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HIGH-TEMPERATURE EROSION-WEAR BEHAVIOUR OF STAINLESS STEEL COMPOSITES

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A dissertation submitted to the Faculty of Engineering and the Built Environment, University of Johannesburg in partial fulfilment of the requirements for the degree Masters of Technology in Chemical Engineering Technology

Supervisor: Dr P.A. Olubambi
Co-supervisor: Dr B.A. Obadele

Johannesburg, 2018
DECLARATION

“I, R.M Mafafo, hereby declare that the dissertation submitted for the Masters of Technology, at University of Johannesburg, is my own original work and has not previously been submitted to any other institution of higher education. I further declare that all sources cited or quoted are indicated and acknowledged by means of a comprehensive list of references”.

________________________________________

Signature
DEDICATION

This study is dedicated to Almighty God, my beloved husband, Malose Victor Kgafela and our Children Kgopotso, Theto and Tshireletso Kgafela.
ACKNOWLEDGEMENTS

I would like to kindly thank all people who made this Research possible by supporting me.

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I would like to thank my parents, Mr. and Mrs. Mafafo, and my siblings, for your continuous prayers, the love and moral support. My sincere acknowledgement goes to my Husband, MALOSE VICTOR KGAFELA who supported and believed in me all the time and my children, KGOPOTSO, THETO AND TSHIRELETSO.

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ABSTRACT

The erosion-wear behaviour of spark plasma sintered austenitic stainless steels matrix composites was investigated under high temperature conditions. As-received powders were characterized using SEM-EDS and XRD. Powders were mixed at varying compositions of austenitic steels/titanium nitride and the admixed powders were characterized using SEM-EDS and XRD to check if the samples have been homogeneously mixed. Size determination was conducted on the feedstock powders by a Particle Size Distribution method. The homogeneously mixed powders were then sintered at 1100 °C with a heating rate of a 100 °C/min, applied vacuum pressure of 50 MPa and holding time of 10 minutes. The study involved metallagraphic preparation of sintered solid specimen, microstructural characterization of consolidated austenitic stainless steel under SEM-EDS and phase identification analysis under XRD. Erosion-wear behaviour of the composites was conducted at a constant velocity of 18 m/s at temperatures from room temperature, 400 °C, up to 600 °C. The material used as the erodent was abrasive alumina particles with particle size of 40 µm. The incident angle of 90° was employed throughout. After SPS, it was observed that the relative density of spark plasma sintered samples decreased with increasing TiN content. The erosion wear rate indicates that there is a proportional relationship between the decreasing percentage of hardness and the erosion rate. The results further indicate that material wastage increases with increasing temperature. XRD reveal that there is a new phase (B2: AlNi) formed after erosion. Microstructures indicate that the erosion damage occurs mainly by plastic deformation and brittle fracture. Macrographs of the eroded specimens show three layers in which the first (central) layer represents high material loss, the
second layer where lesser amount of material wastage is experienced and the third (outer) layer where the least amount of material was removed.
# TABLE OF CONTENTS

DECLARATION......................................................................................................................... i

DEDICATION......................................................................................................................... ii

ACKNOWLEDGEMENTS ......................................................................................................... iii

ABSTRACT ............................................................................................................................ iv

CHAPTER ONE ....................................................................................................................... 1

1.0. INTRODUCTION........................................................................................................ 1

1.1. BACKGROUND .................................................................................................... 1

1.2. RESEARCH PROBLEM ....................................................................................... 3

1.3. AIM AND OBJECTIVES ...................................................................................... 4

1.4. JUSTIFICATION FOR THE RESEARCH .......................................................... 4

1.5. SCOPE OF WORK .............................................................................................. 5

1.6. STRUCTURE OF THE DISSERTATION ............................................................ 6

CHAPTER TWO .................................................................................................................... 7

2.0. LITERATURE REVIEW ......................................................................................... 7

2.1. INTRODUCTION.................................................................................................. 7

2.2. Stainless steels .................................................................................................. 7

2.3. Classification of stainless steel: ........................................................................ 9

2.3.1. Austenitic stainless steels ............................................................................ 9

2.3.2. Martensitic stainless steels ........................................................................ 9
2.7.3. Wear ................................................................. 19

2.8. Wear mechanism ......................................................... 20

2.8.1. Adhesive wear ........................................................ 21

2.8.2. Abrasive wear ....................................................... 21

2.8.3. Corrosive wear ...................................................... 22

2.8.4. Fatigue wear ........................................................ 23

2.8.5. Erosion wear ........................................................ 24

a) Cavitation erosion ....................................................... 24

b) Slurry erosion .......................................................... 25

c) Solid particle erosion .................................................. 25

2.9. Parameters affecting erosion wear ......................... 26

2.9.1. Concentration of the particle ............................. 27

2.9.2. Particle size and shape ....................................... 27

2.9.3. Velocity of flow ............................................... 27

2.9.4. Impact angle ...................................................... 28

2.9.5. Hardness ............................................................ 28

2.10. Erosion wear tester ............................................... 28

2.10.1. Gas-blast type erosion tester ........................ 29

2.10.2. Centrifugal accelerator type erosion tester ....... 30

2.11. Erosive wear behaviour of austenitic stainless steel matrix composites ...... 31
CHAPTER THREE ................................................................................................ ........ 34

3.0. EXPERIMENTAL PROCEDURE ................................................................................. 34

3.1. INTRODUCTION ........................................................................................................... 34

3.2. POWDERS AND MATERIALS ..................................................................................... 34

3.3. POWDER CHARACTERIZATION ............................................................................... 36

3.3.1. Microstructure and phase analysis ...................................................................... 36

3.4. SPARK PLASMA SINTERING OF POWDERS .......................................................... 36

3.5. CHARACTERIZATION OF SPARK PLASMA SINTERED SPECIMEN ................. 38

3.5.1. SEM-EDS analysis .............................................................................................. 38

3.5.2. XRD analysis ..................................................................................................... 39

3.6. MECHANICAL TESTS ................................................................................................. 40

3.6.1. Micro-hardness .................................................................................................... 40

3.6.2. Erosion Wear Studies ......................................................................................... 41

CHAPTER FOUR ................................................................................................ ........ 45

4. RESULTS AND DISCUSSION ....................................................................................... 45

4.0. INTRODUCTION ......................................................................................................... 45

4.1. CHARACTERIZATION OF AS-RECEIVED POWDERS ........................................... 45

4.1.1. SEM/EDS of starting powders ............................................................................ 45

4.1.2. XRD analysis of feedstock powders .................................................................. 49

4.2. CHARACTERIZATION OF ADMIXED POWDERS ................................................. 50
REFERENCES.................................................................................................................................................. 84

LIST OF FIGURES

Figure 2.1 Fe-Cr phase diagram (Banda, 2013)............................................................................................ 8
Figure 2.2 Illustration of friction .................................................................................................................. 18
Figure 2.3 Wear processes on a tribosystem ................................................................................................. 20
Figure 2.4 Flow chart of various wear mechanism ......................................................................................... 20
Figure 2.5 An illustration of adhesive wear mechanism (Kato and Adachi., 2001). ................................. 21
Figure 2.6 Schematic two-body and three-body abrasive wear mechanism (Devaraju., 2015). ............ 22
Figure 2.7 Corrosive wear mechanism (Williams, 2010). ....................................................................... 23
Figure 2.8 Schematic illustration of fatigue wear mechanism (Buchholz., 2011) .................................. 23
Figure 2.9 Cause and effect diagram of erosion wear (More et.al., 2017) ................................................. 26
Figure 2.10 Schematic diagram of gas-blast type of erosion tester (Deng et al., 2009). ...................... 30
Figure 2.11 Schematic diagram of centrifugal acceleration type erosion tester (Deng et al., 2009) .... 30
Figure 3.1 Microtrac Particle Size Distribution (PSD) analyzer (Dada, 2015) ............................................. 35
Figure 3.2 Turbula shaker T2F (Obadele., 2014) ....................................................................................... 36
Figure 3.3 (a) Schematic diagram of Spark Plasma Sintering (SPS) apparatus (Sulima et al., 2015) (b) SPS image. ................................................................................................................................. 38
Figure 3.4 TESCAN Vega 3 SEM-EDS ....................................................................................................... 39
Figure 3.5 Rigaku Ultima IV X-ray diffractometer .................................................................................... 40
Figure 3.6 InnovaTest Falcon 500 microhardness tester .......................................................................... 41
Figure 3.7 Ultrasonic cleaner. ................................................................. 42
Figure 3.8 Schematic diagram of the experimental set up. ................. 44
Figure 3.9 Experimental setup (a) erosion tester equipped with a furnace (b) interior of the erosion tester (c) laser temperature reader. ........................................ 44
Figure 4.1 Scanning electron microscopy morphology of as-received 304 ASS powder particles with EDS analysis.............................. 47
Figure 4.2 SEM morphology of as-received TiN powder magnifications at (a) 1000x and (b) EDS analysis...................................................... 48
Figure 4.4 SEM image and EDS analysis of 304 ASS + 2TiN.............. 51
Figure 4.5 SEM morphology and EDS analysis of 304 ASS/4TiN......................... 52
Figure 4.6 SEM morphology and EDS analysis of mixed 304 ASS/6TiN........ 53
Figure 4.7 XRD analysis of admixed powder 304 SS with 2, 4 and 6 wt% TiN. ....... 55
Figure 4.8 PSD of feedstock 304 stainless steel and TiN and admixed 304 stainless steel matrix composite......................................................... 56
Figure 4.9 Relative density of composites after SPS process............... 57
Figure 4.10 SEM micrograph of SPS 304 ASS and EDS analysis............. 58
Figure 4.11 SEM micrograph of SPS 304 ASS + 2TiN and EDS analysis........ 59
Figure 4.12 SEM image and EDS analysis of 304 ASS/4TiN................. 60
Figure 4.13 SEM micrograph of SPS and EDS analysis of 304 ASS + 6TiN. ......... 61
Figure 4.14 x-ray diffractograms of 304 SS composite after SPS process........ 63
Figure 4.15 microhardness of 304 SS composites before erosion studies........ 64
Figure 5.7 SEM micrograph of 304 SS with 4 wt% of TiN a) at room temperature; b) at 400 °C and c) at 600 °C.................................................. 75
Figure 5.10 XRD patterns for 304 ASS composites at room temperature. .................... 79
Figure 5.11 XRD spectra of 304 ASS matrix composites at 400 °C. ............................ 80
Figure 5.13 XRD spectra of 304 ASS matrix composites at 600 °C. ............................ 81

