanium alloys and that titanium has been used as an implantation material because of their high tensile strength, lower modulus, high resistance to corrosion and good biocompatibility (Donachie, 2000). The alloys are also used for military aircraft; and they are mostly located in the aircraft engines. A typical example is the turbine blade. Below are general application of titanium alloys (Donachie, 2000):

- **Chemical engineering**: High resistance to corrosion is the major reason for the selection of titanium and its alloys in the chemical engineering application. The prime reason for the use in the chemical engineering environments is the excellent corrosion resistance.

- **Power generation**: Titanium alloys perform better in large steam turbines.

- **Automobile industry**: Many parts can be produced using titanium alloys that can lead to critical weight reduction. In Japan, titanium is used for engine valves and springs.

- **Marine**: High corrosion resistance and good quality: titanium is an attractive candidate for use in the marine environment due to its weight ratios and good resistance in sea water,

- **Military hardware**: Titanium is used for military vehicles, in order to save weight.

- **Sports**: Titanium alloys are used for the manufacture of golf balls, tennis rackets, bicycle frames and running shoe spikes

- **Biomaterials**: Titanium demonstrated excellent resistance to corrosion by body fluid, and shows lower elastic modulus. In biomaterial field, titanium are used for prosthetic application like bone and implant object

- **Architecture**: Recently in Spain, titanium is used for building structures like Museum.
Figures 2.1 (a) to (d) show pictures of the areas, where titanium alloys are applied.
2.4 Overview of tungsten (W)

According to (Pierson, 1996) reported that tungsten has low reactivity, low friction, high melting point, very high hardness, good thermal, as well as electrical conductivity.

It is well known that tungsten exhibits a melting temperature of 1400–1600°C. However, other studies have shown that tungsten can attain a melting temperature that is lower than 1400°C (Ma and Zhu, 2010).

Tungsten stands out amongst the most generally utilised wear safe coatings in industry, specifically in aviation, automobile, and other transportation systems. As indicated by (Koc and Kodambaka, 2000) shared the same view as (Pierson, 1996) that tungsten has a high melting point, very high hardness, low friction coefficient, low reactivity, high
oxidation resistance, and good thermal and electrical conductivity. According to (Li et al., 2010) revealed that the suitable reinforcement for Ti6Al4V alloy to strengthen its composite is by agglomerating it with tungsten. This is actually due to their similar thermal expansion coefficients.

The combination of Ti6Al4V and W has also shown a minor crack tendency, depending on the mixing proportion (Choe et al., 2005).

Recently, it has been demonstrated that tungsten(W) can be added to a titanium matrix bringing about Ti6Al4V + W with a remarkable strength and hardness and reduced ductility (Choe et al., 2005)

The following properties: wear resistance, good quality strength and good thermal conductivity, low erosion rate and low thermal expansion have made tungsten attractive for use in the different range of applications (Antusch et al., 2015). In addition, it has high dissolving temperatures, high hardness values, high chemical resistance, and good electrical and thermal conductivities. Russian aircraft and aerospace used tungsten to strengthen the blades of the aircraft (Moiseyev, 2005).

Tungsten alloys contain materials that can perform different functions in fuel cells (Antolini and Gonzalez, 2010). Other applications of tungsten include light bulbs, cutting tools, jewellery making, mining tools, medical application, saw blades, milling cutters, ammunition, and application in automotive, aerospace industry, in electronics, bullet-proof material and wear parts. Tungsten has a high melting temperature and a low vapour pressure; it is used in high-temperature applications, such as light bulbs. The
tungsten metal powder will be used to reinforce the titanium alloy (Ti6Al4V), in order to improve its wear resistance properties.

2.5 Application of titanium alloys and tungsten

Titanium based tungsten alloy (Ti6Al4V + W) is an important metal in the production of high-performance materials. Ti6Al4V + W composite is mostly used in the automobile and aeroplane industry. This research will focus on the application of titanium-based tungsten metal for the aerospace industry application.

2.5.1 Aerospace industry application

The titanium alloy and tungsten composites are important in the aerospace industry. However, in order to improve passenger and pilot comfort, and reduce vibration in the aerospace industry, critical and high strength materials can achieve this with the use of Ti6Al4V+W. Titanium and tungsten are used in jet engine components and in airframe applications, where high strength is required. Because of the high cost of repairing the worn jet engine blades, the LMD process is the preferred means to repair the worn blades (Akman et al., 2009).

Figure 2.2 shows the titanium-tungsten turbine blade.
The effect of commercially pure titanium and tungsten on the aircraft jet engine forged blades made of titanium alloy WT3-1 to repair worn abutments surfaces was conducted by (Klimpel et al., 2011). The mixture of the powders (40 – 50 wt % of Ti and 60 – 50 wt % of WC) were deposited on titanium alloy plates. A high quality of Ti and WC deposition was achieved on the substrate with a uniform distribution of spherical WC with the lattices of Ti. The average microhardness values of the deposited composites ranged between 413 HV - 460 HV.

These authors (Klimpel et al., 2011) concluded that the laser parameters, such as the laser power and the powder flow rate have a controllable effect on the hardness values of the composites. The LMD process provides the highest quality of repairing of worn turbine blade formed from titanium-tungsten (Klimpel et al., 2011).
2.5.2 Army platforms

Titanium alloys have been used in the aerospace industry for reducing weight in airframes and engine parts. The high cost of titanium has prevented the application into military vehicles. However, in recent years, the cost of titanium alloy has dropped; and titanium has become an option for application on the army platforms due to its weight-reduction and high strength. Titanium in the military industries has been used to reduce weight and to increase speed or mobility (Gooch and Ground, 2010). The titanium alloy and tungsten have also been used in the manufacture of military-vehicle components.

2.6 Coating application methods

Surfacing methods are used to apply a protective coating to restore damaged turbine blades by filling the voids. The surface preparation is very vital when applying coatings; since the surface condition would dictate the adhesive nature of the coating; and any contamination on the surface can cause diminished mechanical properties in service. The coating is also affected by the base material in the following manners (Day, 2004).

- Composition – Diffusion can occur between the base material and the coating; and the surfacing material is not necessarily the same as the substrate.
- Mode of surface preparation – There are varying methods and processes, such as Arc welding, laser powder fusion, and thermal spray coatings.
- Degree of surface roughening.
- The temperature that the substrate reaches during the process.
2.6.1 Laser powder fusion

The laser process will be used in this study to apply hardfacing material. Laser powder fusion is often used in welding and cladding application for aero-engine components. The fine metallic powder is injected into a CO\textsubscript{2} gas or solid-state YAG laser beam; the particles undergo rapid melting; and they are deposited onto the substrate and solidify after cooling. In a single pass, the thickness of the deposit can be adjusted between 0.005mm and 0.030mm (Day, 2004). The laser process fusion accomplishes weld build-up with little heat transfer; the heat input is in an order of magnitude lower than the conventional methods, such as the Arc welding process. High precision is attainable with the laser process; with the capability of allowing the depositions of near net shapes. Figure 2.3 shows the schematic view of the laser powder build-up process.

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{laser_powder_build-up.png}
\caption{Figure 2-3: laser powder build-up (Day, 2004)}
\end{figure}

1 Laser beam  
2 Cladding  
3 Powder  
4 Melt-proof  
5 Substrate  
6 Cover gas  
7 Layer  
8 Heat-affected zone
2.6.2 Plasma transferred arc (PTA)

The deposited material begins in the powdered form, thus permitting the deposited composites to be premixed in powder shape by consolidating the weight rates of the substances. This likewise implies that PTA can be applied on materials that cannot be effectively formed into wires or bars; or that are not metallic by any means. Cermet’s (WC, TiC, and so on.) can be utilized to make super-hard layers. Some powder deposited conveyance frameworks include shifting an incited vibration in the powder tank to change the powder sustenance rate. A conveyancer gas then supplies the powder to the light. Cracking can happen when the deposit and the base materials contrast in heat extension. Care must be taken when working with alloying components of varying densities (Owa et al., 2002).

2.6.3 Thermal spray coating

Thermal spray coatings are incorporated here; as they are a suitable method for applying wear safe coatings to gas turbine parts. A thermal spray coating is made up of two steps; melting of a wire and/or spraying of molten material on the workpiece. Thermal spraying can be applied to any of the following processes:

- Combustion wire spraying
- Combustion powder spraying
- Arc wire spraying
Laser is an acronym for Light Amplification by Stimulated Emission of Radiation. H. Theodore was the first person to build the laser in 1960. The laser can be used in different applications, such as laser printers, barcode scanners, laser surgery, laser lighting displays, laser measuring and many more.

There are numerous types of laser that are accessible for exploration, industrial, medical, and business users. Most of the lasers are classified by the kind of lasing medium they use; thus, lasers comprise solid state lasers, dye lasers, gas lasers, excimers or semi-conductor lasers (Csele, 2004). The four common types of laser are explained as follows:

a) Solid state lasers: The laser has lasing material distributed in a solid matrix. The ruby or neodymium YAG (yttrium aluminium garnet) is a typical example of a solid state laser. The solid-state lasers are lasers whose active medium is typically a minority ion in a solid-state host. However, this host is usually a single crystal with about 1% of a different species, such as a neodymium ion (Nd\(^{3+}\)) dropped into the solid matrix of the host. The "neodymium: YAG" (Nd:YAG) laser consists of a crystal of yttrium aluminium garnet (YAG) with a small amount of neodymium added as an impurity (J.J. Ewing, 2000).

b) Gas laser: This type of laser is the most common gas lasers; and they are used for cutting hard materials. An electric current is discharged through a gas to produce coherent light. An example of a gas laser includes CO\(_2\) gas laser, Helium-Neon (HeNe) laser, Nitrogen Laser, Transversely Excited Atmospheric (TEA) laser.
c) Dye laser: They are used in liquid solutions. The dye lasers produce an output whose wavelengths are in the visible, ultra violet and near infrared spectrum; and they vary from 390 to 1000 nm. The output beam diameter is typically 0.5 mm and the divergence of the beam varies from 0.8 to 2.0 milliradians.

d) Semiconductor laser: This type of laser is sometimes called a diode laser; and they are not solid-state lasers. They use low power for their operation. The semiconductor laser is very small in size and appearance. It is similar to a transistor; and it has the operation of a LED. Gallium Arsenide is the material used in the laser.

Most of the above laser types are not capable of processing metallic materials; because of the low power distribution [Laser training, Accessed 2016]. Laser absorption into material relies upon the type of laser employed. Table 2.1 shows the types of laser and their applications in the industry.

Table 2-1: Types of laser, application and operation wavelength (Laser types, accessed 2016)

<table>
<thead>
<tr>
<th>Laser Types</th>
<th>Operation wavelength</th>
<th>Application</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gas Laser</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Helium-neon laser</td>
<td>632.8 nm- 3.3913 μm</td>
<td>The applications for helium-neon laser are a barcode, spectroscopy, and holography.</td>
</tr>
<tr>
<td>Argon Laser</td>
<td>244nm - 528nm</td>
<td>Argon laser used in retinal phototherapy, lithography</td>
</tr>
<tr>
<td>Laser Type</td>
<td>Wavelength</td>
<td>Description</td>
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<td>-------------------------</td>
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<td>----------------------------------------------------------------------------</td>
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<tr>
<td>Krypton laser</td>
<td>416 nm - 799.3 nm</td>
<td>The Krypton is known as noble-gas ion laser and are used for light shows</td>
</tr>
<tr>
<td>Carbon Dioxide Laser</td>
<td>10.6 µm, (9.4 µm)</td>
<td>This laser is a mixture of helium and nitrogen and is applicable for cutting, welding, dental laser.</td>
</tr>
<tr>
<td>Carbon Monoxide Laser</td>
<td>2.6 µm - 8.3 µm</td>
<td>Carbon monoxide laser with the wavelengths of up to 8.3 µm have been around for decades and not been used as much as carbon dioxide laser which has an output wavelength of 10.6 µm. Carbon monoxide laser are applicable for engraving and welding,</td>
</tr>
<tr>
<td>Excimer Laser</td>
<td>193 nm - 353 nm</td>
<td>Excimer laser are capable of performing laser surgery</td>
</tr>
</tbody>
</table>

**Chemical Laser**

<table>
<thead>
<tr>
<th>Laser Type</th>
<th>Wavelength</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen Fluoride Laser</td>
<td>2.7 µm - 2.9 µm</td>
<td>Hydrogen fluoride laser are used in research for laser weaponry</td>
</tr>
<tr>
<td>Deuterium Fluoride Laser</td>
<td>3.6 µm - 4.2 µm</td>
<td>Deuterium laser are used military laser prototypes</td>
</tr>
<tr>
<td>COIL</td>
<td>1.315 µm</td>
<td>COIL laser are used for military laser</td>
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</tbody>
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<tr>
<th>Laser Type</th>
<th>Wavelength</th>
<th>Applications</th>
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<tbody>
<tr>
<td>Agil laser</td>
<td>1.315 µm</td>
<td>Agil laser is used in aeroplane industry</td>
</tr>
<tr>
<td>Dye Laser</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dye Laser</td>
<td>390 nm - 640 nm</td>
<td>Laser medicine, spectroscopy, birthmark</td>
</tr>
<tr>
<td>Metal Vapour Laser</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Helium cadmium</td>
<td>441.563 nm - 325 nm</td>
<td>Helium laser are used for printing industry and fluorescence</td>
</tr>
<tr>
<td>Helium mercury</td>
<td>567 nm - 615 nm</td>
<td>Helium mercury and helium silver are used for scientific research</td>
</tr>
<tr>
<td>Helium silver</td>
<td>224.3 nm</td>
<td>Scientific research</td>
</tr>
<tr>
<td>Gold vapour laser</td>
<td>627 nm</td>
<td>This laser is mostly used in the photodynamic therapy</td>
</tr>
<tr>
<td>Solid state laser</td>
<td></td>
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</tr>
<tr>
<td>Ruby laser</td>
<td>694.3 nm</td>
<td>Tattoo removal, holography</td>
</tr>
<tr>
<td>Nd: YAG laser</td>
<td>1.064 µm</td>
<td>Material processing, hair removal</td>
</tr>
<tr>
<td>Laser</td>
<td>Wavelength (µm)</td>
<td>Applications</td>
</tr>
<tr>
<td>------------------------------</td>
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<td>------------------------------------------------</td>
</tr>
<tr>
<td>NdCrYAG laser</td>
<td>1.064 µm</td>
<td>Experimental production of Nanopowders</td>
</tr>
<tr>
<td>Er:YAG laser</td>
<td>2.94 µm</td>
<td>This laser is used in dental laser, skin resurfacing</td>
</tr>
<tr>
<td>Nd: YLF</td>
<td>1.053 µm</td>
<td>This laser is mostly used for pulsed pumping</td>
</tr>
<tr>
<td>Nd: YVO laser</td>
<td>1.064 µm</td>
<td>used for continuous pumping</td>
</tr>
<tr>
<td>Holmium YAG</td>
<td>2.1 µm</td>
<td>This laser is used for removal of kidney stone and tissue ablation</td>
</tr>
<tr>
<td>Yb: YAB laser</td>
<td>1.03 µm</td>
<td>Optical refrigeration, material processing</td>
</tr>
<tr>
<td><strong>Semiconductor laser</strong></td>
<td></td>
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<tr>
<td>Laser diode</td>
<td>0.4 µm - 20 µm</td>
<td>holography, telecommunication</td>
</tr>
<tr>
<td>Gan</td>
<td>0.4 µm</td>
<td>Blu-ray Discs</td>
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</tbody>
</table>

2.8 Laser based additive manufacturing (LBAM)

LBAM is capable of manufacturing innovation that can be broadly applied to part preparation, surface modification and fabrication. The application of additive manufacturing is varied from the development of super alloy jet engine parts to titanium
for aerospace and the repair of components. Several projects have been developed to encourage the broader use of the technology outside the traditional manufacturing world. Laser-Based Additive manufacturing is different from the traditional manufacturing process; since the parts are generally acquired by machining, laminated, injected or moulded raw material. Additive manufacturing comprises building an object from the beginning, or a semi-completed part acting as a substrate to a final component using the CAD program. Laser deposition is the mixture of an alloy powder on a substrate with the application of a laser beam.