LIST OF TABLES

Table 3-1 Erosion wear test conditions ........................................................................ 43
Table 4-1 Nominal chemical compositions of the as-received powders (wt%) ............ 45
Table 4-2 Chemical compositions of admixed powders ............................................. 45

APPENDICES A: LIST OF SYMBOLS AND ABBREVIATIONS

ASS    Austenitic stainless steel
DSS    Duplex stainless steel
C      Carbon
Cr     Chromium
Cu     Copper
EDX    Energy-dispersive X-ray spectroscopy
F      Frictional force
FCC    Face-centered cubic
Fe     Iron
<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
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<tbody>
<tr>
<td>HIP</td>
<td>Hot isostatic pressing</td>
</tr>
<tr>
<td>Nb</td>
<td>Niobium</td>
</tr>
<tr>
<td>Ni</td>
<td>Nickel</td>
</tr>
<tr>
<td>Min</td>
<td>Minutes</td>
</tr>
<tr>
<td>Mo</td>
<td>Molybdenum</td>
</tr>
<tr>
<td>Mn</td>
<td>Manganese</td>
</tr>
<tr>
<td>MPa</td>
<td>Megapascals</td>
</tr>
<tr>
<td>N</td>
<td>Normal Force</td>
</tr>
<tr>
<td>PVD</td>
<td>Physical Vapour Deposition</td>
</tr>
<tr>
<td>PM</td>
<td>Powder metallurgy</td>
</tr>
<tr>
<td>S</td>
<td>Sulphur</td>
</tr>
<tr>
<td>SE</td>
<td>Secondary electron</td>
</tr>
<tr>
<td>Si</td>
<td>Silicon</td>
</tr>
<tr>
<td>SPS</td>
<td>Spark plasma sintering</td>
</tr>
<tr>
<td>Ti</td>
<td>Titanium</td>
</tr>
<tr>
<td>TiN</td>
<td>Titanium Nitride</td>
</tr>
<tr>
<td>V</td>
<td>Vanadium</td>
</tr>
<tr>
<td>Term</td>
<td>Description</td>
</tr>
<tr>
<td>--------</td>
<td>--------------------------------------------------</td>
</tr>
<tr>
<td>XRD</td>
<td>X-Ray diffractometry</td>
</tr>
<tr>
<td>°C</td>
<td>Degrees celsius</td>
</tr>
<tr>
<td>°C/min</td>
<td>Degrees celsius per minute</td>
</tr>
<tr>
<td>µ</td>
<td>Coefficient of friction</td>
</tr>
<tr>
<td>γ</td>
<td>Austenite or gamma phase</td>
</tr>
<tr>
<td>~</td>
<td>Approximately</td>
</tr>
<tr>
<td>%</td>
<td>Percentage</td>
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CHAPTER ONE

1.0. INTRODUCTION

1.1. BACKGROUND

In modern industrial applications, engineering materials are being subjected to harsh demanding environments that require materials to possess good mechanical properties, good oxidation and corrosion resistance. Engineering materials such as titanium, aluminium and stainless steels are finding wider application in such environment (Obadele, 2014). Austenitic stainless Steels (ASS) type 304 is widely used in these engineering environments. 304 ASS has excellent corrosion resistance properties in various environments; as a result austenitic stainless steels find application in various industrial application which include thermal power plants, power generation plants, marine, urban water, chemical, aircraft gas turbine engines and food industries (Bregliozzi et al., 2004). Apart from corrosive environment application, high impact velocity of particles is frequently encountered in components used in these industries. Examples are coal fired boilers used in the power generation industries (Samandi et al., 1993). Although austenitic stainless steels are well known for their good corrosion properties, they are famous for poor mechanical properties such as friction and wear. According to Sun and Bell (2002), when ASSs are in contact with each other or other materials, they experience severe metallic wear which results from the adhesion junction and severe plastic deformation occurring between the surfaces of the materials in contact with each
other. Furthermore, ASS fails mainly because of wear rather than corrosion related material loss problems.

According to More et al., (2017), 50% or more investigators focuses on steel as their target material because steel is used in many industrial applications, however, it has been noticed that steel material can be effectively replaced or alloyed with some other materials such as titanium. Mainly because titanium is considered to be the second largest target material and they have good wear properties. Titanium nitride (TiN), which has relatively good wear properties, is intentionally alloyed to 304 ASS to improve the hardness and wear properties of the resulting material.

Wear can be defined as the loss of materials due to the mechanical action occurring between two or more bodies in contact with each other (Bayer, 2012). Wear phenomena include several mechanisms such as abrasive, adhesive, corrosive and erosion wear. Erosion wear mechanism plays an important role in determining the life span of components used in several applications such as in mining, solids transportation in pipelines, power generation and marine (Chawla et al., 2013). Erosion wear is a material deterioration process due to impacting particles on solid surfaces.

There are several methods that can be used to consolidate 304L ASS with TiN. Such methods include hot isostatic pressing (HIP), hot pressing (HP), microwave sintering and spark plasma sintering (SPS) (Smuk, 2004 and Brytan et al., 2009). The ASSs used in the study are spark plasma sintered. Reviews show that there is
rich literature on the erosion-corrosion and corrosion behavior of ASS (Smuk, 2004; Aribo et al., 2012; Kang and Lee, 2014 and Chaudhari et al., 2016,) wear behavior of this material has been extensively studied (Pietrowski, 2012 and Davanageri et al., 2016). Little literature on the erosion behavior of the ASS has been reported. However, no effort has been made to study the erosion behavior of the spark plasma sintered ASS high temperature parameter, especially when ASS is alloyed to TiN. Therefore, this study focuses on the erosion behavior of ASS grade 304 reinforced with TiN under high temperature conditions.

1.2. RESEARCH PROBLEM

Approximately around 20% failure of boiler tubes used in power generation plants results from the erosion wear mechanism and this leads to power plants shutting down in order to maintain and replace damaged boiler tubes. The process of maintaining and replacing damaged boiler tubes is costly and affect the entire production costs. The product of coal combustion consists of different amount of Sulphur and some non-combustible minerals known as fly ash. The fly ash are mainly hard abrasives minerals such as quartz, this hard material impact with the surface of the boiler tubes and erode them (Kumar and Kanwar, 2012).

From powder metallurgy processes, the choice of mixing parameters affects the resulting feedstock. If incorrect mixing parameters are employed an inhomogeneous feedstock will be obtained. Sintering parameters also have an impact in the resulting consolidated specimen if inappropriate sintering parameters are used. Poor densification on the consolidated solid specimen is achieved in this case. Due to the high melting temperature (2950 °C) of TiN arising from its strong
covalent bond, good densification is not easily achieved when TiN is present in the material during sintering process (Russias et al., 2006). These problems mentioned could be solved by properly selecting optimal powder mixing parameters, sintering parameters and erosive-wear parameters.

1.3. AIM AND OBJECTIVES

The aim of this project is to study the erosion wear behaviour of stainless steels matrix composite under high temperature environment.

The aim of the study will be achieved through the following objectives:

- Optimize spark plasma sintering parameters
- Characterize the stainless steels matrix composites for phase identification and microstructural evaluation.
- Determine microhardness value of the sintered composites.
- Evaluate the effect of temperature on erosion wear of the sintered composites.

1.4. JUSTIFICATION FOR THE RESEARCH

The desire to employ austenitic stainless steels in many harsh environments is generally due to their good resistance to corrosion and they are also targeted for high temperature applications when low-alloyed steels and carbon steels cannot offer such favourable properties. However, they lack good wear properties as compared to other families of stainless steels and these could lead to preterm failure in some of engineering industries. Among other failures that could be experienced in such industrial application, is erosion wear. Erosion wear is a major problem and these failures make it necessary to improve the erosion wear
behaviour of ASS (Recco et al., 2007). Engineering interest is to estimate the life span of equipment that is faced with degradation problems such as erosion wear problems and to investigate the possibilities of enhancement of their life.

Many researchers reported on the erosion-corrosion behaviour of stainless steels (Lopez et al., 2007 and Aribo et al., 2012) and cavitation erosion of ASS (Heathcock et al., 1982; Kwok et al., 2000; Bregliozzi et al., 2005). Several research studies have focused on erosion wear behaviour of ASS 304, ASS 316 and 904L and Duplex stainless steels standard grade 2205 and super duplex stainless-steel grade 2507 to name a few (Lindgren and Perolainen 2014; Yoganandh et al., 2014). However, many of these studies reported mainly on low temperature parameters (Sun et al., 1999; Patel et al., 2016) and parameters such as impact velocities and impact angle. No studies have been reported on high temperature erosion wear of spark plasma sintered specimen of austenitic stainless steels, especially when ASS grade 304 is alloyed to TiN.

1.5. SCOPE OF WORK

This work was carried out by initially mixing powders and the admixed as well as received powders were characterized using particle size distribution, XRD and SEM-EDS. The powders were consolidated using spark plasma sintering technique. The sintered specimens were characterized prior to testing for phase identification and microstructure. The mechanical properties such as hardness and the erosion behavior of austenitic stainless steel were studied under high temperature conditions as the main focus of the study.
1.6. STRUCTURE OF THE DISSERTATION

The structure of the dissertation outlines the summary of this dissertation. Chapter One presents an introduction, research problems, aim and objectives and the scope of the research.

Chapter two this chapter presents a literature review on stainless steels and the family of the stainless steel, main elements in stainless steels, Wear and its mechanism, spark plasma sintering, erosion wear studies on stainless steels.

Chapter Three provide a presentation on the materials and equipment used in the research, and experimental methodology adopted for achieving the set objectives of this research.

Chapter four focuses on the results and discussions of the erosion wear studies.