The construction with the anticipated hardfacing strategies, using arc welding and thermal spraying, or laser cladding, can deliver much-improved coatings, with a dense and good metallurgical bonding to the substrate (Choe et al., 2005). The laser deposition can also be defined as the blending of an alloy particle on a substrate. These authors concede that the dissolving of the substrate happens just inside of a thin layer. Another study by (Ion, 2005) showed that the laser metal deposition process is the same as the additive manufacturing procedure, which is that of depositing a durable metallurgically bonded coating.

2.9 Laser metal deposition (LMD)

Various names for rapid assembling methods are utilized, including Laser engineered net shaping (LENS) (Shao and Yan, 2005, Griffith and Schlienger, 1999), Direct Metal Deposition (DMD)(Mazumder et al., 2000, Bhattacharya et al., 2011), laser solid forming (LSF)(Yu et al., 2010, Liu et al., 2013). All these comparable systems are connected to the complete creation of a 3-D image.
LMD is a powder-based procedure to develop 3D parts layer-by-layer, in which the powder or wire is dissolved by the power source and deposited on the substrate surface. The powder is delivered by an inert gas; and it passes through the depositing nozzle to form a layer-by-layer composite (Kobryn and Semiatin, 2001). LMD has the capacity of manufacturing difficult component in the medical and automotive industries (Mahamood, 2017). Another standpoint for LMD process is that, it has the ability to repair damaged parts that were not repairable previously and also complex part can be created by LMD (Bergan, 2000).

After hardening, a strong metallurgical bonding layer is coated on the substrate. Laser melting deposition (LMD) innovation involves utilising laser power, as a heat source to melt the powder or wire to form an object. As indicated by (Liu et al., 2013), revealed that the laser metal deposition is an innovation that creates a strong metallurgical bond, deposited on the substrate.

LMD is an additive manufacturing technique that builds components from Computer Added Manufacturing (CAD) layer-by-layer from the powder (Lü et al., 2001). Through the additive manufacturing processes, powder is heated to form a solid object; and a complex part is built (Zaeh and Ott, 2011). Powder bed fusion (PBF) is one of the laser additive manufacturing (LAM) technologies, which uses a laser beam to solidify the powdered metallic materials layer-wise to form functional 3D parts (Islam et al., 2013). The process itself is often called by different names, e.g. selective laser sintering (LSL) and selective laser melting (SLM), and direct metal laser sintering (DMLS). The three-dimensional nature of the process and the volumetric energy input describe the
amount of energy input through the selective laser melting process. This is usually applied with the powder bed fusion technologies.

The energy is calculated according to the following equation:

\[ E = \frac{P}{v \cdot h \cdot d} \]

2.1

Where

- \( E \) = Volumetric energy input in (J/mm)
- \( P \) = Laser Power in W
- \( v \) = scan speed in mm/s
- \( h \) = Hatch spacing in mm
- \( d \) = Layer thickness in mm

The spot size is not always varied; it is only the processing parameter such as laser power, scanning speed. These parameters can cause a change in the energy density. The LMD process is considered to be a robust process; because the laser power create a melt pool on the substrate surface or work piece surface and the powder is fed directly into the interaction zone (Li et al., 2016). LMD can produce difficult product and has the ability to use more than one material simultaneously.

As indicated by (An and Xudong, 2004) gave the different advantages of Laser metal deposition, which are: low cost, small heat affected zone, digital flexible processing, less distortion and being environmental friendly. A summary of the advantages of Laser metal deposition is highlighted in the next paragraph. Figure 2.4 shows the repairing of blades by LMD process. Figure 2.5 presents the Kuka robot cladding.
According to (Saqib et al., 2014), the laser metal deposition technology is a procedure that utilizes the laser beam or a laser source to melt the material on the substrate to form a solid object, bearing in mind that the end goal is to accomplish metallurgical bonding with insignificant weakening of included material and substrate, so as maintain the dilution of the added material and substrate, in order to retain the original properties of the coating material. Figure 2.6 shows the laser cladding process.
The powder particles are infused into the melted pool via the axis nozzle; while at the same time, a high powered laser beam dissolves the deposited material over the substrate.

Right now, there is a developing enthusiasm in the manufacturing sector for utilising additive manufacturing innovations for metallic production, functional components, repairing of the tool, and turbine engine parts. Most engineering applications require materials that have high quality and high corrosion resistance for performance and long-term reliability. Laser deposition technology is being investigated as a practical answer for additive manufacturing.

Laser cladding is a low-cost saving and exceptionally adaptable repair system, which is, as of now, being applied in the aircraft engine industry to build-up the worn tips of blades (Zaeh and Ott, 2011).
Laser cladding has been characterized as a procedure, which is used to merge with a laser beam another material that has particular metallurgical properties on a substrate whereby only a thin layer of the substrate must be broken up, in order to fulfill metallurgical bonding with immaterial debilitation of the included material and the substrate, remembering that the true objective is to maintain the primary properties of the covering material (Vilar, 1999).

The aim of the laser cladding is to achieve good quality clad layers; and quality implies no deformation of the material, no porosity, a good adhesion to the substrate and a low dilution of the coating material by the substrate. The deposited layer-by-layer metal deposition process shows similar good performance as that of the alloy powdered materials in terms of corrosion, hardness, mechanical properties, wear resistance, and fatigue (Vilar, 1999).

A vital part of the laser surface cladding process is to supply cladding material to the substrate. We have two powder coating methods. These are pre-placed powder and powder injection. The end goal of this study is to concentrate on the powder injection process. These powder injection techniques begin with the formation of a dissolved pool in the substrate, where the deposited powder particles are being fed into the dissolved pool. But before the particles are fed into the melt pool, the material, such as wire or powder particles absorbs laser power on the way through the laser beam. Gas transported the powder particles and injected them into the dissolved pool. In the laser deposition process, the different metallurgical compounds could be produced by regulating the velocity of the powder flow, the nozzle stand-off distance and other
parameters. The cladding layer and the substrate zone are realised; while avoiding any excessive melting of the substrate.

Laser metal deposition processes have many advantages; and they are summarised as follows:

a) The laser metal deposition process demonstrates the ability to fulfil repair requirements (Saqib et al., 2014, Graf et al., 2012). Repair is done to prolong the life of the part. For example, the turbine blade uses the same technique (LMD) to repair the edge of the blade (Klimpel et al., 2011). The high cost of turbine blade replacement makes repairing an alternative to extending the lives of blades; since it delays the need for the replacement of expensive components. LMD techniques in the aero engineering industry are used in both new and repair parts (Saqib et al., 2014).

b) Digital flexible processing (Saqib et al., 2014). This process provides much flexibility for part design. LMD is more flexible; because modification can be done on the old part or design.

c) Small heat-affected zone. The heat-affected zone and the base material are evenly distributed (Liu et al., 2016).

d) Less distortion (Saqib et al., 2014). The distortion is reduced through a reduction in the energy input of the process.

e) The new part is built directly on the old part (Song et al., 2006). LMD creates a metallurgical bond that forever attaches the deposited metal onto the base material (Shao and Yan, 2005).
There are a number of challenges facing the laser metal deposition process; these challenges will be discussed in the next section.

Despite its huge potential, laser metal deposition has its own challenges. The main challenge is to control the process; and in order to achieve a homogeneous layer with specified properties, the laser power and the powder must be frequently controlled (Shao and Yan, 2005). Another limitation is that, the technique is relatively new and the procedure is not yet completely or fully understood. The procedure cannot be controlled manually; Computer Numerical Control (CNC) is required, and the powder cost is typically greater than that of wire.

2.10 Material systems

Investigation of Ti6Al4V alloy and W deposited by the LMD process will be covered extensively in this study. These materials are used in various applications; and this will be the first attempt to combine these materials with each other by using the laser powder process. An overview of W and Ti6Al4V is given below for reference and comparison purposes to this study.

2.11 Corrosion resistance behaviour of titanium

Corrosion refers to material loss due to a strong surface of the mechanical contact between that surface and the liquid or strong particles. According to (Rajendran, 2012) described corrosion as that which is brought on by the imposition of strong particles or water beads. The compressors of aeroplane engines are inclined to show performance
losses; because of corrosion on the blades’ sharp edges, while being worked in areas with a dusty and sandy environment; and when single, fly powder(sand), salt and ice, precious stone, or volcanic slag are ingested (Rajendran, 2012). The coating prevents the sharp edges of the blades from the early loss of material. The high temperature in an aeroplane environment contains pollutants, such as sodium, sulphur and various halides; and this requires special attention to the problem of hot corrosion.

The titanium alloy is highly resistant to general corrosion in sulphuric acid, sodium hydroxide and sea water. According to (Giardini-Guidoni et al., 1998), their study has shown that titanium coating has become an increasingly important area in corrosion. The tungsten particles have been broadly utilized for boring tools, high-speed steel, tool steel, wires, rods, mill products, cutting and mining, because of their high hardness and outstanding wear resistance (Koc and Kodambaka, 2000).

As indicated by Rajendran, 2012, suggested that titanium alloys should perform better under environments of corrosion fatigue; because of their good resistance to corrosion. Titanium has exceptionally corrosion resistant properties in the human body, when used as an implant.

Titanium is used extensively in prosthetic devices due to its corrosion resistance (Donachie, 2000). Titanium alloy (Ti6Al4V) also shows excellent corrosion resistance in the engine-combustion environment and oxidising conditions that cause various types of degradations, Ti6Al4V also shows excellent resistance in sea water (Pascal et al., 2003). The corrosion resistance of the titanium enhancement was credited to the presence of Ti metallic phases that generate the protective oxide (Wong et al., 2012). The next section summarises the process parameters.
2.12 Process parameters

To effectively deposit powdered material on a substrate, or for repair by using a laser-cladding process, the impact of numerous parameters for instance, such as the laser-control rate, travel speed, the powder feed rate should be well-understood, in order to create the wanted geometry and anticipated shape with the related variations. As indicated by (Klimpel et al., 2011), discuss the process parameters for the direct metal deposition of titanium and W. This research focuses on the different levels of gas flow rates, laser power, traverse rate, and several levels of the added titanium. The study was performed to build up the possibility of utilising LMD’s ability to deposit Ti6Al4V + W particles on a titanium substrate.

Different experiments were conducted on the deposited samples, in order to check for the quality of deposition, such as mechanical testing, visual investigation, and microstructure examination. From the outcomes of the analyses, it was decided that Ti6Al4V + W powder could be the cladding on the titanium alloy substrate with a quality appropriate for manufacturing applications.

During the laser cladding process, the laser parameters used influenced the quality, the microstructure, as well as the clad layer shape and the properties. The main reason for the laser deposition with pre-placed particles and particle injection is to understand the result of the parameters’ variation on the cladding; and to decide on the appropriate parameters.
2.12.1 Influence of laser scanning speed variation on the geometrical dimensions of the laser clad layer

The most important parameter that affects the size of the laser clad layer is the scanning speed; and it also affects the energy distribution as well. To attain the scanning speed, the laser head should move against the fixed substrate. By doing so, the length of time that the material interacts with the laser power is determined. The experiments will be performed for fixed laser power, as well as the laser beam spot diameter of the pre-placed powder, in order to isolate the effect of the scanning speed.

A lower speed, depending upon the power of the laser, can bring about considerable weakening of the materials being prepared for melting. Likewise, the scanning speed could bring about deficient preparing of the materials, for example, dissolving or no melting at all. There is a strong relationship between the scanning speed and the laser power, despite the fact that the relationship adversely affects the properties of the material.

2.12.2 Effect of laser power variation

Laser power is a vital parameter that affects the size of the laser clad layer and the distribution energy. It is important to describe the impact of laser power variation on the dimension and shape of the laser clad layer. The laser power is known to influence the material properties – from the physical properties to the microstructural characteristics (Mahamood et al., 2013). The laser power can be changed during the laser cladding tests utilising a pre-put powder strategy. The increase of laser power makes the molten pool to absorb more energy; but if the laser power is too high, it can bring about dilution
of the deposited material by the introduction of melted substrate; and the desired results may not be achieved. On the other hand, a too-low laser power can bring about porosity in the deposited material.

This study will focus only on the influence of the laser power on the shape and geometrical dimensions of the laser-clad layer.

2.12.3 Impact of laser beam diameter variation on the laser clad layer dimensions

The laser beam spot diameter is another parameter that influences the geometry of the laser clad layer and the distribution of energy. The size of the laser beam spot diameter will remain constant at 2 mm to perform the laser-cladding experiments, using the pre-placed powder method. The diameter size is inversely correlated to laser power, the smaller the diameter size, the more concentrated the laser power (Senthilkumaran et al., 2009, Steen and Mazumder, 2010).

2.12.4 Gas flow rate

The powder particles are transported by the gas flow rate into the laser beam. The powder is conveyed via the carrier gas that shields the particles from contamination. Gas flow rates can vary; and it is important to select the correct flow rate for each application; as this can improve the efficiency and ensure a quality weld. It also varies, according to the type of application — for example, manual welding typically requires a lower flow rate than mechanized or automated welding systems.
The flow rate protects the powder from environmental contamination; and it protects the gas during the laser metal deposition process. However, with high gas flow rates, the particles become more prone to bouncing off the melt pool surface. Hence, an optimum gas flow rate would exist (Mazumder et al., 1997). According to Montgomery (2008), the gas flow rate also affects the properties of the deposited particles.

2.12.5 Impact of the powder feeding rate

The distribution of the tungsten powder particles in the deposited layer is a key factor for the reinforcement phases in the metal matrix composition (Li et al., 2016). At the point when the material flow rate is too high, contingent upon the available energy density, the vast majority of the material may not be melted; because the available power may not be sufficient to melt the material; and this would bring about poor material utilization (Mahamood et al., 2013).

The powder feed rate has an instant influence on the distribution of the powder density in the melt pool and on the microstructure during the laser-metal deposition, as well as the layer height (Liu and Dupont, 2003).

2.13 Microstructural evolution of LMD of titanium alloys

Titanium alloys can display a wide assortment of microstructures contingent on the material composite, processing, and the deposition treatment. Titanium alloy displays many varieties of phase transformations; and these transformations are associated with \(\alpha\) or \(\beta\) phases (Joshi, 2006). An alpha \(\alpha\)-grain microstructure has a hexagonal
crystallographic structure; while the beta $\beta$-grain microstructure has a body-centred cubic structure.

During the machining operations, the workpiece material is exposed to the mechanical and chemical reactions that can be prompted to strain hardening and the recrystallization of the material. Because of the strain hardening procedure, the material may get to be distinctly harder, but less ductile; and recrystallization may cause the material to end up distinctly milder and more ductile. The microstructure can be complex; but it is rigorously controlled to provide the best mechanical properties, depending on the service application.