Chapter Five In this chapter, the conclusions and recommendations are presented and lastly, a list of reference materials and appendices are given.
CHAPTER TWO

2.0. LITERATURE REVIEW

2.1. INTRODUCTION

The erosion-corrosion mechanism of stainless steel has been extensively studied. To understand the erosion-corrosion mechanism, the erosion and corrosion should be studied individually and the synergistic effect between erosion and corrosion should also be determined. Although this mechanism involves the two phenomena, it was found that in most cases the erosion in the erosion-corrosion contribute a lot on material degradation than corrosion. Therefore, there is a need for a research on the erosion behavior mechanism, especially of stainless steels, because they are used in many engineering fields. This chapter gives an overview of what has been done; present and what should be done in future to understand the erosion study of Austenitic Stainless Steels.

2.2. Stainless steels

A stainless steel as the name entails is a material that is very resistant to stains. They are iron-based alloys which consist of a minimum content of chromium of approximately 10.5% chromium and carbon content in some certain grades ranges from less than 0.03% to over 1.0%. This sufficient chromium forms a passive film of an oxide layer on the surface and thus preventing the material from the reactive electrolyte/media and causes the stainless steel to be corrosion resistant. Other alloying elements are added to improve characteristics of the stainless steels and this alloying elements (except chromium) include nickel, molybdenum, sulfur, copper, aluminum, silicon, titanium, niobium, manganese, and selenium (Budinski
and Budinski, 2009; Lo et al., 2009). Appropriate alloying elements in the steel can result in the steel being ferritic, austenitic or a combination of ferrite and austenite (duplex stainless steel) or martensitic. Stainless steels are produced in cast, powder metallurgy, and wrought forms (Afolabi et al., 2009). The typical industrial application of stainless steel includes those the pharmaceutical, biomedical, oil and gas, petrochemical, construction sector, marine as well as household appliances to name a few (Bregliozi et al., 2005).

Figure 2.1 illustrates transformation of phases possible at particular temperatures and the compositions of Fe-Cr for the Fe-Cr system by not focusing on the effect of other alloying elements. This figure helps to illustrate how temperature influences the stability of the phase for the ferritic ($\alpha$), austenitic ($\gamma$) and duplex stainless steel ($\alpha + \gamma$) alloys. Furthermore, the figure illustrates the effect that the heat processes such as annealing, welding as well as high temperature applications of stainless steels causes on these phases (Banda, 2013).

![Fe-Cr phase diagram](image-url)

Figure 2.1 Fe-Cr phase diagram (Banda, 2013).
2.3. Classification of stainless steel:

The stainless steel can be categorized into four main groups and this classification is on the basis of their microstructure at ambient temperature: Austenitic stainless steel, Martensitic stainless steel, Ferritic stainless steel and Duplex stainless steel.

2.3.1. Austenitic stainless steels

The austenite phases are formed around temperatures of 912 and 1394 °C as shown in figure 2.1. Apart from iron, austenitic stainless steels (ASSs) consist of the two major alloying elements of chromium (17-18%) and nickel (8-9%), and some additional elements of molybdenum, carbon and nitrogen. Nickel in the austenitic stainless steels is responsible for stabilizing the structure (face-centred cubic crystal-γ structure) while chromium allows for high temperature corrosion resistance (Pascal et al., 2016). Their austenitic (face-centered cubic) structure is very tough and ductile down to absolute zero. These types of stainless steels are highly employed in many industrial applications owing to their excellent material properties such as being non-magnetic and excellent corrosion resistance (Vashishtha et al., 2016). ASSs can be made soft with yield strength of roughly 200 MPa to be easily formed; they can also be made very strong by cold work with yield strength up to 2000 MPa. They lack toughness at subambient temperatures and they also have lack of great ductility, specifically if more than about 30% elongation is needed.

2.3.2. Martensitic stainless steels
The martensitic grades adopted the name owing to the fact that heating them to about \( \sim 870 \, ^\circ C \) which is the critical temperature and cooling them rapidly, the resulting metallurgical structure known as martensite is obtained. They contain about 12-18\% of chromium and roughly 2-4\% nickel and they are heat treatable (Loto, 2016). They are well known for their high strength and this property is highly affected by heat treatment. The martensitic steels are usually used in applications where high corrosion resistance, high temperature, creep resistance and hardened conditions are required. In these conditions, the strength increases with the carbon content. Steels with more than 13\% Cr and carbon content above 0.15\% are completely martensitic after hardening. Martensitic stainless steels are used as scalpels, knives, hooks and tweezers in medical applications, drive systems and high-performance parts for airplanes (Laffler, 1999).

### 2.3.3. Ferritic stainless steels

Ferritic stainless steel is a grade of the stainless-steel family that has properties that are almost the same as those of the austenitic grade. They are non-hardenable and non-heat treatable alloys with a low carbon (0.08 to 20\% C) content. Ferritic stainless steels have about 11-17\% chromium and small amounts of ferrite stabilizers, such as aluminum, niobium, and titanium. Among all types of stainless steels, ferritic grades are characterized by low nickel content which results in a more stability in terms of costs and economic material as compared to austenitic stainless steels, however, their corrosion resistance is said to be low as a results of low nickel content. They are different from austenitic grades in that they have higher mechanical strengths and have lower thermal expansion, high thermal
conductivity and are easier to cut and work (Baddoo, 2007; Cashell and Baddoo, 2014). Ferritic stainless steels are mainly used in automobile and household appliances. They are magnetic at room temperature and have good resistance to atmospheric corrosion (Charles et al., 2009).

2.3.4. Duplex stainless steels

Duplex stainless steel, as the name implies “duplex”, have a two-phase microstructure that consists of grains of ferritic and austenitic stainless steel of approximately 1:1 ratio. These steels offer a favorable combination of properties such as good corrosion resistance in chloride containing environments, abrasion resistance and weldability, mechanical strength and ductility, and they are economically stable (Afolabi et al., 2009). The toughness and ductility of ferritic grades are lower than that of the DSS; however, austenitic stainless steels have higher values of toughness and ductility as compared to the DSS. When melting the DSS, the solid formed from the liquid phase is a complete ferritic structure. As the temperature drops to a room temperature, approximately around half of the ferritic grains are converted to austenitic grains resulting in a microstructure of about 50:50 ratio of ferrite:austenite (Kocijan and Conradi, 2010 and Ramaswamy and Ranganath, 2016). The strength of DSS is about half that of austenitic or ferritic stainless steel individually. However, DSS are more likely to be harmed by precipitation of phases than austenitic steels and this result in embrittleness (Olaseinde et al., 2014). DSS contains high content of chromium as well as high molybdenum and nitrogen giving rise to a high pitting-corrosion resistance in chloride solutions. Molybdenum increases the stability of the stainless steel and
this gives them the ability to resist localized corrosion, pitting and crevice corrosion in chloride ions containing to be specific (Kojican et al., 2009). Duplex stainless steels can be produced by powder metallurgy technology wherein pure ferritic powder and austenite (stabilizing element) powder are mixed together and sintered, the resulting dual phase microstructure of equal proportions of ferrite:austenite grains combines the attractive properties of both austenitic and ferritic steels (Kazior et al., 2004 and Garcia et al., 2007).

2.4. Effect of alloying elements on stainless steel properties:

Alloying elements in most cases are intentionally added to improve properties (such as corrosion, mechanical properties etc.) of specific alloys. Some alloying element with the help of the other makes properties of the alloy even more attractive, for example, steels consisting of Cr in the presence of Ni and Mo (Cunat, 2004). A few of alloying elements used in stainless steels is discussed below;

2.4.1. Nickel (Ni)

Nickel has superior strength and is very much corrosion resistant, therefore, it increases the strength of the steel by increasing the ferrite strength. The role of nickel when added to stainless steels is to increase the stability, toughness as well as the general corrosion resistance, chloride stress corrosion cracking resistance and pitting/crevice corrosion resistance (Muthupandi et al., 2005).
2.4.2. Molybdenum (Mo)

Molybdenum modifies the polarity of the passive film by causing molybdenum oxide layer in the outer part of the film thus preventing it from pitting corrosion attack. All stainless steels that contain molybdenum are generally more corrosion resistant than molybdenum free grades. When Molybdenum is used in the presence of carbides it also helps to increase creep resistance at elevated temperature mainly for application in hot working tools (Pardo et al., 2008).

2.4.3. Chromium (Cr)

Chromium is considered to the basic alloying element. Chromium in stainless steels offers favorable properties such as an increase in hardenability and strength, excellent corrosion resistance by forming stable oxide surface (it is responsible for passivation) and is typically used in combination with Ni and Mo (Todic et al., 2011).

2.4.4. Carbon (C)

Carbon is always present in metallic alloys, the most important constituent of stainless steel. It promotes strength and hardness by heat treatment, and resistance to wear and abrasion. However, carbon reduces ductility, toughness and machinability. In some instance, a small amount of C enhances the austenite stability behavior and it changes the tensile behavior (Noh et al., 2017).

2.4.5. Silicon (Si)

Silicon is one of the gasifier and deoxidizer. Silicon added to steels increases tensile and yield strength without loss of ductility, forgeability and enhances
magnetic properties. Silicon and copper are often added to austenitic steels in the presence of molybdenum in order to improve their corrosion resistance, especially in sulfuric acid environment (Basu and Yurek, 1991).

2.4.6. Titanium (Ti)

Titanium is very reactive and it forms TiN in the presence of nitrogen while it forms TiC in the presence of carbon. Titanium and its alloy have high strength and are have good erosion resistance. It improves hardness, strength and erosion resistance when added to steel, limits austenite grain size. Its carbide forming tendency in the presence of carbon is very strong that most often it is found in steel as undissolved carbides causing the hardenability to decrease (He et al., 1999).

2.4.7. Vanadium (V)

Vanadium stabilizes ferrite. It increases strength, hardness, creep resistance and wear resistance and impact resistance due to formation of hard vanadium carbides, limits grain size. It retards grain growth, permitting higher quenching temperatures (Abassi and Shokuhfar, 2007).

2.4.8. Manganese (Mn) & Sulfur (S)

Sulfur is normally not desirable in steel because it causes brittleness, however, it may be added deliberately to improve the machinability of steels. A very small amount of sulfur required because too much addition of sulfur can results in sulfide inclusion (Anmark et al., 2015). Manganese on the other side, small amount (<1%) of manganese, is present in steel as a deoxidizer or desulfizer to prevent brittleness and hardening (Pardo et al., 2008). When an amount of about 11-14%
of manganese is used as an alloy in steel, properties such as high hardness, good
ductility, high strain hardening capacity and excellent wear resistance are
achieved.

2.4.9. Niobium (Nb)

The function of niobium added to steels to improve the strength of the steel at high
temperatures (Fujita et al., 1996). Nb functions as a stabilizing element in ASSs
and it allows for the formation of carbide and nitride formers and thus preventing
the formation of chromium-rich carbide at grain boundaries. This lead to an
increase in intergranular corrosion resistance and pitting corrosion resistance of
ASSs during aging process (Desale et al., 2005)

2.4.10. Nitrogen (N)

Nickel sometimes replaces carbon to form high nitrogen steels (austenitic) and this
help achieve favourable properties because nitrogen is a more effective solid-
solution strengthener than carbon. Nitrogen increases the resistance to localized
pitting attack and inter-granular corrosion. Carbon is also used as austenite
stabilizer and thus reduces the need for adding nickel (which is known to be costly)
for stabilization (Simmons, 1995).

2.4.11. Phosphorus (P)

Phosphorus increase the strength and hardness of the steel, however if too much
of it is added to steel (0.03-0.05%), the steel resulting steel tend to have poor
ductility and toughness as such it is considered to be the impurity. (Rofagha et al.,
1993).
2.4.12. Copper (Cu)

Addition of about 0.10% to 0.50% of copper to stainless steels improve the general corrosion resistance especially in sulphuric media and provide reduced surface quality and hot-working ability. These are commonly used in low carbon sheet steel and structural steels and are also used to stabilize austenite (Pardo et al., 2007).

2.5. Titanium Nitride (TiN)

Titanium Nitride (also known as Tinite or TiNite) is a type of nitride material which is widely being used as coating material due to its good mechanical properties (hardness). It is classified as a refractory material due to the high melting points and its good chemical stability (Bavadi and Valedbagi, 2012). Additional to these properties, TiN has good electrical conductivity at room temperature and the bulk specimen can be machined by electro discharge machining (EDM) process and these results in the material being relevant to the recent technological advantage. TiN properties make it interesting in various potential industrial applications were material with good mechanical properties is required. It is mainly used as additives to other materials, as coating deposited by physical vapour deposition (PVD) or chemical deposition vapour (CVD) methods (Russias et al., 2006).

2.6. Spark plasma Sintering (SPS)

Sintering is a powder metallurgy (P/M) technique used to consolidate metal, ceramic or less often polymer powders into solid specimen. It takes place at elevated temperature usually close to the melting temperature with or without
pressure being applied (Rojek et al., 2017). During sintering, Powder particles are bonded together by diffusion to form a solid compact body. The densification occurs during sintering and the physical and mechanical properties are improved. Sintering is governed by the parameters such as holding time and sintering temperature, composition of the mixed powder, density of the powder compact and the composition of the protective atmosphere in the sintering furnace (Suárez et al., 2013). Different powder metallurgy processes exist to fabricate powders. These methods include some conventional powder metallurgy technique such as hot pressing (HP) and hot isostatic pressing (HIP) and spark plasma sintering (SPS). SPS has advantages over the others because densification of about ~98% is achieved in a short period of time and lower sintering temperature than the conventional technique due to its heating mechanism. Another advantage is that with the material fabricated by SPS have improved mechanical properties, optical properties and physical properties because grain growth is prevented (Shinohara et al., 2017).

2.7. Tribology

The term tribology originates from the Greek word “tribos” simply meaning “rubbing”. Tribology involves and focuses on the interacting objects in relative motion and it is generally known as the science of friction, wear and lubrication (Nilsson, 2012).

2.7.1. Friction
Friction is the resistance to the motion when one solid body is sliding over or along another. Frictionless surfaces do not occur in engineering application. When two surfaces are in contact the resistive, tangential force parallel to the direction of motion known as friction force will develop if one body begins to move on the other. The force required to maintain sliding is known as the normal force and it is directly proportional to the frictional force as shown on figure 2.2 Friction depends on surface topography, temperature, mechanical properties, and load (Buchholz, 2011).

\[ F_f = \mu N, \]

\[ F_g = mg, \]

\[ F_d = mg \sin \theta \]

Where: \( N \) = normal applied force, \( F_f \) = frictional force, \( F_g \) = force of gravity, \( F_d \) = force acting tangentially, \( \mu \) is the coefficient of friction, \( m \) is the mass (g) and \( g \) is the gravitational acceleration (m/s\(^2\)) (Wassink et al., 2001).
2.7.2. Lubrication

Lubrication is the process of reducing the wear between two or more parts sliding/moving against each other. While lubricants are the substances used to reduce the friction between surfaces rubbing against each other. These lubricants are classified as liquids (typically consists of 90% base oil and 10% additives) and non-liquids (include grease, Teflon tape used in plumbing industries and dry graphite powder or molybdenum disulphide powder). The characteristics of lubricants can be improved by adding a small addition of chemicals knowns as additives and these additives include anti-oxidant, foam depressant, corrosion inhibitors, detergent dispersers and oiliness improvers. Lubricant performance is affected by different factors such as loading parameters, the type of the lubricant used and the additives, friction induced heat; interface temperature, humidity etc. and this may affect the formation of tribofilms thus making the lubrication on the boundary less effective. The friction and wear would then increase due to the metal rubbing against the other metal as a result of the tribofilm failing. It is crucial to understand the friction and wear behaviour of the material subjected to lubrication environment in order to achieve maximum life span of materials without repeatedly stopping/starting the equipment for maintenance purposes (Zhang et al., 2015).

2.7.3. Wear

Wear is the progressive removal of materials from the surface of the solid body resulting from mechanical action (Bayer, 2012). Wear processes can be illustrated in terms of a tribosystem as shown in Figure 2.3. A tribosystem consists of a solid body, a counterbody, an interfacial element and the environment. The action
and/or interaction between the different parameters in the tribosystem lead to different types of wear taking place, for example, sliding, rolling, and impact wear (Sacks, 2002).

Figure 2.3 Wear processes on a tribosystem.

2.8. Wear mechanism

Wear mechanism can be divided into five main mechanisms of wear and the specific wear failure that takes place in each mechanism are shown in Figure 2.4 and further are summarized below:

- **Adhesion**
  - Fretting
  - Adhesive
  - Galling
  - Oxidative wear
  - Seizure

- **Abrasive**
  - Low Stress
  - High Stress
  - Gouging
  - Polishing

- **Fatigue**
  - Pitting
  - Spalling
  - Impact
  - Brinelling

- **Erosion**
  - Solid & Fluid impingement
  - Cavitation
  - Slurry Erosion

- **Corrosion**
2.8.1. Adhesive wear

Adhesion wear, often also called galling or scuffing, results from shearing mechanism of the interface caused between two or more bodies sliding or pressed into each other as can be seen in Figure 2.5 When a normal load is applied or in the sliding area of the material, the local pressure increases to higher values and if there are no lubricating films the surfaces adhere to each other and thus promoting material transfer from one body to the other (Luan and Robbins, 2006). To reduce this mechanism of wear certain factors such as lower load, harder materials, presence of lubricants are employed.

Figure 2.5 An illustration of adhesive wear mechanism (Kato and Adachi., 2001).

2.8.2. Abrasive wear

Abrasive wear is encountered in most of the areas, especially the severe environment such as excavation, mining, and minerals processing industries, where wear loss occurs on different parts of the equipment such as, rock drill bits, crushers, ball mills and rod mills, chutes and pumps, to name a few examples (Ndlovu, 2009). In a situation where dust and wear debris are not controlled or eliminated abrasive wear becomes a major problem. The wear of parts, the cost of
repair and replacement of these parts, and the associated downtime related to these activities result in significant costs to many industries (Watson et al., 1995). There are several factors that affect the abrasion wear behavior of a material. A few of these properties include, properties of material (hardness, particle shape, yield strength and fracture properties), wear material properties (microstructure, fracture toughness, corrosion, ductility, modulus of elasticity etc.) and concentration properties (force/impact level, velocity, impact/impingement angle, sliding/rolling, temperature, wet/dry, pH etc.) (Navhi, 2011)

![Two-body and Three-body Abrasive Wear Mechanism](image)

Figure 2.6 Schematic two-body and three-body abrasive wear mechanism (Devaraju., 2015).

### 2.8.3. Corrosive wear

Corrosive wear is the removal of material exposed to severe environment (such as acids, gases, and alkalis) as a result of two bodies in contact or rubbing. The corrosive type of wear occurs in a wide variety of environments (both lubricated and unlubricated). The fundamental cause of these forms of wear is a chemical reaction between the worn material and a corrosive medium that can be a chemical reagent, reactive lubricant or even air (Jones, 1992).
2.8.4. Fatigue wear

Fatigue wear is a material deterioration caused when a material is subjected to cycling loading. Fatigue usually occurs when a material is subjected to load that is greater than the strength of the material (Lundy, 1998).