The study of Ti6Al4V alloy revealed that a thin layer of plastic distortion is shaped in the quick sub-surface of the workpiece; and in this manner, the thickness of the distorted layer is expanded, because of microstructural modification (Che-Haron and Jawaid, 2005).

It is fundamentally important to repair an exhausted or harmed part for re-use, as opposed to merely replacing them (Rajendran, 2012). The development requires the right dimensional tolerance; and it needs to follow the surface smoothness prerequisites.

As a moved surface-change strategy, the laser cladding pulls in the usage of new materials mix, surface repairing and 3D geometric time. Porosity in the deposition layer will reduce the quality of the cladding and this must be avoided through applying proper standard LMD parameters. Regardless thereof, even for exceptional applications, porosity is ubiquitous; and it is sometimes required as in a permeable medicinal insert.
Loss of material at the top of the blade and distortion of the blade are visible in Figure 2-7.

![Damage turbine blade (Esaklul, 1992)](image)

**Figure 2-7**: Damage turbine blade (Esaklul, 1992)

There are different types of microstructures found in the titanium alloys, which are fully lamellar, bimodal, and equiaxed. The structures are finer, as the cooling rate is increased, which tend to improve their strength.

2.13.1 **Bi-modal microstructures**

Bi-modal or duplex shapes can be obtained in alpha and beta titanium alloys by mechanical processing. In this situation, the lamellar microstructure is deformed in the \((\alpha+\beta)\) stage field; and afterwards, it recrystallizes promptly to a blend of equiaxed \(\alpha\) and \(\beta\) grains, when the lamellar microstructure is deformed (Joshi, 2006). After cooling from the duplex structure, at the \(\beta\) transition temperature, the grains are changed again to a lamellar structure (Lütjering, 1999). The bimodal microstructure is shown in Figure 2.8.
Bi-modal microstructures result from the combination of lamellar and equiaxed microstructures; and they display a good balance of properties.

2.13.2 Lamellar microstructures

According to (Lütjering, 1999) revealed that, lamellar structures have high resistance to fatigue crack and high toughness. The alpha colony size (a function of the cooling rate) is the most important microstructure of the mechanical properties. The cooling rate determines the lamellar fine or coarse structure. The cooling rate has a major impact on the development of the microstructure (Lütjering, 1999). In addition, the lamellar structure gets to be finer, as the cooling rate is increased; and the finer structures are prompted to increase their strength; while the coarse structure leads to creep resistance (Joshi, 2006). The alpha and beta lamellar microstructures of titanium alloy are shown in Figure 2.9.
2.13.3 Equiaxed microstructure

Equiaxed microstructures result from the recrystallization procedure. Equiaxed alpha microstructures give high quality, flexibility and moderately low fracture toughness; while the lamellar structure gives great crack durability, but with some compromise on strength and ductility (Rack and Qazi, 2006, Joshi, 2006).
2.14 Properties of titanium and tungsten

Mechanical and electrical properties of titanium and tungsten are mostly influenced by process parameters of laser power (Brandl et al., 2011). Laser cladding properties, such as the mechanical properties, the structure, and the grain size will be discussed in this section.

2.14.1 Mechanical properties

The mechanical properties of tungsten powder and titanium alloy are of great interest. As of late, it has been demonstrated that tungsten particles can be added to a titanium matrix, bringing about a Ti6Al4V + W composite with extraordinary strength and hardness; and minor ductility penalty and ductility reduction is achievable by increasing the W particles (Choe et al., 2005). Reinforcing mechanisms can be recognised for the expansion in both strength and hardness of the Ti6Al4V and W composite, when contrasted with Ti6Al4V alloy (Dewidar, 2010). In addition, tungsten is non-magnetic; and it is well known to reduce the elastic modulus of titanium (Choe et al., 2005).

According to (Liu et al., 2015) the crack tendency of the W/Ti6Al4V composites is very low; and the reaction between Ti6Al4V + W is moderated at high temperatures; and in this way, W is viewed as a necessary metal for strengthening the Ti6Al4V. The coatings around the W particles result in great bonding at the Ti6Al4V + W boundaries; and they show the outstanding ability of load transfer (Liu et al., 2015).
An article that discusses the titanium blocks deposited by the additive manufacturing process (Brandl et al., 2011) showed that the cladding of titanium could attain good strength and ductility properties that satisfy the aviation industrial specifications. Furthermore, the article has also reported on the mechanical properties and the chemical compositions of additively manufacturing titanium squares and contrasted them with those of the plate material and the aviation determinations.

Titanium reacts with any material that comes into contact with and this results in the difficulty to machine titanium (Arrazola et al., 2009) and the poor wear resistance of Ti6Al4V (Dewidar, 2010). Titanium alloys are perceived as hard-to-cut materials, because of their low thermal conductivity, high chemical action and small elastic modulus (Yang et al., 2016).

The current research will focus on the mechanical properties of Ti-6Al-4V and W; since further studies revealed that tungsten-reinforced composite coatings are successively produced on Ti6Al4V (Saqib et al., 2014).

### 2.14.2 Titanium hardness

The titanium hardness on the surface layer is usually high, because of the phase composition that consists of titanium nitrides. According to Chen et al. (2008) reported that the high microhardness value of the surface layer relies on the formation of the reaction product. As indicated by (Zhecheva et al., 2005), revealed that the high hardness values on the surface layer depends on the reaction of the material and the processing parameters. As indicated by (Kamat et al., 2016) conducted an investigation
into the surface nitride of titanium; and there was a hardness improvement after the nitriding process.

2.14.3 Grain size

Grain size is considered to be the most important factor in alloy performance for the microstructure of the deposited layer; and it is one of the first features that should be considered. An important effort in both research and the production of alloys is directed at the control of grain growth and grain size distributions.

The Hall-Petch equation provided an overall relationship to relate yield stress and other mechanical properties to grain size, based on the idea that the grain boundary acts as obstructions to disengage the movement (de Araújo Alécio et al., Petch, 1953). While the later work emphasised the role of the grain boundaries, as dislocation sources; and the experimental observations confirmed the general relationship between the decreased grain size and the increased yield (Li, 1963).

Grain size and structure additionally assume an essential part in the strength and temperature capabilities of a turbine blade material. Grain structure is usually dependent on the manufacturing method used to fabricate the blade.

2.15 Wear resistance

Wear exists when two solids under load experience relative motion at their interface. Many types of motion can lead to wear, because of varying the kind of motion and the
material. The main types of wear are: adhesive, abrasive, fretting leading to fatigue wear, and erosive (Day, 2004).

Poor wear resistance amid the machining procedure of Ti6Al4V is easy to hold to the surface of the instruments, which causes adhesive wear (Long and Rack, 2001). Moreover, the chemical reaction of the titanium at high temperatures brings about the creation of a solid layer leading to work hardening and declining tool life span (Matikas and Nicolaou, 2009).

Titanium has a poor wear resistance; but coating it with W powder would improve its wear resistance (Zhou et al., 2012). Laser cladding gives a good wear resistance to the substrate. Laser deposition can deliver superior coating, for example, at low dilutions, high wear resistance and the metallurgical bonding to the substrate (Zhou et al., 2012). For the purpose of this study, abrasive wear will be used to characterise the material transfer caused by hard granules. The wear test will focus on the effect of micro-hardness.

On the other hand, tungsten has a very good wear resistance. There are several recent references to the deposition of tungsten coating, claiming good wear resistance (Kitamura et al., 2008). Tungsten has very high deposition efficiencies up to 90% with the mechanised system that reduces the manufacturing costs and increases productivity (Zimmerman et al., 2006). Also, there is the high substrate adhesion and inter-particle cohesion with the high mechanical strength (Schmidt et al., 2006). (Vreeling et al., 2002) studied the wear behaviour of Ti6Al4V via laser melting; and they noted a significant improvement in the strength of Ti6Al4V compared with that of the uncoated alloy. As
indicated by (Chen et al., 2008) revealed that the microhardness value on the top of the cladding of the Ti6Al4V and W composite produced by the laser melting process can be ascribed to an increase in the hardness value with the formation of the Ti6Al4A+W composite.

2.16 Surface modification of titanium and tungsten

Tungsten powder is an important material, due to its high strength, high hardness and wear resistance; however, it can be machined by using non-conventional machining procedures, such as surface alloying (Ho and Newman, 2003, Thomas, 1991), surface nitride (Kuzin, 2003) and surface finish (Gangadhar et al., 1991, Mohri et al., 1993, Tsunekawa et al., 1994, Samuel and Philip, 1997).

Conventional methods have the limitation that includes long processing times, easy distortion of the material, which can be treated. Laser surface modification is the common process of a surface modification technique (Tikekar et al., 2007).

The laser re-melting surface has been utilised to enhance the surface of the workpiece; since it builds the life expectancy of the workpiece; and furthermore, it reduces the manufacturing cost (Janmanee and Muttamara, 2012), creating surface hardness (Frish et al., 1987).

The laser beam can be focused onto the metallic surface to achieve its treatment, for example, by re-dissolving, alloying and deposition (Tian et al., 2005). Cladding can be
utilised to enhance the wear and erosion resistance. The cladding can also be achieved by depositing material on top of the surface.

Re-melting can be used to re-melt the surface of the material, in order to achieve a controlled grain structure.

Thermal spraying is used as a surface modification; since it combines melting and quenching. The study by (Krishnan et al., 2006) showed that bonding strength limits the thermal spraying technique.

Many metal framework composites have been used for enhancing the wear resistance of the designing materials (Jian and Wang, 2005, Janaki Ram and Stucker, 2008).

2.17 Laser engineering net shaping

A study on the usage of laser engineering net-shaping (LENS)–type machine for the deposition of Ti6Al4V and W has been investigated (Gopagoni et al., 2011). Detail analyzation of Ti6Al4V and W composite on the interface, utilising high electron microscopy was carried out. This research focuses on the relationship between laser power, microstructure, microhardness and wear resistance of coated deposited Ti6Al4V and W, using the laser cladding process.

Another article discussed the use of a LENS-type machine for the deposition of nickel alloy (Ulutan and Ozel, 2011). The research demonstrated that laser power is the most essential element that controls the component quality. The goal of the study was to build up an overall term to predict deposition quality.
Another article was studied to examine the impact of process parameters on the deposition of Ti-6Al-4V (Kobryn et al., 2000). This research also involved the determining of laser power effects and scanning speed on the porosity of the laser deposited Ti6Al4V. The microstructure, macrostructure, and build height of samples were evaluated to determine the best operating parameters. The results showed that porosity decreased at higher laser powers and traverse speed. Build height decreased when the traverse rate was increased; but the effects of laser power on the deposition height were inconsistent.

Selective laser melting is utilised to reach the required temperature level. Two fundamental innovative advantages of selective laser melting are that it can deliver parts with high geometric complexity; and also its ability to utilise a wide range of metal powder. Laser engineering was discussed in section 2.9.

2.18 Metal matrix composites (MMC)

Titanium alloys are extensively used in the aviation manufactory, because of the good strength and better corrosion resistance (Box and Bisgaard, 1987). In any case, poor wear resistance helps to maintain their potential application. Ensuring titanium alloys with strengthened metal matrix composite (MMC) coatings is a superior answer for enhancing surface properties (Destefani, 1992). According to (Bejjani et al., 2011), titanium properties can be improved by reinforcing them with ceramics. Metal matrix composite reinforcement with titanium powder particles has been shown to be a better method to improve its wear resistance behaviour. (Çelik, 2013) showed that, by using a
metal matrix composite for property enhancement, the wear resistance will improve the strength of the material.

Laser metal Deposition (LMD) is a propelled strategy to create a particulate-strengthened surface layer on a metal substrate. The LMD is an appropriate procedure for Titanium alloys on the surface, to produce a dissolved pool at first glance (Bejjani et al., 2011). Tungsten powder particles are utilised as strengthened particles to create titanium matrix composites by laser metal deposition (Dai et al., 2015).

Different powder particles have been used to reinforce Ti6Al4V alloys by metal matrix composite. (Box, 1978) suggested that, by injecting ceramic particles into a laser melting pool by the laser metal deposition process improves the surface layer. “In the LMD process, the laser beam is used to melt the top layer of the metal substrate; while additive powder is injected into the melt pool simultaneously (Box, 1978).”

Metal matrix composite material should be chosen, based on the chemical compatibility and the ability to wet the reinforcement (Li et al., 2009). (Bejjani et al., 2011) suggested that a suitable reinforcement for Ti6Al4V alloys is tungsten powder (W), due to the comparable expansion coefficients of Ti6Al4V and W. It was also suggested that W/Ti6Al4V Metal Matrix Composite (MMC) demonstrated a lower cracking tendency. Silicon carbide particle (SiCp) and tungsten powder reinforcement have been successfully produced (Box and Bisgaard, 1987, Destefani, 1992, Bejjani et al., 2011, Li et al., 2009). Tungsten layer brings about enhancing of the joining at the Ti6Al4V + W interface; and it demonstrates the superb capacity of the load exchange (Liu et al., 2015). Tungsten powder has been proven to be stable thermodynamically in Ti6Al4V;
and it bonds well with the Matrix (Liu et al., 2015, Lloyd, 1991). The addition of tungsten powder particles improves the wear resistance of Titanium (Liu and Dupont, 2003).

2.19 Design of experiment

DOE was introduced as a statistical strategy utilised to explore the impacts of various variables at the same time. The essential guideline of DOE alludes to the procedure of arranging, planning and dissecting the investigation; so that substantial and target conclusions can be drawn successfully (Montgomery, 2008). The success of designed experiments depends on sound planning, in order to obtain the appropriate data that can be statistically analysed (Montgomery, 2008). According to (Bisgaard, 2000), there are two objectives for the design of experiments. These are to describe a phenomenon, or to uncover the cause and effect relationships that might exist within a population, sometimes with the aim of taking action on the current elements in the population.

The second objective is to use the current elements in the population/frame to deduce the knowledge whereby the elements of the process can be selected or adjusted to:

a) Improve future outputs of the universe, as desired by the next process/the supra-system; or to

b) Reduce the undesirable outputs of the process/system;

c) To select or adjust the elements in the system of a universe to improve the future elements of the universe.
2.20 Types of experimental design

Figure 2-11: Types of experimental designs
The design of an experiment formulates examples (response, factors, levels) of the following types of experimental design (Montgomery, 2008), which are shown in Figure 2.10.

### 2.20.1 One factor design

A one-factor design plan includes researching one factor. The goal of the one-component configuration is to discover whether the setting up of such an element at various levels has any noteworthy impact on the reaction. A one-factor design configuration can be valuable at the preparatory phases of an examination.

### 2.20.2 Factorial experiment

A factorial design includes multiples factors in the experiment (Box, 1978) called complex experiments; however, as the descriptor factorial demonstrates, the impacts of a few variables of a variation are contemplated and researched at the same time; with the treatment being all the combinations of the various factors under study. If the levels of the various factors are equal, then a factorial experiment means a trial with a number of factors. Consequently, the number is positive at each level that is greater than or equal to two. For example, a $2^3$ experiment means an experiment with three factors at two levels each; and a $3^2$ experiment means an experiment with two factors at three levels each.

Fractional factorial designs are subdivided into three fundamental sorts; and these are: factorial design with two-level fractional, Taguchi Orthogonal Arrays, and Plackett-
Burman Designs. A factorial design is the place at which not all the element level mixes are considered in the trial; a few communications can be examined, and certain cooperation among the components cannot be resolved. The Taguchi Orthogonal Arrays are utilised to gauge just the fundamental impacts, by leading a couple of exploratory runs. These designs are appropriate to two-level factorial investigations, as well as to multi-and blended-level elements. The Plackett-Burman Design is the place at which just a couple of particularly picked runs can be performed, so as to research just the fundamental impacts. They are exceptionally valuable as screening investigations, when there is a need to reduce the quantity of variables to be focused on.

2.20.3 Response surface method (RSM)

The Response surface method (RSM) is a collection of scientific and statistical methods given the attack of a polynomial mathematical statement of the exploratory data, which should describe the behaviour of the dataset with the objective of making statistical precision. It can be associated when a response or a course of action of responses to the leisure activity are influenced by a couple of factors. The objective is to enhance the levels of these factors to finish the best system performance (Bezerra et al., 2008).

Box and collaborators developed response surface methodology in the 50s (Gilmour, 2006) and (Bruns et al., 2006). (Bezerra et al., 2008) demonstrated a few phases in the utilization of RSM as a streamlining technique; and these are stated as follows:

(i) The determination of free factors of genuine effects on the system through screening considerations and the delimitation of the trial range, as shown by the objective of the review and the experience of the expert;
(ii) The choice of the exploratory plan and finishing the investigation, according to the chosen test matrix;
(iii) The mathematical–statistical treatment of the gained trial information through the attack of a polynomial limit;
(iv) The evaluation of the model's wellness;
(v) The confirmation of the need and probability of performing a dislodging in bearing to the ideal district; and
(vi) Getting the ideal qualities for each analyzed variable. The various stages in the response surface methodology are discussed as follows:

- **Screening of Variable**: Various variables might influence the reaction of the framework concentrated on; and it is essentially difficult to distinguish and control these little contributions from each other. Along these lines, it is important to choose those variables with significant impacts. Screening design ought to be done to make sense of the couple of exploratory factors and their affiliations; since they show huge effects. Full or incomplete two-level factorial design might be used for this objective essentially; since they are capable and conservative (Bezerra et al., 2008).

- **Decision of test plan**: The least-difficult model that might be utilised as a part of RSM depends on a direct function. For the uses of RSM, it is fundamental that the reactions acquired are tailored to the equation below:

\[
y = \beta_0 \sum_{i=1}^{k} \beta_i x_i + \varepsilon_i
\]

Where \([y]\) is the independent variable; and
\([x_i]\) is the dependent variable; and
$x_1, x_2, x_3, \ldots, x_k$ are the input factors,

and $\beta_0$ is the unknown parameter.

- **Mathematical statistical treatment of the data**: It is important to fit a numerical comparison to depict the conduct of the reaction as indicated by the levels of qualities.

- **Evaluation of the fitted model**: The mathematical model found in the wake of fitting the capacity to the information cannot attractively depict the test area considered. The more dependable approach to assess the nature of the fitted model was to use the analysis of variance (ANOVA). The focal thought of ANOVA was to contrast the variation due to the treatment (change in the blend of variable levels) with the variation, because of irregular blunders characteristic of the estimations of the produced reactions (Vieira and Hoffmann, 1989).

- **Determine the optimal conditions**: The surfaces created by straight models can be utilised to show the heading, in which the first plan must be dislodged, bearing in mind the end goal, which is to achieve the ideal conditions.

### 2.21 Summary

The literature was a review on Ti6Al4V alloy and Tungsten powder (W). The literature highlighted the technological backgrounds of all the techniques that have been used to deposit the Ti6Al4V and the tungsten related composites. The study also focused on the utilisation of the LMD techniques for depositing Ti6Al4V and W coatings. The accomplishment of LMD process, particularly in the surface modification of the primary alloy, and for the fixing of the costly components would depend on a point-by-point
comprehension of the relationship among the procedural parameters and the subsequent material properties.

The LMD procedure is fit for delivering substantial, complex and defect-free end-use parts with the desired surface properties. It is regarded as the most encouraging innovation for making complex end-use parts, and also practically evaluated materials. The procedure can be upgraded to accomplish better property control of practically evaluated Titanium based composite for end-use items.

Tungsten powder coatings have been deposited on Ti6Al4V alloy to improve the surface properties of the material, or the mixture of both powders in various proportions. The surface modification was utilised to improve the surface properties of titanium alloy (Ti6Al4V), in order to enhance the wear-resistance properties.

The literature also reviews the impact of procedural parameters on the subsequent microhardness, microstructure, and wear conduct of the laser metal deposited Ti6Al4V and W composite, and other related titanium based composites for improved mechanical properties.

This research has concentrated on the investigation of different procedural parameters that represent laser additive manufacturing, with specific reference to the LMD procedure of Ti6Al4V and W composite. The next chapter will focus on the experimental materials and methods.
CHAPTER 3

3 EXPERIMENTAL METHODS

3.1 Introduction

This chapter will focus on the experimental methods; and it will also present the materials that were used for the deposition, the experimental equipment and the set-up of the experiment. The experimental procedures will be discussed in this chapter. It will also show the positions of the workpieces, the tool design, the process parameters, the cladding process and the microstructures. The sample preparation will also be discussed in this chapter, as well as other characteristics, such as the wear test and wear calculation, x-ray diffraction (XRD), the Scanning electron microscope (SEM) with the electron dispersive spectroscopy (EDS), the Optical microscope (OM). Similarly, the statistical tool of the design expert used in the design of the experiment will be presented.

3.2 Equipment and experimental set-up

The laser metal deposition technology utilises an Ytterbium Laser System with a 3 kW maximum power and a co-axial nozzle fixed to the end effector of a Kuka robot. The laser equipment used is available at the National Laser Centre (NLC), the Council for Scientific and Industrial Research, CSIR, Pretoria, South Africa. The Laser system is linked with a Kuka robot that performs the deposition of powders onto the substrate via a three-way nozzle.
The robot arm controls the movement of the laser beam. The hoppers of the powder feeder are loaded with titanium alloy and tungsten powders. The chambers are loaded with argon gas that was utilised to prevent oxygen pollution on the cladded composite. The argon gas shields will be utilised to protect the cladding from oxidation amid the operations. The argon gas flow rate was kept constant. The laser power selection ranges between 250 W and 3 kW maximum; while the laser beam speed ranges between 1 mm/s and 10 mm/s. The 3-way nozzle is fixed to the robot arm, in order to control the laser focal length, as well as the beam size.

The beam diameter was kept at 4 mm, with a central separation of 12 mm between the substrate and the deposition zone. Figure 3.1 shows the experimental set-up.

![Figure 3-1: Experimental set-up](image)

The powder particles are infused into the dissolved pool through the three-way coaxial nozzle; while the laser beam melts the powders on the substrate.

The powder feeder storage containers are located away from the Kuka robotic arm; and they are connected with powder hoses. The gas flows through the gas cylinders located
behind the machine into nozzle’s manifold. The manifold routes the gases and powders to where they are delivered out of the tip of the nozzle.

The two powders were deposited on the Ti6Al4V substrate. The powders were then transported from the powder feeder through the hoses and to the nozzle end. It is also advantageous to use two different hoppers, rather than pre-mixed powder, due to the segregation of the powder, as a result of the different densities. Argon will be utilised to separate the gas, to direct the powder and the dissolved metal flow in the required direction. Figure 3.2 shows two distinct powder feeders. The two powders utilised were situated in various positions relative to each other. Each powder feeder was a part of the experiment set-up.

Figure 3-2: Feeding hoppers

3.3 The Laser deposition process

Before the actual laser process is performed, the set-up of the equipment must be accomplished. The set-up of the machine and the process parameters should be in place. These parameters includes scanning speed, laser power, transverse speed and powder feeder rotational speed. These factors are programmed into the control systems, which
control the movement of the robot arm. Figure 3.3 shows a schematic view of the experimental set-up.

![Schematic view of the experimental set-up](image)

**Figure 3-3: Schematic view of the experimental set-up**

The laser deposition process was defined by (Komvopoulos and Nagarathnam, 1990, Schneider, 1998) as a procedure that utilises the laser power to dissolve the powder or wire, in order to accomplish a good metallurgical bond that has less dilution of the material and the substrate, in order to maintain the properties of the deposited material.

The laser deposition process was explained in section 3.2, after the deposition of powder into the substrate; however, the cooling of the deposited composite and the substrate was allowed, in order to wire-brush the flux on the deposit. Thereafter, one must prepare the sample for further characterisation. The materials used for the experiment are described below. The photograph of the deposition process is shown in Figure A1 (Appendix A).
3.4 Materials and methodology

The Ti6Al4V alloy powder with a particle size distribution between 45 – 90 µm was used for this research; and it was delivered by Brodmann F.J. and Co., Louisiana L.L.C of Industrial Analytical (Pty) Ltd. The substrate used is Ti6Al4V alloy solid with a dimension of 70 x 70 x 5 mm. Table 3.1 shows the chemical composition of the substrate material. The substrate was cleaned and sandblasted, in order to remove the contaminants and to prepare the surface for good metallographic bonding.

**Table 3-1: Chemical composition of the substrate material**

<table>
<thead>
<tr>
<th>Element</th>
<th>Al</th>
<th>V</th>
<th>Fe</th>
<th>C</th>
<th>N₂</th>
<th>H₂</th>
<th>O₂</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>W%</td>
<td>6.42</td>
<td>3.91</td>
<td>0.19</td>
<td>0.008</td>
<td>0.006</td>
<td>0.004</td>
<td>0.155</td>
<td>Balance</td>
</tr>
</tbody>
</table>

The powders used for the experimental set-up were Ti6Al4V alloy and W. The chemical composition of Ti6Al4V alloy and W is shown in Table 3.2 and Table 3.3 respectively. The particle size of the Ti6Al4V powder is between 120 and 350 µm; and the mesh size of tungsten powder is between 45 – 90 µm. The powders were premixed in the ration of 5 wt.% of tungsten and 95 wt.% of Ti6Al4V.

**Table 3-2: Chemical composition of Ti6Al4V powder**

<table>
<thead>
<tr>
<th>Element</th>
<th>Al</th>
<th>V</th>
<th>Fe</th>
<th>C</th>
<th>N₂</th>
<th>H₂</th>
<th>O₂</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>W%</td>
<td>6.2</td>
<td>3.9</td>
<td>0.18</td>
<td>0.008</td>
<td>0.005</td>
<td>0.005</td>
<td>0.155</td>
<td>Balance</td>
</tr>
</tbody>
</table>

**Table 3-3: Chemical composition of tungsten powder**

<table>
<thead>
<tr>
<th>Element</th>
<th>Al</th>
<th>Mo</th>
<th>Fe</th>
<th>Cu</th>
<th>Cr</th>
<th>Ni</th>
<th>O₂</th>
<th>W</th>
</tr>
</thead>
<tbody>
<tr>
<td>W%</td>
<td>0.005</td>
<td>0.02</td>
<td>0.03</td>
<td>0.002</td>
<td>0.01</td>
<td>0.02</td>
<td>0.05</td>
<td>Balance</td>
</tr>
</tbody>
</table>
The design of the experimental (DOE) method will be used to build up a trial design to ascertain the important parameters and the interdependencies with an insignificant measure of information. The various process parameters of the experimental set-up are described below.

### 3.5 Process parameters

The process parameters were be determined by optimising the effect of variation in the parameters. Each parameter were be studied separately to get the best results.

Laser powers of 800 W, 900 W, 1000 W, 1100 W, 1200 W, and 1400 W were investigated. The substrates were exposed to the laser beam; and the values of the power meter readings were recorded or noted. The parameter’s values such as the scanning speed, laser power, overlapping percentage and the numbers of depositing tracks were input, using the controller; while the powder flow rates were input through the powder flow rate stand. The laser power and the scanning speed were changed; and the different parameters were kept constant.

A trial run was performed to determine the process window for the deposition of the powder and to check for pores; if there were any. The outcome of the trial run determined the way forward for the preliminary studies. Table 3.4 presents the processing parameters used for the trial run.
Table 3-4: Trial run process parameters

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>Laser Power (W)</th>
<th>Scan Speed (m/min)</th>
<th>Powder Feed Rate (%)</th>
<th>Gas Flow Rate (rpm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Ti6Al4V (%)</td>
<td>W (%)</td>
</tr>
<tr>
<td>N3.1</td>
<td>800</td>
<td>0.3</td>
<td>95</td>
<td>5</td>
</tr>
<tr>
<td>N3.2</td>
<td>900</td>
<td>0.3</td>
<td>95</td>
<td>5</td>
</tr>
<tr>
<td>N3.3</td>
<td>1000</td>
<td>0.3</td>
<td>95</td>
<td>5</td>
</tr>
<tr>
<td>N3.4</td>
<td>1200</td>
<td>0.3</td>
<td>95</td>
<td>5</td>
</tr>
<tr>
<td>N3.5</td>
<td>1600</td>
<td>0.3</td>
<td>95</td>
<td>5</td>
</tr>
</tbody>
</table>

The laser power was varied between 800 W and 1600 W; while the scanning speed and all the other parameters were kept constant.

3.6 Material characterisation techniques

All the participating samples were characterised by microstructure (optical and SEM), wear and corrosion.

3.6.1 Optical microscopy

The microstructural analyses were conducted by using the BX51M Olympus optical microscope to study the physical appearance of the deposit. The optical microscope used was available at the University of Johannesburg, in the metallurgy laboratory. The instrument was utilised for characterising the coatings for bonding, porosity, cracks, and dilution. The grain sizes of the deposited samples were measured, according to the standard test method for determining average grain size (ASTMF1372-93, 2012). The
chemical composition of the phases at the joint interface was determined by the scanning electron microscope. A photograph of the Olympus BX51M optical microscope is shown in the Appendix, Figure A2.

3.6.2 Scanning electron microscopy

The JOEL Scanning Electron Microscope (SEM) was used to take a high-magnification image, ranging from 25X to 1000000X and with an acceleration voltage between 1 – 30kv. The JOEL SEM equipment has been equipped with Electron Dispersion Spectrometry (EDS), in order to give quantitative and qualitative elemental analyses of a certain location on the image. The motivation behind the utilisation of SEM analysis is to provide high magnification and high amplification images of the samples, bearing in mind that the end goal is to explore the metallographic changes in the samples, as a consequence of the changing parameters. A picture of the JOEL Scanning electron microscope is shown in the Appendix, Figure A3.

The SEM is an advanced light-image microscope that can be used to analyse the microstructures of the coating, the porosity, the cracking and the elemental composition. The equipment is available at the Tshwane University of Technology, chemical engineering and metallographic laboratories under the guidance of the Lab technician. The x-ray was conducted, according to (ASTMF1372-93, 2012).

3.6.3 X-ray diffraction

Panalytical EMPYREAN Xpert PRO was used to study the phases in the samples, to characterise the powders and the coatings of the phase composition. The XRD uses a
Cu Kα monochromatic radiation at a power setting of 45 mA, 40kV. The equipment is also available at the Tshwane University of Technology, Pretoria Campus, and South Africa. The x-ray was conducted, according to (ASTMF1372-93, 2012). The photograph of the XRD is shown in Appendix A4.

3.7 Microhardness test

The unetched samples were indented on the laser deposited samples from the top to the substrate, by utilising the Metkon Vickers hardness machine analyser. This machine is available at the University of Johannesburg, Auckland Park Kingsway Campus, South Africa. The space load utilised was 500 g; and a dwelling time of 10 s was used. The spacing between the indentations is 100 μm; however, the hardness was conducted, according to the E384-11el ASTM (Standard). The formula or equation to calculate the Vickers hardness is as follows:

\[ VHN = 1854.4 \frac{P}{d^2} \]  

(3.1)

Where \( d_1 \) and \( d_2 \) represent the length of the vertical and horizontal indentations and \( P \) represents the load applied in Newtons. A photograph of the Olympus micro-hardness machine is shown in Appendix A8.