Figure 2.8 Schematic illustration of fatigue wear mechanism (Buchholz, 2011).
2.8.5. Erosion wear

Erosive wear is loss of material as a result of solid/liquid particles impacting against the surface of a target material. Examples of this include dust particles impacting on gas turbine blades or slurry impacting on pump impellers (Kumar, 2011). It is a widely encountered mechanism in industries such as mineral and mining, thermal power plants, hydraulic turbines, and oil and gas production systems. The target materials that are usually used include: nickel hardened alloy steels, mild steel, stainless steel, aluminium alloy, brass, and chromium alloyed cast iron, to name a few. While the solid particles used as erodent include: silica sand, fly ash, quartz, feldspar, muscovite, biotite, garnet, and clays (Kumar and Ratol, 2013). Erosion wear is a problem that can be classified into three classes: solid particle erosion, cavitation erosion, and the liquid impact erosion. Amongst the three categories of erosion wear, solid particle erosion is the most common and the most studied. Several types of erosion wear exist. These include cavitation erosion, slurry erosion, and solid particle erosion wear. Each form of erosion wear is discussed below.

a) Cavitation erosion

Cavitation erosion wear is loss of material due to generation of gas inside the flowing liquid under certain conditions. Usually it occurs when the partial pressure of gases inside the liquid drops due to the vibration or turbulent flow. Cavitation is experienced in a wide range of applications including hydraulic systems or fluid machinery. Examples are pump impellers, turbines, marine impellers. Material
deterioration is mainly dependent on the duration the component is exposed to cavitation (Bregliozzi et al., 2007).

b) Slurry erosion

Slurry erosion is material loss due to slurry particles impacting on the surface of a component. It is mainly encountered in slurry handling and transportation equipment mainly in the mineral and mining industries. Slurry transport used in mines, for example, usually transport ore tailings or through pipelines over a distance to the tailing disposal or to the concentrate stockpile. Slurry erosion occurs when there is mechanical actions and interactions between the tailings/concentrate and the surface of the slurry transport equipment resulting in the equipment being damaged due to wear failure.

c) Solid particle erosion

The solid particle erosion is basically defined as the material removal from the target material because of repeated impingement of solid particles against the surface of the component (Desale et al., 2014). Particle solid erosion is different from other forms of erosion in that the material loss due to solid particle erosion results from a series of independent but similar impact. The hard particles come in contact with the surface of the component in a shorter duration. This distinguish them from wear mechanism such as sliding wear, abrasion etc. Solid particle erosion at room and high temperature applications is encountered in industries such as Chemical plant, Hydraulic mining machinery, Combustion system, Coal gasification and Coal liquefaction, and Fluidized bed combustor (Sundararajan and
Manish Roy, 1997). In order to understand and predict the erosion wear rate, the erosion wear mechanism and the effect of parameters on erosion should be investigated.

2.9. Parameters affecting erosion wear

More et.al (2017) studied and reported on the recent research status on erosion wear, they discussed in detail the parameters affecting erosion wear. They deduced a cause and effect diagram that consists of five parameters that are characteristics of carrier fluid, slurry, solid particles, target materials and condition of flow and the five characteristics were classified into sub parameters.

![Cause and effect diagram of erosion wear (More et.al., 2017)](image)

The parameters that are highlighted in red colour in the diagram show the most effective parameters that handle the erosion wear and are considered by most of
the investigators for the study. The importance of the controlling parameters on erosion wear is highlighted individually below:

2.9.1. Concentration of the particle

Concentration of solids is defined as the amount of solid particles by weight or by volume in the fluid. The more the concentration of the particle, the more particles strike the surface of the equipment and thus increased rate of erosion is achieved. Very high concentration of particles results in the decrease of the striking velocity of the particles and thus reduced erosion rate (Sundararajan and Roy, 1987).

2.9.2. Particle size and shape

Particle size and shape also forms part of important parameter affecting the erosion wear of a material. Mostly, investigations are made on the solid particle size as opposed to the particle shape. The rate of erosion increases with increasing particle size according the power of law whereas the relationship on particle shapes on erosion is not well understood (Sundararajan and Roy, 1987).

2.9.3. Velocity of flow

The effect of Impact velocity and impact angle have been found to be the parameters that have a major influence on wear rates in tribosystems that suffer erosion and erosion-corrosion (López et al., 2005). Usually erosion wear increases with an increasing velocity because at relatively higher speeds, high kinetic energy is seen at the surface of sample thus resulting in more erosion wear taking place (Mishra, 2011).
2.9.4. Impact angle

Impact angle can be defined as an angle between the surface of the target material and the direction at which solid particles impact on the surface of the target material at a specific impact velocity (Mishra, 2011). Angle of impingement has a detrimental effect on rate of erosion wear and wear mechanism. Many researchers reported that the maximum wear rate of ductile material is caused by the effect of impinging angles ranging from $15^\circ$- $30^\circ$, while brittle materials shows its maximum wear rate at $90^\circ$ (Finnie, 1971; Lindgren and Perolainen, 2014; Kosa and Göksenli, 2015).

2.9.5. Hardness

The hardness of a metal is used as a measure of the metal’s wear resistance. Surface hardness of the material and the hardness of the abrasive solid particles have an effect on erosion wear. It is believed that the harder a metal, the higher the resistance to wear is expected to be. If the material is harder than the solid particles, then the particles will not be able to scratch the material thus the wear rate will be decreased.

2.10. Erosion wear tester.

Several devices exist to quantify material degradation caused by the impacting solid particles against the component or the target material. The devices are typically modelled on a bench scale to simulate the conditions of the industrial application. The devices present include Gas-blast type erosion tester, Centrifugal accelerator type erosion tester, Gas gun impact tester, Whirling arm tester and
Free-fall test rig (Deng et al., 2008). The gas blast and the centrifugal accelerator tester are most commonly used on the basis of particle acceleration mechanism and as a result the two are discussed. The two facilities accelerate particles differently, the gas blast erosion tester uses pressurized gas to accelerate the particles while in the centrifugal acceleration tester, particles are accelerated by the centripetal forces in a rotating disc (Deng et al., 2008). Apart from the difference in how each device accelerates particles, the erosion rate achieved from these testers could be different although the test conditions are the same. This could be caused by the fact that the response of particles in each tester will vary depending on their characteristics and their interactions with the device. Although the gas blast and the centrifugal accelerator tester are commonly used, other erosion tester devices such as the free-fall type erosion tester exist. This accelerates the projectile by falling from a fixed height on to the surface of the target and they are usually used for single particle impact tests or other purposes (Deng et al., 2009). They are infamous and were last reported by Bitter (1963).

2.10.1. Gas-blast type erosion tester

In a gas-blast type of erosion tester (as shown in figure below), the abrasive particles mixed with compressed air are fed through a nozzle that can be adjusted to control the particle flux. The targeted material is fixed on a holder that can be adjusted to produce required angle of impingement (Stevens and Hutchings, 1995). In the case of high temperature experimentations, the tester is equipped with the electric furnace that provides heat to the erosion tester and target material could be heated to desired temperature.
2.10.2. Centrifugal accelerator type erosion tester

In this type of a feeder, the erodent particles are fed through a conical hopper feed down to the centre of rotating disc. The particles are then accelerated in the acceleration chamber to maintain desired velocity to impact on the surface of target materials.

Figure 2.11 Schematic diagram of centrifugal acceleration type erosion tester (Deng et al., 2009).
2.11. Erosive wear behaviour of austenitic stainless steel matrix composites.

Wear is the progressive removal of materials from the surface of the solid body resulting from mechanical action (Watson et.al, 1996). Wear results from contact between a surface and a body or substance that is moving relative to it. Wear is progressive because it increases when the amount of motion used increases, thus resulting in the transfer of material between surfaces (Bayer, 2012). In the previous years, effort has been made to understand the erosion wear behaviour of ASSs. Some of the studies carried out on the fabrication of the material and erosion wear behaviour of the materials are discussed below;

Study on solid particle erosion behaviour of stainless steel 304 at room temperature was carried out by Patel et.al (2016) and they conducted their tests using air-jet erosion test rig on boiler tube steel SS 304. The impact velocity used was 40m/s, the impingement angle was 30° and 90° on SS 304 at room temperature. Alumina particles of size 50 microns were used as the erodent. The initial and final weight of the specimens was taken using electronic weighing machine having a resolution of 0.01mg. They concluded that, erosion takes place through plastic deformation of material along with the formation of craters and lips. The eroded samples at impact of 30° showed that the material removal occurred by the ploughing effect and shear mechanism. The erosion rate of the SS 304 at impingement angle of 30° was found to be a little higher than the 90° angled and hence SS 304 exhibits ductile erosion mode.
Kumar and Kanwar (2012) investigated on erosion behavior of Cr$_2$O$_3$ coating on SS-304 boiler steel tubes in simulated coal fired boiler conditions. Erosion tests were conducted on uncoated as well as plasma sprayed coated samples. The erosion experiments were carried out using an air-jet erosion tester at impingement angles of 60 and 90°. It was found that the uncoated samples (SS–304) had the higher erosions rate at 60° than the 90° impact angles. The coated samples, SS–304 (coating of chromium oxide) gave better results for both 90° than at 60° angle. In the both cases high erosion rate occurred at 60°.

Shimizu and Araya (2011) conducted a study on solid particle erosion and mechanical properties of stainless steels at elevated temperature. The main aim of the study was to correlate the mechanical properties of stainless steel at high temperature and its resistance to erosion to estimate high temperature erosion behaviour. The material used in the study was two types of stainless steels, SUS403 and SUS630. High temperature solid particle erosion tests were performed using 1 mm alumina particles, at impact angles between 30° and 90° and a particle velocity of 100 m/s, with changing test temperature of room temperature, 573 K, 873 K and 1173 K.

The weight loss of a specimen was determined using an electronic scale. The erosion rate was then defined as following formulas to evaluate the removed material volume.

Volumetric removal per second (cm$^3$/s)  

\[
= \frac{\text{Mass removal per second (g/s)}}{\text{Average density (g/cm}^3)}
\]
Erosion rate (cm$^3$/kg)

= Volumetric removal per second (cm$^3$/s) ÷ Mass amounts of impact particles per second (kg/s)

They concluded that; in the case of high temperature erosion, the material softens by the high temperature, and the cutting wear is appeared remarkably. The erosion at an impact angle of 90°, where deformation wear is predominant, suggested that there is a proportional relationship between the decreasing percentage of hardness and the erosion rate. While at an impact angle of 30° at a test temperature of 1173 K, the difference of erosion rate between materials is thought to be a difference of the elongation, suggesting that in the case of erosive wear at a shallow angle, the material with a higher elongation easily formed protrusion that can be removed by successive impacting of erodent.
CHAPTER THREE

3.0. EXPERIMENTAL PROCEDURE

3.1. INTRODUCTION

Experimental procedures were performed to determine the erosive-wear behaviour of the austenitic stainless steels under high temperature environment. This chapter includes the materials used, details of the experimental methods and the equipment used in the study. Various techniques used to characterize the alloys in the study have also been highlighted. The chapter is divided into four main sections;

- Materials
- Powder characterization
- Sintering of powders by spark plasma sintering method
- and mechanical tests

3.2. POWDERS AND MATERIALS

As-received 304-austenitic stainless steel and TiN were chosen as the starting powders. The two feedstock powders were characterized for particle size distribution. Particle size is the most crucial physical property of many particulate powder or solid specimens. Particle size distribution measurements are performed in a variety of industries and these provide a clear understanding of the final products and processes. Microtrac S3500 PSD analyzer used in this study measures particle sizes from 0.02 to 2800 microns. It uses the 3 red laser diodes
(tri-laser) that provides accurate and repeatable particles sizes (Dada, 2015). The PSD analyzer is presented in Figure 3.1.