3.8 Wear testing (Tribological test)

Wear testing was conducted at the Tshwane University of Technology (TUT) to determine the wear-track surface profile. The wear resistance test was performed by using the UMT-2 CETR tribotester on a dry sliding wear tester. The ball is of tungsten carbide, with a diameter of 10 mm, under a load of 25 N and the reciprocating frequency of 20 Hz and for 1200 s. To achieve the load of 25 N, both the frictional force and the normal force were set to zero, with the sample’s dimensions of 20 mm X 10 mm X 10
mm. The wear surface is analysed by the SEM. According to (ASTMG133-05, 2010), all the sample were tested on a disk dry sliding wear tester. The trial experimental run will be discussed in the next section. This machine was made available at the Tshwane University of Technology, Pretoria Campus, South Africa. A photograph of the CERT tribotester is shown in Appendix A9. Figure 3-4 shows the schematic wear principle, or the operating technique between the sample and the wearing tool.

![Figure 3-4: Wear schematic diagram(Anawe et al., 2017)](image)

The schematic diagram in Figure 3-4 shows the wear process that was performed on the laser coated samples.

According to (Qu and Truhan, 2006) and (Sharma et al., 2013), equation 3.2 is used to calculate the wear volume loss.

\[
V_w = L \left[ r^2 \sin \left( \frac{W}{2r} \right) - \frac{W}{2} \left( r^2 - \frac{W^2}{4} \right) \right] + \frac{\pi}{3} \left[ 2r^3 - 2r^2 \left( r^2 - \frac{W^2}{4} \right)^{1/2} \right. \\
- \frac{W^2}{4} \left( r^2 - \frac{W^2}{4} \right)^{1/2} \right] (3.2)
\]

Where \( V_w \) = wear volume in \( \text{mm}^3 \)
L_s = stroke length  
R = pitch radius  
W = wear width

The wear depth is acquired from the wear results; and the wear scars were analysed from the SEM.

### 3.9 Corrosion test

The Potentiostat (GALVANOSTAT) equipment that uses GPES Manager Version 4.9 software was used to measure the corrosion on the surface of the coatings. A beaker tube was prepared with a solution; and the reference electrode (RE) tube was filled with Potassium Chloride. The RE and the counter electrode were inserted into the beaker tube to complete the circuit. The samples were inserted into the beaker tube and the reference electrode was also placed in the centre of the sample, in order to sense the potential difference of the coating. Before the sample can be placed inside the beaker tube, the conducting wire was placed on top of the coating; and this was covered by aluminium tape to hold the conductive wire. The connected wire was placed inside the mould; and it was stirred with epoxy resin. The resin takes between 2 – 4 hours to dry and form a good adhesive bonding.

Potentiodynamic polarisation curves were measured from -1.5 V to 2 V. the corrosion measurement of Ti6Al4V/W was evaluated at room temperature, using a 3.5 wt % NaCl solution. The samples were run in an Open Circuit Potential (OCP) for 3600 s. the corrosion measurement were prepared by using the standard practice, according to the (ASMHANDBOOK, 2005). A photograph of the Potentiostat (GALVANOSTAT) equipment is shown in Figure A10.
3.10 Sample preparation

The samples were cut to reveal the cross section by using a special grinding disc on the Mecatone T300 machine. After cutting, the Lecco PR 25 machine was used to prepare the samples for mounting using polyfast resin. A photograph of the Mecatone T300 is shown in Figure A6; while Figure A7 depicts the Lecco PrR 25 Mounting Machine. After mounting, the variable-speed grinder-polisher was used to get rid of any impurities on the surface. Two different grinding discs were used; where the first grinding was set to 300 revolutions per minute; and the second grinding (smooth grinding) was set at 150 revolutions per minute (rpm). The grinding was performed for 7 min. After grinding, the sample surfaces were washed and rinsed with clean water and then dried off by hand dryer. Smooth and fine grinding was performed on the samples using the MD-Largo disc with Dia-Pro Allergro suspension.

Polishing was performed after all the grinding had been completed. The polishing disc was used to smooth the surface and to remove unwanted scratches on the deposited sample; and the machine was set at 150 revolutions per minute (rpm). OP-s chemical suspension was used to smoothen the surface of the sample, until a mirror-like surface was achieved. The polishing was performed for 8 minutes. All the specimens were carefully grinded, polished and etched, according to (Standard, 2012) E3-11 ASTM Standard.

The polished metallic samples were etched, according to the Kroll’s reagent. For the preparation for the microstructure, the samples were etched with 100 ml of water and 2 ml of hydrofluoric acid and 3 ml of nitric acid. The next section will present the results of the preliminary studies.
3.11 Design expert software

The design of the experiment’s (DOE) methodology was used to build up a trial preparation, to recognise the important parameters and the interdependencies with an insignificant measure of information. The DOE is an important tool in engineering. The experimental results were evaluated, based on the statistical methods; and the impacts of the procedural parameters on the trial results can be calculated. The Response Surface Methodology (RSM) was implemented to analyse the experimental parameters. The design of the experimental tool was used to establish the relationship between the parameters and microhardness.

The Design Expert 10 software was utilised to run the design of the experiment.

Table 3-5: Design summary for experiment

<table>
<thead>
<tr>
<th>Number</th>
<th>Laser Power (W)</th>
<th>Scanning Speed (m/min)</th>
<th>Powder Flow Rate (rpm)</th>
<th>Gas Flow Rate (l/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Ti6Al4V (%)</td>
<td>W (%)</td>
<td>Ti6Al4V (%)</td>
<td>W (%)</td>
</tr>
<tr>
<td>1</td>
<td>800</td>
<td>0.5</td>
<td>95%</td>
<td>5%</td>
</tr>
<tr>
<td>2</td>
<td>800</td>
<td>0.7</td>
<td>95%</td>
<td>5%</td>
</tr>
<tr>
<td>3</td>
<td>800</td>
<td>0.9</td>
<td>95%</td>
<td>5%</td>
</tr>
<tr>
<td>4</td>
<td>900</td>
<td>0.5</td>
<td>95%</td>
<td>5%</td>
</tr>
</tbody>
</table>
The trial was utilised to lessen the quantity of components by removing the slightest impeding element. Central Composite Design (CCD) was used to plan the screening trial. The process parameters used were the gas flow rate, the powder flow rate, the scanning speed and the laser power. The central composite design is suitable for this experiment. The input parameters are shown in Table 3-7.

A summary of the experimental layout design is presented in Table 3.6; it displays the input parameters, which are the gas flow rate, the powder flow rate, the scanning speed and the laser power. Table 3.7 shows the input parametric factors.
Table 3-6: Input parametrics for the design of the experiment

<table>
<thead>
<tr>
<th>Factors</th>
<th>Name</th>
<th>Minimum</th>
<th>Maximum</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Scanning Speed</td>
<td>0.3</td>
<td>0.9</td>
</tr>
<tr>
<td>B</td>
<td>Laser Power</td>
<td>800</td>
<td>1400</td>
</tr>
<tr>
<td>C</td>
<td>Gas Flow Rate</td>
<td>1.5</td>
<td>3</td>
</tr>
<tr>
<td>D</td>
<td>Powder Flow Rate</td>
<td>2</td>
<td>3</td>
</tr>
</tbody>
</table>

Table 3.8 shows the experimental runs created by the design expert 10 software. Thirty experimental runs were conducted.

Table 3-7: Design Layout of the experiment

<table>
<thead>
<tr>
<th>Run Order</th>
<th>Standard Order</th>
<th>Block</th>
<th>Scanning Speed (m/min)</th>
<th>Laser Power (kW)</th>
<th>Powder Flow Rate (rpm)</th>
<th>Gas Flow Rate (l/min)</th>
<th>Space Type</th>
<th>Leverage</th>
<th>Hardness (Hv)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>27</td>
<td>1</td>
<td>0.6</td>
<td>1100</td>
<td>2.75</td>
<td>2.25</td>
<td>Axial</td>
<td>0.1667</td>
<td>0.5833</td>
</tr>
<tr>
<td>2</td>
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The power calculations were performed by using a continuous response type. Table 3.9 shows the power at 5% alpha level to detect the noise level in the model.
Table 3-8: Power at 5% alpha level to detect signal/ noise ratio

<table>
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<tr>
<th>Term</th>
<th>Standard Error</th>
<th>VIF</th>
<th>Ri-Squared</th>
<th>2 Standard Deviation</th>
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<tr>
<td>B</td>
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<tr>
<td>C</td>
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<td>1</td>
<td>0</td>
<td>99.50%</td>
</tr>
<tr>
<td>D</td>
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<td>1</td>
<td>0</td>
<td>99.50%</td>
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<td>AB</td>
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<td>0</td>
<td>96.20%</td>
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<tr>
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<tr>
<td>AD</td>
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<td>0</td>
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<tr>
<td>BC</td>
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<td>1</td>
<td>0</td>
<td>96.20%</td>
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<tr>
<td>BD</td>
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<td>1</td>
<td>0</td>
<td>96.20%</td>
</tr>
<tr>
<td>CD</td>
<td>0.25</td>
<td>1</td>
<td>0</td>
<td>96.20%</td>
</tr>
<tr>
<td>A²</td>
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<td>1.05</td>
<td>0.0476</td>
<td>99.90%</td>
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<tr>
<td>B²</td>
<td>0.19</td>
<td>1.05</td>
<td>0.0476</td>
<td>99.90%</td>
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<tr>
<td>C²</td>
<td>0.19</td>
<td>1.05</td>
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<tr>
<td>D²</td>
<td>0.19</td>
<td>1.05</td>
<td>0.0476</td>
<td>99.90%</td>
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</tbody>
</table>

The Variance Inflation Factor (VIF) for this model is 1.0, which is perfect. The VIF over 10 is the reason for concern; since it shows that the coefficients are inadequately assessed. The Ri-squared for this model is 0.0; and is the better Ri-squared. The high the Ri-squared points to a poor model, because of the terms that are related to each other.
3.12 Summary

This chapter has presented the experimental materials, the experimental procedure and the parameter settings for the deposition of Ti6Al4V+W composites, utilising the LMD process. The trial run and the preliminary studies were conducted in this chapter. This chapter also looked into the material characterisation techniques, the mechanical testing, the tribological testing and the corrosion testing. The Design of Expert software was utilised to deliver the experimental results and to perform the CCD experiments. The results of the enhanced coatings are reported and discussed in the next chapter.
CHAPTER 4

4 RESULTS AND DISCUSSION

4.1 Introduction

The main objective of this thesis was to deposit Ti6Al4V+W composite using the LMD process. The preliminary study was conducted in the previous chapter. The preliminary study was performed to establish the process window for depositing the Ti6Al4V+W composite using the LMD process. The outcomes and discussion report on the characterization of the deposited samples using an optical microscope, and the scanning electrode microscope with energy dispersion spectrometry, in order to establish the microstructure and the chemical compositions, and also to characterise the powders and coatings, in order to determine the phase composition, as well as to measure the hardness of the coating, the corrosion resistance and the friction wear, respectively. The results of the trial run and preliminary studies were also presented.

4.2 Laser metal deposition of Ti6Al4V+W coatings

4.2.1 Preliminary studies

The results reported in this section are obtained with the optimised parameters. The preliminary studies were coated with 95 wt. % of Ti6Al4V alloy and with 5 wt. % of W using two different hoppers. The laser power ranged from 800W to 1400W. The preliminary study was conducted for the repairing of a turbine blade. The preliminary studies are discussed in the next section.
4.2.2 Microstructural characterisation

The microstructure presented in the Figures 4-1 (a), (b), (c), (d), (e), (f) and (g) show the Ti6Al4V+W coatings of the deposited samples with various laser powers and various scanning speeds at the cladding zone. A Table of laser power is shown in the Appendix, Table B3 to B7.

(a) Grain boundary

(b) Dendrites
Widmanstettan

\( \beta \)-phase

\( \alpha \)-phase

Basket Weavelike

Heat Affected Zone

Substrate Zone
Figures 4-1: (a) High magnification view of dendrite with 1200W laser power and 0.7 m/min scanning speed; (b) SEM image showing dendrite with laser power of 900W and scanning speed of 0.5 m/min; (c) high magnification view of the primary dendrites with laser power of 1200W and the scanning speed of 0.5 m/min; (d) SEM image showing titanium needles with laser power of 1000W and the scanning speed of 0.5 m/min; (e) interface between the heat affected zone and the substrate with laser power of 1000W and the scanning speed of 0.5 m/min; (f) Cross-section view of composite coating with laser power of 1200W and scanning speed of 0.7 m/min; (g) Microstructure of cladding zone with laser power of 1200W and the scanning speed of 0.3 m/min;
Figure 4-1(a) shows the microstructure of Ti6Al4V+W composite deposited at a laser power of 1200 W, and a scanning speed of 0.7 m/min. The Titanium strips with hexagonal particles are shown with thick-grain boundaries. The microstructure is characterised by Widmanstettan structures. Figure 4-1 (b) shows the microstructure of sample N5.2 deposited at the laser power of 900 W. The primary dendrites are visible at the interface between the cladding and the heat affected zone. Figure 4-1(c) displays the microstructure taken at a higher magnification which shows the dendrite structures. The dendrite arms were growing bigger when the laser power was increased. This is characterised by alpha and beta phases. According to (Baik, 2006), it is likely to recognise the \( \beta \)-phase as light phase and \( \alpha \)-phases as dark phases. Figure 4-1 (d) shows the microstructure of the cladding zone deposited at a laser power of 1000 W, and the scanning speed of 0.5 m/min. The microstructure is characterised basket weave-like (Widmanstettan) and carved with needle-like shaped structures. The structures are formed from the titanium needles. This process is related to the process temperature that occurs during the laser material's processing (Gu et al., 2012).

Figure 4-1(e) shows the interface of the heat affected zone in the substrate of sample N5.3 deposited at the laser power of 1000 W and the scanning speed of 0.5 m/min. The microstructure shows a strong bond between the cladding and the substrate. Figure 4-1(f) displays the morphology of the Ti6Al4V+W coatings; and it is possible to identify the coatings, the white spots and the Ti6Al4V substrate. The white spots that appear on the deposited material are evidence of unmelted tungsten powder. These white spots are concentrated at the bottom of the coating because of their high density; and they are characterised as having little or no porosity. Figure 4-1 (g) shows the microstructure of sample N3.4 deposited at the laser power of 1200 W and the scanning speed of 0.3 m/min. The porosity and the cracks can be observed on the cladding zone. This could
happen as a result of the high laser power, as well as the densely packed effect of the unmelted W along the crack propagation. The crack could also be due to the influence of residual stresses with the coating vicinity (Folorunsho, 2015)

4.2.3 Phase composition

Figure 4.2 displays the phase composition of a typical sample of Titanium alloy and Tungsten composite produced at different laser power and 0.7 m/min scanning speed. The XRD was utilised to analyse the phase compositions of the laser deposited Ti6Al4V+W composite.