The weighed powders were then mixed (under dry environments) using Turbula Shaker Mixer T2F for 8 h at a speed of 72 rpm as shown in Figure 3.2. The function of mixing is to achieve uniformly blended samples without altering the properties required for the next steps. Admixed powders were then characterized using SEM-EDX and the phases formed were analyzed using XRD (Obadele, 2014). The admixed powders were also characterized for PSD.

The admixed powders were then consolidated using the spark plasma sintering technique in which specimens with dimensions of 35mm (length) and 1.5mm (thickness) were produced. The substrates were machined using the robofil 240-SL electrical discharge machine (EDM) wire cut.

Figure 3.1 Microtrac Particle Size Distribution (PSD) analyzer (Dada, 2015).
3.3. POWDER CHARACTERIZATION

3.3.1. Microstructure and phase analysis

Scanning Electron Microscopy (SEM, Tescan Vega 3) which has the Energy-dispersive X-ray spectrometer (EDS) attached to it was used to analyze the morphology of the feedstock and admixed powders to confirm the element present in the powders. Phase identification of the powder was also analyzed with X-ray Diffraction (XRD Rigaku Ultima IV).

![Figure 3.2 Turbula shaker T2F (Obadele., 2014).](image)

3.4. SPARK PLASMA SINTERING OF POWDERS

Admixed powders were sintered using spark plasma sintering technology (H-HPD25-FCT Systeme GmbH Germany) shown in Figure 3.3 a) and Figure 3.3 b) shows a typical image of spark plasma sintering device. Powders were sintered in a 40 mm graphite die and the thickness between the graphite foil (used to separate the graphite die and the punches) and the powder was 6.5 mm. The densification process took place at varying operating parameters under heating rate of 100 °C/min from room temperature up to the desired temperature (1100 °C) and holding
time of 10 min. The temperature was monitored using the optical pyrometer throughout the SPS process. A constant vacuum pressure of 50 MPa was applied in each test. The sintering parameters used are presented in Table 3.1. Disc specimen with dimensions 40 mm in diameter and approximately 6.5 mm in height at the end of the sintering process was achieved. After sintering, the discs are sand-blasted to remove any excess graphite material on them. After sintering, the density measurements of the sintered specimen were accurately weighed for five times using Mittler electronic balance and the average densities were recorded using the Archimedes' principle according to the equation below;

\[ SG = \frac{W_{\text{air}}}{(W_{\text{air}} - W_{\text{water}})} \]

Where, \( W_{\text{air}} \) the weight of sample in air and \( W_{\text{water}} \) is the weight of sample in water.
Table 3.1 Spark plasma sintering conditions

<table>
<thead>
<tr>
<th>Sintering Conditions</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Sintering temperature (°C)</td>
<td>1100</td>
</tr>
<tr>
<td>Holding time (min)</td>
<td>10</td>
</tr>
<tr>
<td>Heating rate (°C/min)</td>
<td>100</td>
</tr>
<tr>
<td>Vacuum pressure (MPa)</td>
<td>50</td>
</tr>
</tbody>
</table>

3.5. CHARACTERIZATION OF SPARK PLASMA SINTERED SPECIMEN

3.5.1. SEM-EDS analysis

The sintered samples were ground using Aka-Piato 120, Aka-Allegran 9 using DiaMaxx Poly 9µm and polished with Aka-Daran using DiaMaxx Poly 3µm. They were further polished with fumed silica using a polishing suspension of 0.2 µm to achieve mirror-like surface. The morphology of the sintered specimen were
investigated using Scanning electron microscope model TESCAN Vega 3 which is equipped with Energy-dispersive Spectrum (Nyembwe et al., 2015) as presented in Figure 3.4. The secondary electron (SE) micrographs were taken.

Figure 3.4 TESCAN Vega 3 SEM-EDS.

3.5.2. XRD analysis

The Rigaku Ultima IV X-ray diffractometer XRD equipped with a graphite-monochromatic Cu Kα radiation source at 40 kV and 30 mA was used to perform the XRD analysis. Collection of diffractograms took place in the 2θ range between 3° and 90° with a step size of 0.01 (Dada, 2015). The purpose of the XRD analysis is to identify the phases present/formed in the composites. XRD equipment used in the study is shown in Figure 3.5.
3.6. MECHANICAL TESTS

3.6.1. Micro-hardness

The effect of adding TiN on the hardness of the resulting composite were carried using the INNOVATEST Falcon 500 Vickers microhardness tester as shown in figure 3.6. The specimen tested under Vicker’s should be metallographically prepared. The specimen were indented with a load of 100 g and dwell time of 10 s on 10 different position were resulting average hardness was calculated. Microhardness was taken before and after the erosion-wear studies.
3.6.2. Erosion Wear Studies

Figure 3.7 shows an ultrasonic cleaner. This device serves as a function for removing contaminants that might have adhered to the surface of the material. It uses the cavitation bubbles formed as a result of high frequency (ultrasonic), high intensity sound waves and the waves agitate a liquid producing a force or vibration on contaminants and thus detaching them from the desired cleaned samples. In Figure 3.8, a schematic diagram of the rig that was developed and the actual laboratory setup is presented Figure 3.9. The erosion test rig used is equipped with a furnace that supply heat for high temperature uses and the compressed air that accelerate the abrasive particles which hit the surface of the sample and wear them out. Compressed air chamber also has an adjustable button that determines the required impact velocity. A temperature reading placed on the sample to
monitor the required temperature for the study. The sample holder on the test rig can also be adjusted to different impact angles such as 30 °C, 60 °C and 90 °C depending on the application or the desired parameters.

Erosion wear testing was carried out using a high temperature erosion tester shown in Fig.3.9. The erosion test rig used in the study was designed at the Universidad Nacional Del Sur (UNS), Argentina. The equipment works similar with that of the ASTM G76-04 standards. Prior to erosion tests, specimen were cut into approximately 35 mm x 1.5 mm x 1.5 mm dimension, ground and polished using sand papers 180, 220, 400 and 600 and cleaned in acetone for 2 mins in an ultrasonic cleaner as shown in figure 3.7. The erosion tester consist of particle feed inlet, air compressor and furnace. The alumina particles were used as erodent material and were accelerated through the feed inlet by compressed air at a constant speed of 18 m/s and this cause the abrasive particles to impact on the
sample. The sample holder could be positioned at different angles (30, 60 and 90°) by adjusting the sample holder. Erosion test were carried out at room temperature, 400 and 600 °C and the specimen were eroded for 10 min. However, at room temperature, specimen were removed every 2 mins to determine the weight lost. At high temperature, the samples were heated up to a desired temperature (400 and 600 °C), eroded for 2 mins and then allowed to cool. The weight loss for these materials at high temperature was recorded at the end of 10 mins. Table 3.1 shows the operating solid particle erosion wear parameters used in the study.

Table 3-1 Erosion wear test conditions

<table>
<thead>
<tr>
<th>Erodent Material</th>
<th>Alumina Particles</th>
</tr>
</thead>
<tbody>
<tr>
<td>Particle size (μm)</td>
<td>45</td>
</tr>
<tr>
<td>Particle velocity(m/s)</td>
<td>18</td>
</tr>
<tr>
<td>Impact angle (°)</td>
<td>90</td>
</tr>
<tr>
<td>Sample Temperature (°C)</td>
<td>25, 400, 600</td>
</tr>
<tr>
<td>Time (minutes)</td>
<td>10</td>
</tr>
</tbody>
</table>
Figure 3.8 Schematic diagram of the experimental set up.

Figure 3.9 Experimental setup (a) erosion tester equipped with a furnace (b) interior of the erosion tester (c) laser temperature reader.
CHAPTER FOUR

4. RESULTS AND DISCUSSION

4.0. INTRODUCTION

In this chapter, the results obtained in the study are discussed. The findings include the characterization of as-received powders, admixed powders and spark plasma sintered specimen using SEM/.EDS to study the morphology and to confirm elements presents in the starting powders. XRD was also used to identify the phase(s) present. The micro-indentations of the material are also presented.

4.1. CHARACTERIZATION OF AS-RECEIVED POWDERS

4.1.1. SEM/EDS of starting powders

Table 4.1 gives the nominal composition of the composites used in the study and in Table 4.2 compositions of weight percentage of ASS 304-TiN are presented.

The chemically stable Pure TiN powders were used as the reinforcement while ASS-304 was used as the matrix.

Table 4-1 Nominal chemical compositions of the as-received powders (wt%)

<table>
<thead>
<tr>
<th>Material</th>
<th>Fe</th>
<th>Cr</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>ASS 304</td>
<td>Bal</td>
<td>18</td>
<td>9</td>
</tr>
</tbody>
</table>

Table 4-2 Chemical compositions of admixed powders

<table>
<thead>
<tr>
<th>No.</th>
<th>Powder</th>
<th>Weight ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>304-ASS</td>
<td>100</td>
</tr>
<tr>
<td>2</td>
<td>304-ASS/2TiN</td>
<td>98:02</td>
</tr>
<tr>
<td>3</td>
<td>304-ASS/4TiN</td>
<td>96:04</td>
</tr>
<tr>
<td>4</td>
<td>304-ASS/6TiN</td>
<td>94:06</td>
</tr>
</tbody>
</table>
The SEM morphologies and EDS analysis of the starting 304 ASS and TiN powders used in this study are given in Figure 4.1 and Figure 4.2 respectively. From Figure 4.1, it could be seen that 304 ASS particles are spherical in shape, smooth and non-porous with a few satellites. This is typical for powders that were produced by gas atomization (Obadele et al., 2017). From Figure 4.2 it can be seen that the TiN powder has an irregular shape and it is porous. The TiN powder could have been produced by sol-gel method. This method is used to fabricate metal-oxide or metal chlorides (usually known as precursors), especially that of silicon and titanium. The morphology of particles produced by this technique is porous (Shongwe et al., 2017). EDS reveal Fe, Cr and Ni in the 304 ASS powder as the major elements and Ti and N as the main elements in the TiN starting powder.
Figure 4.1 Scanning electron microscopy morphology of as-received 304 ASS powder particles with EDS analysis.
Figure 4.2 SEM morphology of as-received TiN powder magnifications at (a) 1000x and (b) EDS analysis.
4.1.2. XRD analysis of feedstock powders

The XRD spectrum of as-received 304 ASS and TiN powders are given in Figures 4.3a and 4.3b respectively. In Figure 4.3 a, the XRD pattern shows the phases present in the starting 304 ASS powder. The phases that have emerged contain elements such as Cr, Ni and Fe which are the major of 304-stainless steel. Similarly, in Figure 4.3b, the diffractometer reveals that there is only TiN present in the powder and this so because the powder did not contain any alloying elements. The XRD results of the starting powders obtained in this study are in line with the EDS analysis.