Figure 4-2: XRD Diffraction patterns of the Ti6Al4V+W coatings
Figure 4-2 indicates the XRD patterns of the samples produced by the LMD process are similar in trend. The major Ti6Al4V peaks are observed at 2 theta 35°, 38° and 40°. The composite coating of the alpha (α) and beta (β) phase of W, Ti, and TiO2 phases were produced by using various laser power. The observation that the Ti has strong peaks has been reported before by (Çelik, 2013). The varying laser power of the LMD process brought a huge change in the microstructure of the coatings and XRD patterns. The particles of Ti + W were the highest at the coatings deposited using a laser power of 1000 W; this is agreement with the obtained hardness results. The diffraction peaks for both Ti6Al4V and W were observed at 38 and 40-degree peak positions. The peak identification for tungsten powder compared with the Ti6Al4V peak was low, due to the small percentage of tungsten powder. The TiO2 peaks are detected in the XRD-diffraction patterns; and these show the excess oxygen environment. The titanium oxide (TiO2) peaks were observed at 2 theta = 35° and 78° peak position. The deposition of the powder for this study was conducted in a closed chamber in an Argon-controlled environment. The TiO2 represents lack of argon during the cooling phase before the temperature drops below the titanium oxidation threshold. A similar observation has already been reported by (Tlotleng, 2015)

4.2.4 Elemental analyses

Figure 4.3 shows the micrograph and EDS analyses of laser deposited Ti6Al4V and W coating, using various laser powers and 0.7 m/min scanning speed.
(a)
Figure 4-3: (a) EDS analyses and SEM micrograph at a laser power of 800 W, (b) EDS analyses and SEM micrograph at a laser power of 800 W and (c), was taken at laser power of 1400 W.
The EDS results showed that the highest peak was identified on the Titanium; and it has
the highest percentage (wt. %) of 78.58. Vanadium, Aluminium and Tungsten were also
detected on the coating. Figure 4-3(a) shows the columnar grain observed at the clad
region of the composite. Figure 4-3(b) shows cross-section SEM micrograph of the
coatings deposited at a laser power of 1400W. It can also be observed that the W were
equally distributed in the composite coating. W penetrate the composite coating due to
its highest density. Figure 4-3(c) shows no porosity or cracks formation on the coating
deposited at a laser power of 1400W. EDS results show that Ti modified layer is mainly
composed of the elements Ti, AL, W and V. The white specks are represents the
tungsten.

4.2.5 Microhardness profiling

The microhardness of the laser deposited Ti6Al4V+W coatings is taken from the top of
the deposit to the bottom of the coating (substrate). Figure 4.4 shows the hardness
profiling of the sample deposited at a laser power of 1200 W and a scanning speed of
0.7m/min. The indentations were captured throughout the deposited surface and down
to the substrate.
Figure 4-4: Microhardness values for Ti6Al4V + W at the various laser powers and the different scanning speeds of 0.7m/min.

The indentation was conducted in three different areas, which are top part (clad), Heat affected zone (HAZ) and substrate. The top part is normally the hardest; the heat affected zone relates to the heat input and dilution; and it also predicts the joint of the coating to the base material. The hardness was initially high at the top, less high at the heat affected zone and low on the substrate. The hardness value at the top of the surface or clad is twice as high as that of the substrate hardness. In other words, the hardness decreases from the top to the fusion zone and to the heat affected zone; and it equally decreases down to the substrate. This observation was also reported by Zhou et al. (2012) and is attributed to the heat input dissipation within the clad zone.
4.3 Analyses of the statistical results of the deposited composites

The analyses of the results were obtained by means of the Design expert 10 software. The investigation of the results consists of three factors. These were the laser power, the scanning speed and the hardness values. The responses are analysed in the following order: the sum of the squares, the model summary statistics, the lack-of-fit tests, and the p-value. Table 4.1 shows the sequential model sum of the squares. The sum of the square’s deviations from the mean of each model is analysed. The sum of the squares is first calculated by means of a linear model, then the quadratic model, the cubic model, the residual and finally, the total. The mean square is the sum of the squares divided by the degree of freedom.

The insignificant lack of fit of the p–value is greater than 0.10. The highest f-value is 4.08 and the smallest is 0.33. The smallest p-value of 0.0196 shows the significance of the variation. The highest f-value of 4.08 and the smallest p-value of 0.0196 fall within the Quadratic vs 2FI. The p-value is less than 0.10; and the model is not aliased.

Table 4-1: Sum of squares sequential model

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<th>Sum of Squares</th>
<th>df</th>
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<th>F Value</th>
<th>p-value</th>
<th>Prob &gt; F</th>
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Table 4.2 presents the model summarised statistics. The model explains the maximum R-Squared value and the Predicted R-Squared value.

4.3.1 Numerical results

The numerical results are analysed and presented in the next section. The results were analysed statistically, numerically and graphically. The analysis of variance (ANOVA) is presented in Table 4.3; and it shows the response surface quadratic model.
### Table 4-3: Analysis of variance table

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</table>

The F-value of the model is 8.49, which indicates that the model is significant; and there are 0.04% chances that an F-value might happen because of the low noise. The values of P>F under 0.0500 show that the model terms from the experiment are significant in the case of A. P-values greater than 0.1000 show that the model is not significant. Table 4.4 shows the R-squared value and the adjusted R-squared value.
Table 4-4: R-squared and adjusted R-squared values

<p>| | |</p>
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<td>Mean</td>
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<tr>
<td>C.V. %</td>
<td>2.47</td>
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<td>-2 Log Likelihood</td>
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</tr>
<tr>
<td>R-Squared</td>
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<tr>
<td>Adj. R-Squared</td>
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<tr>
<td>Pred. R-Squared</td>
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<tr>
<td>Adeq. Precision</td>
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<tr>
<td>BIC</td>
<td>236.89</td>
</tr>
<tr>
<td>AICc</td>
<td>232.89</td>
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</table>

The Pred. R-Squared of 0.0491 is not anywhere near the Adj. R-Squared value of 0.4364; as one may typically expect; i.e. the distinction is greater than 0.3. This might show a substantial large-block effect. Adeq. Precision measures the signal-to-noise ratio of more than 4, which is proper. The Adeq. Precision of 12.998 demonstrates a satisfactory signal. Table 4.5 shows the coefficient estimate of the model.
<table>
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<th>Factor</th>
<th>Coefficient Estimate</th>
<th>df</th>
<th>Standard Error</th>
<th>95% CI Low</th>
<th>95% CI High</th>
<th>VIF</th>
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<td>433.52</td>
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<td>9.39</td>
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<tr>
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<td>2.19</td>
<td>-8.62</td>
<td>0.39</td>
<td>1</td>
</tr>
<tr>
<td>$A^2$</td>
<td>8.24</td>
<td>1</td>
<td>4.13</td>
<td>12.36</td>
<td>1</td>
<td></td>
</tr>
</tbody>
</table>

The equation for the coded factors is shown below:

Harness Value = $428.31 + 4.88A - 4.1B + 8.24A^2$

The coded factors equation can be utilised to make expectations regarding the reaction for particular levels of each factor. The formulae for the coded factors are helpful for distinguishing the relative effect of the factor by matching the factor coefficients.

The actual factors’ formulae are shown in the equation below:

Hardness value = $529.47 - 0.18\text{ Laser Power} - 13.71\text{ Scanning Speed} + 9.16\text{ Laser Power}^2$

The actual factors’ formulae can be utilized to make expectations regarding the reaction of the various levels of each factor.
4.3.2 Statistical analysis

The typical likelihood plot demonstrates whether the residuals follow a normal distribution, in which the data point will follow the straight line. The data points are linear, which shows no complexities in the data obtained. The normal plot of the residuals confirms that the residual is normally distributed; while the externally studentized residual shows that the data are not outside the red line. Figure 4.5 demonstrates the normal plot of the residuals between the normal probability and the externally studentized residuals.

![Normal Plot of Residuals](image)

Figure 4-5: Normal plot of residuals

The normal probability plot demonstrates whether the residuals follow a normal distribution; in which case, the focuses would take after a straight line. The plot shows that the residual is normally distributed. The externally studentized residuals check whether a run is predictable when compared with alternate runs. Figure 4.6 shows the residuals versus the increasingly
predicted response values. The plots test the hypothesis of steady variance across the prediction range. The plot is randomly scattered across the graph.

**Figure 4-6:** Plots of residuals versus predicted values

Figure 4.7 shows the externally studentized residuals versus the run number. It looks for prowling factors that may have impacted the results during the analysis. The plot demonstrates random scattering. The patterns demonstrate a period associated with variable prowling in the background. The residuals plot displays that the residual is normally distributed; since there are no data outside the red line. If there are data points outside the red line, then there is a cause for concern. The design shows a hardness value of between 413 HV and 457 HV.
Figure 4.8 shows the graph of the predicted response values versus the actual response values. From the predicted plot graph, it can be observed that the data are scattered all over the line. The predicted plot displays that the predicted values are normally distributed.
4.3.3 Graph model

Design expects the software to provide various graphs in two-dimensional and three-dimensional views. The graphs explain the behaviour of the process. Figure 4.9 shows the contour graph. The contour shows the relationship between the laser power, the hardness values and the scan speed. The power is from the range between 800 W and 1400 W; while the scan speed is between 0.5 m/min and 0.9 m/min.
Figure 4-9: Contour graph of the scanning speed between 0.3m/min and 0.9m/min and the laser power between 800W and 1400W

The 3D surface plot is shown in Figure 4.10, together with the surface plot of the scanning speed and the laser power. The graph reveals that the higher laser power – from 800 W to 1400 W – causes the hardness values to decrease to a point; and they later increase as the laser power continues to increase. The rate of cooling at a low laser power is fast, there is no full melt of deposition and there is no room of gas to disappear or dissolve whereas at higher laser power, the melt pool dissolved and more gases are formed resulting in slow cooling rate. In addition, it can also be observed that as the scanning speed increases, the hardness of the Ti6Al4V+W composites decreases. The increase in the laser power leads to an increase in the hardness values. A similar observation was reported by (Folorunsho,
The relation between the parameters have greater influence on the hardness values.

The relation between laser power, scan speed, powder rate and the gas flow rate

The cube plot of the hardness is shown in Figure 4.11. Three actual responses were used, which are: the laser power, the scanning speed and the powder flow; while the actual factor is the gas flow. The laser-power values between 800 kW and 1400 kW were plotted on the base of the cube. The hardness value increased from 413HV to 457HV at the laser power between 800 kW and 1400 kW and the scanning speed between 0.3 m/min and 0.9 m/min. The relationship between power and speed has an influence on the hardness of the material. The gas-flow rate shows a minimal influence on the hardness of the material.

**Figure 4-10**: The surface plot of hardness values deposited between laser power of 800kW and 1400kW, with the scanning speed from between 0.3 m/min to 0.9 m/min
4.3.4 Optimisation of the experimental results

The optimisation is achieved by selecting the preferred goal to each factor; and the likely goals are: minimise, within range maximise, target, and none. The target was chosen for the hardness values, with the lower value of 413, and the higher value of 457. The values were chosen between the heat-affected zone and the cladding zone. The constraints parameters are shown in Table 4.14.

Figure 4-11: Cube plot of the hardness value deposited at the laser power of between 800kW and a scanning speed of between 0.3 m/min and 0.9 m/min.
### Table 4-6: Constraints for optimised parameters

<table>
<thead>
<tr>
<th>Name</th>
<th>Goal</th>
<th>Lower Limit</th>
<th>Upper Limit</th>
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<tr>
<td>A: Laser Power</td>
<td>maximize</td>
<td>800</td>
<td>1400</td>
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<tr>
<td>B: Scanning Speed</td>
<td>minimize</td>
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<td>0.5</td>
</tr>
<tr>
<td>C: Powder Flow</td>
<td>is equal to 2.75</td>
<td>2.5</td>
<td>3</td>
</tr>
<tr>
<td>D: Gas Flow</td>
<td>is in range</td>
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<td>3</td>
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<tr>
<td>Response</td>
<td>Cpk</td>
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<td>457</td>
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</table>

### Table 4-7: Optimised processing parameters

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<th>Scanning Speed (m/min)</th>
<th>Powder Flow (rpm)</th>
<th>Gas Flow (l/min)</th>
<th>Response</th>
<th>Cpk(Response)</th>
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<td>0.708</td>
</tr>
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</table>
The Numerical Optimisation Ramps are shown in the graphical analysis, as shown in Figure 4.12. The red dot on each line of the ramp explains the factor setting; while the blue dot explains the optimal expected response values. The following parameters were analysed on the ramps: the gas-flow rate, the laser power, the hardness response and the scanning speed.

The 3D surface plots are shown in Figure 4.13; and they displace the desirability of laser powers between 800 W and 1400W; scanning speed of between 0.3m/min and 0.9m/min. It also shows the constant power-flow rate and the powder-flow rate.
**Figure 4-13:** Desirability surface plot for different laser powers from 800W to 1400W and the scanning speeds between 0.3 to 0.9m/min of the powder flow rate and gas flow rate.

The contour of the desirability is shown in Figure 4.14 – with the varying laser power and varying scan speed. The contour plot is a projection of the 3D surface plot that gives the colour and a 3D surface. The desirability of the scanning speed is shown at 0.6 m/min; while the laser power is between 800 kW and 1100 kW. The blue and green colours drawn on the yellow block base show the desirability surface.
Figure 4-14: Contour plot for desirability at scanning speed of 0.6m/min and varying laser power between 800kW and 1100kW.

4.4 Powder characterisation

The Ti6Al4V and W powders were analysed with XRD, in order to reveal the phases in the powders. Figures 4-15 (a) and (b) show the phases of Ti6Al4V alloy and W powders. Figure 4-15 (a) identified Ti6Al4V as a major element and with the highest percentage peak. The phases present are: Ti6Al4V, AlV and βTi. The two major peaks are observed at 39° and 40°. Some peaks were found at 2-theta angles of 35° and 40°. Figure 4-15 (b) shows that tungsten powder (W) has the highest peak is at 40 %. Other major peaks of W were observed at 58° and 73°. Some other peaks were also detected of tungsten
beta phase at 86°. Figures 4-15(a) and (b) demonstrate the particle size analysis of Ti6Al4V alloy and W powders. The particle size of tungsten powder is between 120 µm and 350 µm; while the particle size of the Ti6Al4V powder is between 45 µm and 90 µm, respectively, which is slightly smaller than the particle size of W powders. The next section will present the second trial run results.
Figure 4-15: XRD powder analyses (a) Ti6Al4V and (b) W

4.5 Laser metal deposited Ti6Al4V+W coating in the trial run

4.5.1 Powder morphology

Figures 4-16 and 4-17 display the morphology of the tungsten powder and the Ti6Al4V powder. The particle size analysis of the Ti6Al4V powder and the W powder is shown in Figures 4-16b and 4-17b.
It was observed that most of the sample had no porosities, sample 1000W, 1100W and 1200W showed good microstructures with the scan speed of 0.05m/s.
The morphologies of the Ti6Al4V alloy powder were observed to be spherical in shape; and the smooth surface was covered with tiny dust particles. The morphologies of the powder also show a few pores. Figure 4.16 shows the SEM image taken at the higher magnification resolution, to show the morphologies of the Ti6Al4V. The morphologies of the Tungsten were also observed to be spherical in shape (block). Tungsten powder is heavier in mass than Titanium alloy powder. Figure 4-17 shows the morphology of Tungsten powder.

**Figure 4-17:** powder morphology (a) SEM image of W; (b) Particle size analysis of W
The morphology of Ti6Al4V alloy is spherical and equiaxed in nature; and most of the peaks are Ti6Al4V. The morphology of W is crushed and non-spherical; and most of the peaks are W. Observations during the preliminary work revealed that, the joint interface between the deposited area and the substrate material showed no crack and no porosity. The purpose behind this is that the tungsten powder’s thermal expansion coefficient is near that of Ti6Al4V alloy.

4.5.2 First trial runs with Ti6Al4V+W

The point of the trial run was to build up the process window for the deposition of defect-free samples. The outcomes from these trial runs have led to the preliminary studies. Distinctive mixes of procedure parameters were attempted; and microstructural examinations were performed to see whether the parameters had revealed any porosity. The morphology of the titanium alloy powder and the tungsten powder is shown in Figures 4-18 and 4-19, individually.

The processing parameters used in the first trial runs are presented in Table 3.4; the laser power was varied from 800 W to 1400 W; The Scanning speed was kept constant at 0.3m/min. Five deposits were made on the substrate; and the deposited samples were labelled N3.1 to N3.5. Prior to the cladding procedure, all the substrates were cleaned by sandblasted machine to remove any undesirable material; and to prepare the samples for metallurgical bonding.

After the deposition process, all the laser deposited samples were cleaned with a metal brush, to remove the unmelted powder and the fluxes created on them. In the microscope observation, all the samples were observed and characterised with
porosities. Sample N3.3 deposited at a laser power of 1000 W and scan speed of 0.3 m/min shows few porosities. Some unmelted W was also observed in the vicinity of the titanium alloy. The results of the preliminary studies are displayed in the next section.

![Micrograph of sample N3.2 with porosity at laser power 800 W and the scanning speed of 0.3m/min.](image1)

**Figure 4-18:** Micrograph of sample N3.2 with porosity at laser power 800 W and the scanning speed of 0.3m/min.

![Micrograph showing samples with little porosity at laser power 1000 W and the scanning speed of 0.3m/min.](image2)

**Figure 4-19:** Micrograph showing samples with little porosity at laser power 1000 W and the scanning speed of 0.3m/min.
The results from the preliminary studies have been accepted or submitted for publication in the conference proceedings. The process window was established. The preliminary studies were coated with 95 wt. % of Ti6Al4V alloy and with 5 wt. % of W, using two different hoppers. Figure 3-1 demonstrates the experimental set-up of the robot arm. The robot arm is connected to the powder-house nozzle.

Preliminary studies of the Ti6Al4V+W layers deposited on the Ti6Al4V substrate were performed. The microstructure of Ti6Al4V strengthening by coated tungsten through LMD was also investigated; and it was found that the laser source specifically impacts the microstructure, as well as the hardness properties of the coating. The impact of laser power on the cladding of Ti6Al4V+W composite was also investigated; and it was found that when increased, the average microhardness diminished. The impact of laser power on the wear resistance of laser deposition Ti6Al4V+W composites was additionally explored. It was found that the laser metal deposition process is appropriate for delivering wear resistance coating on Ti6Al4V+W and that the laser power has an influence on the performance of the wear resistance coatings.

Table 4-8 displays the process parameters for the preliminary studies of Ti6Al4V+W coating. The laser power was varied from 800 W to 1400 W; while all the other parameters were kept constant. The second trial run is presented in the next section.
Table 4-8 First preliminary processing parameters

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>Laser Power</th>
<th>Scanning Speed</th>
<th>Feed Rate (unit)</th>
<th>Gas Flow Rate (rpm)</th>
</tr>
</thead>
<tbody>
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<td></td>
<td>W</td>
<td>m/min</td>
<td>Ti6Al4V</td>
<td>W</td>
</tr>
<tr>
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<td>0.5</td>
<td>95%</td>
<td>5%</td>
</tr>
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<td>N5.2</td>
<td>900</td>
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<td>95%</td>
<td>5%</td>
</tr>
<tr>
<td>N5.3</td>
<td>1000</td>
<td>0.5</td>
<td>95%</td>
<td>5%</td>
</tr>
<tr>
<td>N5.4</td>
<td>1200</td>
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<td>95%</td>
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<tr>
<td>N5.5</td>
<td>1400</td>
<td>0.5</td>
<td>95%</td>
<td>5%</td>
</tr>
</tbody>
</table>

4.5.3 First trial runs with Ti6Al4V+W

The second trial run was coated with 95 wt.% of Ti6Al4V alloy with 5 wt.% of W using two different hoppers. The laser metal deposition process set-up was reported in Figure 3 chapter 3.

4.5.4 Microstructural characterisations

The microstructural image of laser deposited Ti6Al4V+W coatings is captured under the SEM. Figures 4-20 and 4-21 show the optical microstructure and SEM of the laser deposited Ti6Al4V+W composite deposited at a laser power of 1000 W and a scanning speed of 0.3 m/min. The study was conducted on the micrographs of samples, labelled as N3.3. The microstructures reveal three regions. These are the clad zone, the heat-affected zone (HAZ) and the substrate. The coatings were etched with Kroll’s reagent to reveal the grain boundaries and other phases. From the Figure 4-20, the coating indicates some porosity. The porosity can be caused by inadequate laser power, or inadequate interaction time, as well as the entrapped gas during solidification.
(Mahamood et al., 2013). The bonding between the deposited zone and the substrate seems to be good; and it shows a well attached fusion bond.

![Figure 4-20: Ti6Al4V/W laser power of 1000W and 0.3 m/min scanning speed](image1)

**Figure 4-20**: Ti6Al4V/W laser power of 1000W and 0.3 m/min scanning speed

![Figure 4-21: A high magnification Ti6Al4V/W at 1000W laser power and 0.3 m/min scanning speed](image2)

**Figure 4-21**: A high magnification Ti6Al4V/W at 1000W laser power and 0.3 m/min scanning speed

It can be seen from the Figure 4-21 that the dominating microstructure was titanium strips with the hexagonal particles that occur at clad zone with fine grains.
4.5.5 Microhardness profiling

The unetched samples were indented, using the Vickers hardness machine Metkon microhardness tester. This equipment is available at Kingsway Campus, University of Johannesburg, South Africa. The indentation load used was 500 g and a dwelling time of 10 s. The spacing applied was 100 μm between the indentations. Moreover, the hardness test was done, according to E384-11el ASTM standard. The deposited samples were indented from the clad zone to the substrate. Figure 4-18 indicates the profile of the 1000W laser power and 0.3 m/min scanning speed. The microhardness test was performed on the samples N3.1 to N3.5. The hardness value of sample N3.1 shows the highest value of 711 HV towards the top of the deposited composite; while sample N3.3 shows the lowest hardness value. The high hardness value is attributed to the addition of 5 wt% of tungsten powder.

![Diagram showing microhardness profile](image)

**Figure 4-22:** Microhardness Value for laser power 1000W and 0.3m/min scanning speed.
Figure 4-22 indicates the microhardness values of the coating and the indent of the substrate respectively at 1000W and 0.3 m/min scanning speed. It may be seen that the highest hardness value is twice the hardness of the substrate.

4.6 Results of optimised processing parameters

4.6.1 Introduction

This section reports on the laser deposited composites in the preliminary studies using the LMD process. The microstructural characterizations of the deposited coatings are discussed in this section, as well as the elemental analysis of the wear results, hardness analysis, the corrosion test results, and the phase identification with XRD analysis. These results are similarly discussed in this section. The characterisations were obtained on the optimised parameters from the preliminary trial run and the preliminary studies. The deposition of Ti6Al4V + W substrate was done successfully, the best parameters was observed at a laser power of 1000W, and the scanning speed of 0.5m/min gave the best results.

4.6.2 Microstructural Analysis

The microstructures of the selected samples are shown in Figures 4.23 (a) to (d). The samples are produced with the direct laser metal process at 0.5 m/min scanning speed, while varying the laser power between 800 W and 1100 W. The selected samples show good bonding with the substrate; and they show little porosity. Figures 4.24 (f) to (g) deposited at 1200 W laser power and 0.7 m/min scanning speed show images of the microstructures.
Figure 4-23: Micrographic images of Ti6Al4V/W deposited at 0.5 m/min scanning speed and various laser powers: (a) 1000 W; (b) 1200 W; (c) 900 W; and (d) 800 W.

The micrographic images shown in Figure 4.23 shows the cladding produced by deposition at different laser powers. Figure 4.23 (a-b) shows the good bonding with little porosity. A high laser power makes the melt pool; and during the reinforcing process, the vast majority of the powders are largely melted. A high laser power results in a slow cooling rate; and this improves the ductility of the composite. The slow scanning speed prolongs the laser power interaction time; and the deposited powder was melted. Figure
4.23 (c-d) show coatings with porosities; and it also reveals the dilution of the material. Figure 4.23 (c-d) shows the lack of fusion; and the absence of fusion porosity can likewise occur when the powder flow rate is higher than that which the accessible power can melt (Kobryn et al., 2000). There is no full melt of deposition during low power, cooling rate is fast and that results in absence of fusion.

Figure 4-24: (f-g) Micrographic images of Ti6Al4V/W deposited at 0.5m/min scanning speed and 1200W laser power
The micrographic images displayed in Figures 4.24 (f-g) show a laser power of 1200 W, showing that a high laser power results in a slow cooling rate. These coatings show some small porosity, which is caused at a high scanning speed. The high scanning speed brings about a reduced laser material contact; the time it takes to contact with the participating materials was too short to create any effective melting of the powder (Mahamood et al., 2013). A too high scanning speed or insufficient laser power can result in the lack of fusion. And as such, improper melting would exist between the powder and the substrate.
Basket-Weave Pattern

Widmanstatten

W particles bonding
**Figure 4-25** (a) shows the microstructure of the heat affected zone and the substrate at the laser power of 1000W with a scanning speed of 0.5 m/min; (b) shows the microstructure forming a basket-weave pattern at a laser power of 1000W and a scanning speed of 0.5m/min; (c) shows the heat affected zone and the cladding; (d) shows the high magnification images of Ti6Al4V + W deposited at 0.5m/min scanning speed and a laser power of 1000 W.

Figure 4-25(a) shows the microstructure of the heat affected zone and the substrate bonding. Figure 4-25 (b) shows the Ti6Al4V needles forming a basket-weave pattern. Figure 4-25(c) shows the heat affected zone and microstructure of the deposited coating. At the joining boundary line, the coating was characterised by a thin strip that was completely covered with W particles. The bonding between the clad layer and the substrate is shown in Figure 4-25(c); where it is possible to identify where the coating and substrate merge. Figure 4-25 (d) shows a high-resolution image at a laser power of 800 W. Ti6Al4V and W coatings produced at laser power of 1000 W were achieved with little dilution. It may be concluded that the coating at the clad layer was strong, with less dilution. Figure 4.1(f) shows that the W coatings at the top surface of the coating reinforce the material.

### 4.6.3 Wear Analyses

The morphologies of worn surface of the Ti6Al4V+W composites are shown in Figure 4.26 (a-b). All the samples were pressed under a load of 25 N and the wear resistance of Ti6Al4V + W coating was evaluated by a sliding distance of up to 2 mm. Figure 4.26 shows that, as scanning speed decreased, the material time interaction is increased and this result in improved wear volume.
Figure 4.26 (a) shows the micrograph of the wear track of Ti6Al4V + W composite with the debris resulting from the detached materials. It also shows the wear width measured from the SEM and the wear depth from the wear analysis. Figure 4.26 (b) shows the wear track obtained during the wear procedure. A tungsten Carbide ball interacts with the surface of the substrate leading the rubbing action of the two surfaces. During rubbing action, strong adhesion occurs; and this creates a high temperature, which makes debris to increase the wear action.

The effect of the load was observed during the Ball-on-Flat sliding wear test. Tungsten wear occurred under expanding load, and a deep groove was formed. All the coatings' claddings occurred at various laser powers of 900 W, 1000 W and 1400 W and a 0.5 m/min scanning speed. The coating performed at the laser power of 900 W shows the increase in the wear resistance relative to original substrate.

Figures 4.27, 28 and 29 show the high magnification images of the wear track. The figures clearly shows wear tracks of the coatings deposited by laser metal deposition at
different laser power. All cladded products produced better wear resistance behaviour than of the substrate; this can be ascribed to the distribution of W particle

The increase in hardness values with laser process can be related to the spreading of W particles in the coating, which increase the hardness in the coatings. The increase in the wear rate of the coating material with the increase of laser power from 1000 W to 1400 W could be linked to the process parameters of the coating deposited at 1400 W. Figure 4-31 shows the coating deposited at the laser power of 1000 W with higher wear rate than that deposited at the laser power of 900 W. Further increases in the laser power to 1400 W prompted the wear rate to increase more than in the lower laser power of 900 W. The contact of two surfaces resulted in strong adhesion; and this causes the coefficient of friction (COF) to increase. The increased in COF can be attributed to the attaching effect of W and the resulting ploughing action. A similar observation has been reported by (Ming et al., 1998). The hardness of the entire Ti6Al4V + W deposited sample were improved under expanding load compare to original substrate.
Figure 4-30: Coefficient of friction at a load of 25 N for Ti6Al4V + W deposited at varying laser powers and a constant scanning speed of 0.5m/min: (a) 800W, (b) 900W, (c) 1000W, (d) 1200W and (e) 1400W.

The coefficients of friction of the composite’s coatings deposited at different laser powers between 800W and 1400 W are compared in Figure 4.30. The wear volumes or wear losses were calculated using the formula devised by (Qu and Truhan, 2006) and (Sharma et al., 2013). Figure 4.31 shows the wear volume or the wear loss in a column chart against the laser powers. The coefficient of friction of Ti6Al4V + W is between 0.029 and 0.073 for all the samples.
The deposited coatings at laser powers from 800 W to 1400 W show lower wear volumes compared to the substrate material and the deposited coatings at the laser power of 1000W. From the plot, laser-deposited sample with a laser power of 1000 W has the lowest wear loss of 0.061 mm$^3$. Table 4-9 presents the wear volume or loss of the coatings deposited at the different laser powers between 800 W and 1400 W.
Table 4-9: the wear volume for the different laser power values

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<th>Coated Samples</th>
<th>Wear Volume (mm³)</th>
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</tr>
<tr>
<td>Laser power 900W and 0.5m/min scanning speed</td>
<td>0.176</td>
</tr>
<tr>
<td>Laser power 1000W and 0.5m/min scanning speed</td>
<td>0.061</td>
</tr>
<tr>
<td>Laser power 1200W and 0.5m/min scanning speed</td>
<td>0.076</td>
</tr>
<tr>
<td>Laser power 1400W and 0.5m/min scanning speed</td>
<td>0.127</td>
</tr>
<tr>
<td>Substrate material</td>
<td>0.314</td>
</tr>
</tbody>
</table>

Figure 4.32 shows the percentages of wear volume or loss of the composites with varying laser powers of 800 W, 900 W, 1000 W, 1200 W and 1400 W and a constant scanning speed of 0.5 m/min. It may be observed that the laser power of 1000 W shows the lowest percentage of the wear volume of 10%. The laser power of 900 W shows the high wear volume percentage of 20%. However, the length and the width of the wear track of the laser power of 900 W is more than that of the remaining samples, with the exception of the substrate. The substrate shows the highest wear volume or loss; and this indicates a poor wear resistance, it can also be observed from Figure 4.32 that the highest wear volume percentage of 35% is obtained for the substrate. The length and width of the wear track plays an important role in determining the wear volume. It can be observed that the presence of Tungsten powder has increased the wear resistance of the main alloy.
Figure 4-32: Percentages of wear volume for the laser power of 800W to 1400W at a constant scanning speed of 0.5m/min

4.6.4 Microhardness profiling

The microhardness of the coatings deposited at different laser powers between 800W and 1400W, and varying the scanning speeds were performed. Figure 4.33 shows the microhardness values of the deposited coatings at the various scanning speeds and a constant laser power of 1000 W. Figure 4.33 shows different scanning speeds; and it may be deduced that the hardness increases with a decrease in the overall scanning speed.
Figure 4-33: The microhardness values of Ti6Al4V+W at 1000W laser power and varying scanning speeds from 0.5m/min to 0.9m/min.