![XRD pattern of the feedstock](image)

Figure 4.3 XRD pattern of the feedstock (a) 304 ASS and (b) TiN powders.
4.2. CHARACTERIZATION OF ADMIXED POWDERS

4.2.1. Microstructural analyses of admixed powders

Obadele, 2014 discussed the importance of powder mixing. The author defines powder mixing as one of the most important techniques in powder metallurgy technology. It is usually applied in the material processing techniques like thermal spray and laser material preparations. The degree of mixing has an impact on the value of the product; ineffective mixing can result in inhomogeneity of the final product. Mixing uniformly and effectively is extremely important for both in a case where the metals or ceramics are to form alloys or composites with particular properties, or to retain their individual characteristics independently (Obadele et al., 2012). Blending or mixing of particulate matter is widely used in various material processing industries. According to (Muzzio et al. 2002; and Obadele, 2012), dry mixing has been widely practiced by various researchers, however, there is little knowledge on the mechanisms taking place during dry mixing.

The starting powders were mixed using the optimum mixing parameters to determine the effect of mixing on the particle size of the as-received powders. The powders used in this study were mixed for 8 h at 72 rpm speed (Ranti et al., 2017). Figure 4.4 to Figure 4.6 display SEM microstructures of mixed composites with their respective EDS analysis. The smaller irregular TiN particle sizes can be seen attached on the 304 stainless steel (large particles with a few satellites). EDS also confirm the presence of TiN as well as the main elements present in the composite.
Figure 4.4 SEM image and EDS analysis of 304 ASS + 2TiN.
Figure 4.5 SEM morphology and EDS analysis of 304 ASS/4TiN.
Figure 4.6 SEM morphology and EDS analysis of mixed 304 ASS/6TiN.
4.2.2. XRD analysis of ad-mixed powders

The XRD spectrum of the blended powder was conducted to deduce the possible change in phases that could have formed. The XRD results of the mixed 304 ASS powder with 0, 2, 4 and 6 wt% TiN are presented in Figure 4.7. The Cu Kα radiation source at 40 kV and 30 mA set up was used and the collection of diffractograms took place in the $2\theta$ range between $5^\circ$ and $90^\circ$. The results reveal the very low peaks of TiN at peak position $2\theta = 36^\circ$, $43^\circ$ and $72^\circ$ respectively. The XRD patterns also display typical austenite phase as the main phase and has the highest peak which are observed at $2\theta = 43.884^\circ$ corresponding to the (111), $2\theta = 51.052^\circ$ which corresponds to the (200) plane and $2\theta = 74.980^\circ$ corresponding to the (220) plane (Kuo et al., 2007). The austenite phase consists of element such as Fe, Cr and Ni which basically forms the major elements present in the austenitic steel and the $\alpha$-Fe peak is also revealed at $2\theta = 45^\circ$ corresponding to (110) plane. These results corresponds to the conclusions made by Zhao et al., (2015). In their XRD results, they reported that their specimen showed a typical austenitic structure that have their peaks at; peak position where $2\theta$ equals to $43.6^\circ$ which corresponded to (111) plane, $50.6^\circ$ which corresponded to the (200) plane and finally $74.5^\circ$ which corresponded to the (220) plane. During mixing or blending, none of the feedstock peaks shifted. This could be so because no solid solution has been formed upon mixing or blending (Obadele, 2014).
4.2.3. PSD of the starting powders

The particle analysis results obtained from Microtrac PSD analyser are presented in Figure 4.8. It could be seen that the 304-ASS, 304 ASS/2TiN, 304 ASS/4TiN and 304 ASS/6TiN showed similar particle size distribution with average particle size of 26 μm while that of TiN showed a different particle size distribution with average particle size of 45 μm. Particle size distribution plays a crucial role in powder metallurgy because the characteristics of processing the powder depend on it. An increased porosity with coarse grain is achieved from particles of same sizes while that with dissimilar grain sizes aim to decrease porosity and thus increasing the density (Obadele, 2012).
Figure 4.8 PSD of feedstock 304 stainless steel and TiN and admixed 304 stainless steel matrix composite.

4.3. SPARK PLASMA SINTERING

Relative densities for the SPS composites used in the study are shown in Figure 4.9. From the graph, it could be observed that the densities of the composites decrease with increasing amount of TiN reinforcing material in the steel matrix. The reason for this trend may be due to lower density of TiN (5.43 g/cm$^3$) than that of 304 SS (8.03 g/cm$^3$). Relative densities of the composites were 97.65 %, 96.64 %, 96.56 % and 96.23 % for compositions 0 wt%, 2 wt%, 4 wt% and 6 wt% of TiN respectively. Sulimi et al., (2015) reported that the apparent densities of the composites decrease with increasing content of the reinforcing material in the matrix. This is so because of the lower densities the reinforcing material usually possess. On their studies on the effect of SPS parameters on densification and properties of steel matrix composites, they indicated that the apparent densities of the steel matrix were decreasing as the content of the TiB$_2$ reinforcing material was
increasing in the steel matrix. On the other hand, a decrease in relative density could result from the formation of pores around the TiN matrix as shown in Figure 4.10.

![Figure 4.9 Relative density of composites after SPS process.](image)

4.4. CHARACTERIZATION OF SPARK PLASMA SINTERED SPECIMEN

4.4.1. SEM/EDS of spark plasma sintered samples

Figure 4.10 to 4.13 present the SEM micrograph and its respective EDS analysis of the spark plasma sintered composites of different content of TiN. SEM also show the presence of nano-ranged of TiN phase which is presented by the light colour in the image and tends to settle at the grain boundaries of the 304 stainless steel matrix. 304 SS is represented by the dark-colour (black precipitates). A homogeneous powder mixing was achieved; this could be observed by the equal distribution of the reinforcement on the grain boundaries. Generally, the microstructures have large grain sizes. The elements present in the composites
have been confirmed with EDS. Fe, Ni, Cr, Ti and N were detected as the main constituents and other elements such as Si and Mn were present in a very small quantity. It could also be observed that the addition of TiN reduces the grain sizes. As the amount of TiN in the 304 ASS matrix increases, more of TiN segregate on the grain boundaries and the grain sizes become smaller.

Figure 4.10 SEM micrograph of SPS 304 ASS and EDS analysis.
Figure 4.11 SEM micrograph of SPS 304 ASS + 2TiN and EDS analysis.

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight %</th>
<th>Atomic %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe K</td>
<td>61.86</td>
<td>48.35</td>
</tr>
<tr>
<td>Cr K</td>
<td>18.91</td>
<td>15.88</td>
</tr>
<tr>
<td>Ni K</td>
<td>8.75</td>
<td>6.51</td>
</tr>
<tr>
<td>C K</td>
<td>4.46</td>
<td>16.22</td>
</tr>
<tr>
<td>N K</td>
<td>3.34</td>
<td>10.69</td>
</tr>
<tr>
<td>Ti K</td>
<td>2.59</td>
<td>2.36</td>
</tr>
<tr>
<td>Totals</td>
<td>100</td>
<td>100</td>
</tr>
</tbody>
</table>
Figure 4.12 SEM image and EDS analysis of 304 ASS/4TiN.

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight %</th>
<th>Atomic %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe K</td>
<td>57.92</td>
<td>43.12</td>
</tr>
<tr>
<td>Cr K</td>
<td>18.40</td>
<td>14.71</td>
</tr>
<tr>
<td>Ni K</td>
<td>7.63</td>
<td>5.41</td>
</tr>
<tr>
<td>C K</td>
<td>4.41</td>
<td>15.25</td>
</tr>
<tr>
<td>N K</td>
<td>5.28</td>
<td>15.68</td>
</tr>
<tr>
<td>Si</td>
<td>0.51</td>
<td>0.76</td>
</tr>
<tr>
<td>Ti K</td>
<td>5.58</td>
<td>5.08</td>
</tr>
<tr>
<td>Totals</td>
<td>100</td>
<td></td>
</tr>
</tbody>
</table>
4.4.2. XRD of spark plasma sintered composites

The XRD analysis of the SPS stainless steel composites with various amount of reinforcement (TiN) in wt% sintered at sintering temperature (1100 °C), heating rate (100 °C/min) and holding time (10 min) is given in Figure 4.14 The peaks were scanned at room temperature under argon atmosphere. The XRD reveals the presence of the following phases; TiN with a chemical formula TiN$_{0.90}$, phases containing iron, chromium and nickel such as FeCr$_{0.29}$Ni$_{0.16}$C$_{0.06}$, austenite. The
main elements of 304 ASS which are Fe, Ni and Cr, are revealed, these elements lie close to each other on the periodic table. Therefore, they possess similar properties and lattice parameters. In situations like this, it is practically impossible to accurately determine the phase composition of the spark plasma sintered 304 ASS composites because the XRD analysis of these composites can only prove the solid solution of iron (Sulima et al., 2015 and Oke et al., 2017). There has been no peak shift during sintering. However, there has been a slight change in peak intensities. The change in peak intensities could result from the sintering temperature, time and heating rate.