Figure 4.33 shows that the microhardness values increased as the scanning speed decreases. The sample deposited with a scanning speed of 0.5 m/min shows the highest microhardness values of 761 HV. The scanning speed of 0.9m/min shows the highest microhardness value of 666 HV. The highest hardness values for the laser deposited Ti6Al4V+W composite at varying scanning speeds were 761 Hv and 666 Hv respectively. The highest microhardness value can be attributed to the slow scanning speed of 0.5 m/min exhibited, and also because of the time it takes for the laser powder to interact with the material was enough to melt the powder. The clad zone associated with the top area of the deposition and the heat affected zone, known as the interface.
of the coating and the substrate show improved hardness value. The clad zone for different scanning speeds behaved similarly at the top with a high hardness value, which was twice times of the substrate. The increase in the hardness is the results of W powder distribution. The hardness of Ti6Al4V + W was improved compared to the substrate. This behaviour was also observed by (Çelik, 2013). The hardness decreases slightly in the heat affect zone for the scanning speed; however, the interface shows a good bonding with the substrate. A noteable increase in the hardness value was seen in all the laser-deposited Ti6Al4V+W samples relative to the hardness of the primary material (Ti6Al4V) refer to the bar chart in Figure 4.31.

This increase is related to the distribution of W particles in the matrices. Lastly, the clad zone and the HAZ showed higher hardness values than the substrate. The scanning speed rate have a greater influence on the measured hardness values of the Ti6Al4V + W composites. Figure 34 indicates the different scanning speed. The average scanning speed shows higher hardness. The hardness of 0.5m/min can be related to the positioning of powder during deposition and is shows good bonding with minor porosity. The hardness of 0.3m/min was the third hardest coating. This coating contains much porosity on the clad surface. A similar observation was made with the 0.9m/min and 0.7m/min coatings. However, the hardness values obtained in this present study were more than the hardness values reported by (Klimpel et al., 2011); although a 300 g force was reported in their research work.

With the addition of 5 wt % of W, the hardness properties were considerably improved.
4.6.5 Corrosion behaviour

Corrosion tests were conducted on the laser deposited Ti6Al4V+W coatings; and the samples were run in Open Circuit Potential (OCP) for 3600 s. The corrosion measurement of Ti6Al4V+W was evaluated at the room temperature, using a 3.5 wt% NaCl solution. Figures 4.35 sample(a)-sample(e) show the open-circuit potential in 3.5 wt% NaCl solutions for Ti6Al4V+W coatings deposited at different laser powers of 800 W, 900 W, 1000 W, 1200 W and 1400 W. Figures 4.30 sample(a)-sample(e) show the open-circuit potential of coating deposited from the laser powers between 800W to 1400W and a constant scanning speed of 0.5m/min.

The open-circuit potential for the coated sample deposited at 800W laser power and 0.5m/min scanning speed is demonstrated in Figure 4.35 sample(a); and this shows a sharp rise in the corrosion rate from -0.086 V to -0.0353 V at 1060 seconds. It also shows a decrease in corrosion at -0.0361, until the end at 3600 seconds. The coated sample shows...
an increase in the corrosion rate and the film growth on the surface of the electrolyte. The specimen of Figure 4.35 sample (b) deposited at 900W laser power and 0.5m/min scanning speed shows an increase of the corrosion potential from -0.136 V to -0.1217 V at 736 seconds; and it shows a sharp decrease from -0.149 V to -0.242 V at 804 seconds.

The corrosion resistance rises again for a longer time from -0.242 V to 0.099 V at 2843 seconds; and it falls again from -0.11 V to -0.19 V at 3087 seconds. The corrosion rate rises again at -0.189 to the end. Figure 4.35 sample (c) deposited at 1000W laser speed and 0.5m/min scanning speed, the open-circuit potential is between -0.134 to 0.108 V until the end of 3600 seconds. The sample coated at laser power 1200 W at 0.5m/min scanning speed showed a rise in corrosion rate from -0.101 V to -0.0846; but it maintained a decline from -0.1 to -0.113 V; while it showed sharp decline at -0.1139 until the end at 3600 seconds.

Figure 4.35 sample (e) shows a sharp increase in the corrosion rate from -0.4096 V to 0.1595 at 1339 seconds; but it falls again from -0.16 V to -0.50 V at 1546 seconds. The fall of the cathodic reading can be related to the oxide formed on the top of the surface. Figure 4.35 presents the variations in the open-circuit potential for the coated samples deposited at the varying laser powers and the scanning speed of 0.5m/min.
Figure 4-35: The variation in OCP as a function of time for Ti6Al4V/W

Specimen (c) coated at 1000W laser power and 0.5m/min scanning speed shows the highest corrosion resistance. Samples (a) and (b) show a better corrosion resistance, when compared with the other remaining samples. Sample (c) shows an oxidation resistance. W has a good resistance to corrosion in the nitric acid solution. This correlates well with the phenomenon that W addition increases the corrosion resistance. The study by (Yi et al., 2013) revealed that the W addition increases the corrosion resistance; and it also increases its content on the surface.
A polarisation test was conducted on the coated samples produced by different laser powers and a scanning speed of 0.5m/min. Figure 4.36 shows the polarisation in 3.5 wt% NaCl solutions for Ti6Al4V+W coatings deposited at different laser powers.

Linear potentiodynamic polarisation measurements were carried out to evaluate the corrosion behaviour of Ti6Al4V+W coatings. Attention was focused on studying the effect of laser power on the alloys passivity, pitting and re-passivation behaviour of the alloys in 3.5 wt. % NaCl solution. Figure 4.36 gives the graphical representations of their response to polarisation. The polarisation curves obtained displayed similar trends for the alloys, with a small active and pseudo-passive transition in almost all the samples. However, well established regions of
active, passivation (0 – 0.5V and ~ 2.03E-06 A/cm²) and transpassive are seen with 900W laser power. The curves showed that there is only a slight difference on the corrosion potentials (E_{corr}) (~ -0.4V) with varying laser power. Though, a slight deviation towards negative potentials (~ -0.6V) was observed with the laser power of 1200W. The highest current densities (i_{corr}) were observed with the laser power of 1000W and 1200W (polarization curves are far to the right). The oscillation potential shown by both 1000W and 1200W show similar kinetic behaviour. The oscillation formation is generally due to the formation of the TiO₂ layer. This observation was reported by (Tlotleng, 2015). Whereas, the laser power of 800W gave the lowest current density and thus, less susceptible to salt attack. The laser power of 900W and 1400W shown to some extent to be parallel in their current densities, with the latter showing higher anodic dissolution rate. In conclusion, these coatings display that the are kinetically and thermodynamically stable in 3.5 wt% NaCl solutions. Overall, an increase in laser power shifts the Ti₆Al₄V+W coating polarisation curves to higher current densities. In divergence, laser power increase from 1200W to 1400W moves the curve back to the left (lower i_{corr}).

**4.6.6 Corrosion behaviour of Ti₆Al₄V/W composites**

Ti₆Al₄V alloy has excellent resistance to corrosion in many environments. The influence of W particles on the corrosion and microstructural behaviour of Ti₆Al₄V in 3.5 wt% NaCl solutions was studied. Figure 4.37 presents the surface morphology of the Ti₆Al₄V+W. Figure 4.37 reveals the presence of tungsten with white cores; and with the addition of W content, the number of white cores increases.
Figure 4-37: Surface morphology of the Ti6Al4V+W in 3.5 wt% NaCl solutions

As observed in Figure 4-37, there is an indication of pitting corrosion on the surface of the Ti6Al4V. The pitting corrosion indicates that the passive film in the region is weak. The addition of W content provided a shield against the pitting corrosions. The white core and the rim phase are W; while the black core-grey rim structures are Ti6Al4V. The white core increases with the addition of W content; or black cores decrease with the addition of white cores. The Ti6Al4V particles, which are not melted during the process, appear as black cores. Figure 4-37 reveals that the width of the rim phase increases with the addition of W. Both the inner rim phase and the white core rim are corroded with the increase of W contents. The Ti6Al4V+W show passivity because of the corrosion rate increases with the addition of W.
4.7 SUMMARY

The microhardness profiles, the corrosion resistance, the XRD analysis, the wear of the microstructure of the laser deposited Ti6Al4V+W samples were analysed and presented in this chapter. The microstructures presenting in this chapter show the coating that is rich in Ti6Al4V + W content on the surface. The selected samples show good bonding with the substrate; and they show little porosity. Since little pores were recognised within the coating, we can conclude that the coatings is essential for the envisioned application.

The hardness values were conducted from the top of the coating until at end of the substrate. The hardness was high at the top coating and fell slightly in the middle of the coating until at the end of the substrate. The deposition of Ti6Al4V + W substrate was done successfully, the best parameters was observed at a laser power of 1000W, and the scanning speed of 0.5m/min gave the best results. The hardness showed that the increased in laser power results in the increase of hardness; and the decrease in scanning speed results in the increased hardness values.

The polarisation current density, the open circuit potential (OPC), and the corrosion rate of the Ti6Al4V + W coating prepared using a 3.5 wt% NaCl solution were analysed. The corrosion resistance test reveal that titanium alloy shows excellent corrosion resistance due to its passivation. Ti6Al4V +W coating is thermodynamically steady in NaCl solution.

The SEM analysis on the wear track and the wear volume of the Ti6Al4V/W composite were presented. Wear resistance of Ti6Al4V was enhanced by adding W. The laser power of 1000W and the scanning speed of 0.5m/min show lowest wear volume loss. The XRD was used to analyse the phase compositions of Ti6Al4V and W coatings.
Design Expect software was used to perform the experiment analysis on the four factors. The factors are: the hardness values, the laser power, the gas flow rate, the scanning speed and the powder flow rate. Four factors were used in the hardness-value responses of the deposited Ti6Al4V/W composite.
CHAPTER 5

5 CONCLUSION AND RECOMMENDATIONS

5.1 INTRODUCTION

The objective of the study was to achieve a coating that is suitable to repair components produced from titanium and its alloys operating in a high-temperature environment, such as a turbine blade. The objective of the research was to produce a Ti6Al4V/W coating by using the Laser metal deposition process. Design expert software was used to achieve the relationship between the processing parameters and the hardness value.

The literature review, as presented in Chapter Two discussed in detail, as was the laser metal deposition process and the importance of process parameters in achieving defect-free clads. It also revealed the significant effect of the laser power and the effect of the scanning speed, as they influence the material properties.

The experimental procedures utilised in this research were presented in Chapter Three. The analyses were done on the coated samples. The following assessments were conducted on the coated sample: wear resistance, corrosion, mechanical testing, the scanning electron microscopy (SEM) with energy dispersion spectrometry (EDS), x-Ray diffraction, and the metallurgical characterisation.

The preliminary results conducted in this research build upon the relationship between the processing parameters and the responses. The next section presents the recommendations and the conclusions.
5.2 CONCLUSION

The research has achieved its aim or main goal by successfully producing a suitable coating using the LMD process. LMD method has been used in the application for the repair of turbine blades and other parts, such as the aero-engine. The coatings were obtained using 900 W laser power and 0.5 m/min scanning speed, with a powder ratio of 95wt.% Ti6Al4V and 5 wt.% W. This study has used the process parameters for the laser metal deposition process. These process parameters were: the laser power, the powder rate scanning speed and the gas flow rate; and they were used as the primary factors for the design of the experiment. Design Expect software was used to design the experimental data, and also to develop the optimised model.

The relationship between the processing parameters and the hardness value was conducted. The hardness values were chosen as the responses for the design of the experiment.

This work can be applicable for the repairing of turbine blades and other jet engine parts. The Ti6Al4V+W coating was produced by using the laser metal deposition process to achieve the suitable coating to repair the gas turbine blade. Titanium blades are exposed to high temperatures, as well as high corrosion stress, which causes degradation of the material. Tungsten powder was chosen because of its high melting point and its wear resistance. The Laser process was suitable for depositing the titanium powder, together with the tungsten powder (W).

The optimised processing parameters were conducted by using the laser metal depositing process at 1000W laser power and 0.5 m/min scanning speed. This shows the good metallurgical bonding without any porosity. The SEM results show the microstructure of the
Ti6Al4V+W with the formation of a basket-weave structure and the Widmanstatten structure. The hardness value of Ti6Al4V alloys was improved by adding the tungsten powder. The hardness value of the top part of Ti6Al4V/W cladding sample is 761 HV, which is twice that of the substrate. This high hardness value can be attributed to the slow scanning speed. The improved hardness value is the result of the addition of the tungsten powder.

The analysis of variance (ANOVA) revealed that the model is significant; and it found that the model relates to the experimental results. The model revealed that the laser power has a significant effect on the hardness of the deposited material.

The XRD patterns for both coatings showed that the Ti6Al4V was the predominant material in the coating; since the Ti6Al4V was observed at the highest peak.

Tungsten powder (W) is responsible for the improved wear resistance of Ti6Al4V/W composite. Tungsten is known for its resistance to wear. The SEM wear photos revealed that the coated samples of Ti6Al4V+W showed the lowest wear loss. The addition of tungsten powder improved the wear resistance of titanium alloy grade 5.

The corrosion rate results of Ti6Al4V+W concluded that a good corrosion rate was achieved when compared with uncoated Ti6Al4V. The addition of W improved the surface integrity of the Ti6Al4V. The microstructures of Ti6Al4V+W displayed the white core-rim structure and the black core-rim structure. With the addition of W, the inner rim increases.
5.3 FUTURE WORK

From the outcomes of this study, and the conclusions that can be drawn from those outcomes, several future works are suggested.

➢ Further testing should include a more detailed characterisation of the samples. The use of transmission electron microscope (TEM) imaging could be employed to analyse the lamellar structures.

➢ The design of the experiment should be extended to include the responses of the wear values.

➢ The design expert software (statistical methods) has predicted optimised processing parameters for the design of experiments. It is significantly important to run test utilising the anticipated level setting, in order to determine whether the quality is indeed enhanced in comparison with that of the past experimental procedure.

➢ Further work should include the characterisation of fracture toughness and fatigue on the Ti6Al4V+W.
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APPENDIX A

Appendix A

Appendix A1: Photographs of equipment used.

A2 Optical Microscope
A3: JOEL Scanning Electron Microscope

A4: Panalytical EMPYREAN Xpert pro XRD
A6: Mecatome T300 Cutting machine

A7: Mounting machine Lecco PR 25

A8: Microhardness tester
A9: CERT Wear tester

A10: Potentiostat
APPENDIX B

Appendix B: Tables

Table B.1  First trial experimental matrix

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>Laser Power (W)</th>
<th>Scanning Speed (m/min)</th>
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Appendix C: Graph
OCP deposited at 800W laser speed and 0.5m/min scanning speed

OCP deposited at 900W laser speed and 0.5m/min scanning speed (b)
OCP deposited at 1000W laser speed and 0.5m/min scanning speed

OCP deposited at 1200W laser speed and 0.5m/min scanning speed
OCP deposited at 1400W laser speed and 0.5m/min scanning speed (e)