The XRD patterns before and after spark plasma sintering process are very similar. There has been no significant transformation or phase change during spark plasma sintering. However, cubic nitride (chemical formula, FeN$_{0.0939}$) has been detected in the major peak of ASS 304 with 2, 4 and 6 wt% TiN at peak position 2$\theta$ = 43.884°. This finding could be due to the intense phenomena that take place during the SPS process, which include surface activation, diffusion, surface melting as well as necking between sintered powder particles and plastic flow. An increase in mass transportation is obtained during sintering and this improves bonding between powder particles. The iron nitride that has emerged in the studied material after sintering is formed as a result of iron and nitrogen diffusing in the grain boundary of the steel matrix. It is very crucial to mention that the SPS process involve a stage where the material undergo a non-equilibrium conditions and this has a great impact on the reactions as well as the diffusions which take place during SPS (Oke et al., 2017).
4.5. MECHANICAL TESTS

4.5.1. Micro-indentation hardness tests

Hardness properties carried out on the ASS 304, ASS 304 + 2TiN, ASS 304 + 4TiN and ASS 304 + 6TiN as used in this study are given in Figure 4.15. These hardness tests were recorded before the solid particle erosion takes place. As expected, the hardness increases as the amount of TiN in the specimen increases. This is due to the fact that TiN has higher hardness and adding it to 304 ASS improves the hardness of the resulting material. ASS 304 6TiN shows high...
hardness value of about 356 HV$_{0.1}$ while ASS 304 + 4TiN, ASS 304 + 2TiN and ASS 304 showed hardness of 338, 330 and 275 HV$_{0.1}$ respectively.

Figure 4.15 microhardness of 304 SS composites before erosion studies.

Figure 4.16 further compares the effect of temperature on the hardness of the composites as the TiN content is increased. From this Figure, it could be observed that higher hardness properties are experienced at room temperature. The reason behind this could be to be the fact that hard abrasive particles have been embedded in the composites and thus increasing the material's hardness properties. Queresh and Tabakoff (1988) have conducted a study on the influence of coating process parameters on surface erosion resistance and substrate strength. They have concluded that an increase in hardness of a material after erosion tests could be achieved due to higher hardness properties the erodent particles possess. At higher temperatures (400 °C and 600 °C), there is a drop in hardness values on the impinged surfaces. According to studies reported by Huttunen-Saarivirta et al.,(2011), when a material is subjected to high temperature
effect, it becomes soft and loose its hardness properties. These findings are in full agreement with the results indicated in Figure 4.16 at higher temperatures.

Figure 4.16 Microhardness of eroded 304 SS composites at room temperature, 400 °C and 600 °C.
CHAPTER FIVE

5.0. INTRODUCTION
This section outlines the solid particle erosion-wear studies carried out on the composite at 25 °C, 400 °C and 600 °C. The erosion wear tests were conducted at a constant velocity of 18 m/s and angle of impingement of 90°. In this chapter, optical microscope of the erodent material used is given. The macrographs of the solid specimen before and after the erosion studies are discussed. Lastly, microstructures as well as XRD graphs of the samples after the erosion studies are discussed.

5.1. EROSION WEAR STUDIES
The effect of temperature (25 °C, 400 °C and 600 °C) on the rate of erosion of 304 ASS, 304 ASS/2TiN, 304 ASS/4TiN and 304 ASS/6TiN at an impact angle of 90° and an impact velocity of 18 m/s is presented in Figure 5.1. From the graph, it could be observed that the erosion wear behaviour of these materials shows a similar behaviour. The graphs show that material wastage increases with increasing temperature and as a result higher material loss is experienced at 600 °C while the least material loss is observable at room temperature. This could be due to the fact that generally, material wastage of stainless steel is dependent on temperature. Material loss increases as the temperature increases. This is observed up to a maximum temperature where a point of maximum material loss is achieved. In this case not only erosion wear occurs but the combined effect of erosion and oxidation takes place (Huttunen-Saarivirta et.al, 2011).
Figure 5.1 also compares the effect of temperature on material wastage as the amount of TiN hard phase is increased in the material. From the graph, it could be observed that at 25 °C the materials show almost similar weight loss between 28.9 mg and 31.4 mg. There could be particles loosening at high temperature and as a result, an increase in wear phenomena on the eroded surface. At 600 °C, it could be observed that 304 with TiN show similar trend and closely related weight loss between 50 mg and 55.15 mg. However, for 304 ASS without TiN at 600 °C, it was observed that weight loss was the highest compared to 304 ASS with TiN. This may be due to the fact that 304 ASS becomes more ductile as the temperature increases and loses its hardness properties (dos Santos et al., 2004 and Huttunen-Saarivirta et al., 2011).

![Graph showing weight loss vs temperature for different materials]
Figure 5.2 compares the effect of temperature (25 °C, 400 °C and 600 °C) changes on the erosion wear behaviour of the composites. The rate of erosion wear indicates that there is a proportional relationship between the decreasing percentage of hardness and the erosion rate, as the percentage of TiN in the material increases (meaning increased hardness) then erosion rate of the material decreases. This indicates that the higher the hardness of material the lesser prone the material is to the erosion wear loss. This is in line with the results reported by Davies et al., (1986). The author reported that the higher the hardness, the lower the rate of erosion of the material. While elsewhere, Hogman and Hedenquist supported studies conducted by Davies et al. On their study they concluded that the higher the hardness of the stainless steels the lesser prone the material is to the erosion wear loss.

Figure 5.2 Comparison of material wastage when TiN is added in the 304 stainless steel.
5.2. CHARACTERIZATION OF ERODED SAMPLES

5.2.1. MACROGRAPH OF ERODED SPECIMEN

The optical micrograph of alumina particles used as the erodent material is shown in Figure 5.3 and in Figure 5.4 (a), (b), (c) and (d) the surface macrograph of the studied materials are presented. Figure 5.4 (a) shows the material prior to erosion wear tests while (b), (c) and (d) shows the materials after being subjected to erosion wear at 25 °C, 400 °C and 600 °C respectively. In every case, in the region where erosion occurred, a circular shape is produced and it can be seen that erosion wear of the materials begins on the centre and spread towards the edges of the samples. These findings are similar to that obtained by Kumar and Kanwar (2012). They found that an erosion study carried out at an impact angle of 90° results in circular shape on the region where erosion has taken place while at an impact angle of 30° an elliptical shape is observable on the surface where erosion has occurred. Furthermore, the materials show the three layers in which the first layer is in the centre, where most of the material is removed, second layer where less material is removed and the last layer where very small amount of material has been removed. This can clearly be seen at Figure 5.4 (d) where 600 °C temperatures was used. This behaviour is similar to that reported by Kumar and Kanwar (2012) and Patel et.al (2016). In both studies, it was indicated that the macrographs of the eroded specimen revealed three regions at which material removal occurred. The first central region; from where most of the material has been removed, the second region; where lesser material has been removed and thirdly the outside region; where very less amount of erosion is observable.
Figure 5.3 Optical micrograph of alumina erodent particles.

Figure 5.4 Macrograph of specimen used during the erosion test; (a) before the erosion wear (b) after erosion wear at 25 °C (c) after erosion at 400 °C and (d) after erosion at 600 °C.
5.2.2. Microstructural analysis of eroded samples

SEM micrograph of the eroded composites is displayed in Figure 5.5, Figure 5.6, Figure 5.7 and Figure 5.8 under 25 °C, 400 °C and 600 °C conditions. The SEM of eroded samples provides useful information about the mechanism of erosion. According to Javaheri et al (2018), the mechanism at which erosion occurs by solid particle is broadly divided into two mechanisms; namely “cutting” and “deformation”. The cutting mechanism is categorized as the mechanism in which particles impact on the surface of the material at an oblique angle with sufficient amount of energy to make a groove on the surface of the target material. On the other hand, deformation mechanism involves particles impacting on the surface of the target material at a perpendicular angle with sufficient amount of energy to cause plastic deformation or subsurface crack on the surface of the eroding material. Apart from impinging angles, erosion can be classified in terms of ductility and brittleness. Ductile-erosion at an oblique angle results in material being removed by cutting or ploughing, while ductile erosion at a normal angle results in material removal by craters with formation of lips. Brittle-erosion results in the formation of subsurface cracks on the surface of the material being eroded. The combination of ductile and brittle mechanism may occur simultaneously, however, depending on the properties of the material one may predominate (Javaheri et al., 2018).

On the damaged surface, pitting action is clearly observed. Wear debris characterized with large fragments and several micro-cuts which could results from severe impingement of abrasive alumina particles are observable. From Figure
5.5, pitting could be seen. The pitting action could have been caused by the abrasive particles which indented on the surface of the material instead of sliding. 304 ASS shows abrasive alumina materials retained in their structure, especially at higher temperature as it could be seen on Figure 5.5b. From Figure 5.6, it is observed that the material is removed by the formation of cracks and plastic deformation and subsequent impact which led to material being displaced accompanied by large fracture fragments in Figure 5.6c. This erosion mechanism is similar to that reported by Patel et al., (2016). The authors concluded that in a steady state condition, a simultaneous action of three phases occurs on the surface of the material at different locations. During the initial phase, a crater is formed by the impacting particles and the material is displaced from the specimen. The second phase involves the material being displaced due to subsequent impact and this result in lateral material displacement accompanied by fracture. The third stage is experienced after few impacts where the materials becomes strained and therefore removed from the surface by fracture. Figure 5.7 and Figure 5.8 reveal the sign of erosion mechanism such as ploughing, micro-cuts, grooves as well as the wear debris on the surface of the materials. This is in full agreement with the study reported by Qin et al., (2017). The authors conducted a study on the erosion-wear and intergranular corrosion resistance properties of AISI 304L austenitic stainless steel after low-temperature plasma nitriding. It was reported that the most erosion damage taking place on the surface of the materials include; ploughing action, embedded particles, cutting grooves, large fragments, pitting action and wear debris on the surface of the sample that was nitride at 673 K.
Figure 5.5 Scanning electron micrograph of 304 SS a) at room temperature; b) at 400 °C and c) at 600 °C.
Figure 5.6 SEM micrograph of 304 SS/2TiN a) at room temperature; b) at 400 °C and c) at 600 °C.
Figure 5.1 SEM micrograph of 304 SS with 4 wt% of TiN a) at room temperature; b) at 400 °C and c) at 600 °C.
Figure 5.8 SEM micrograph of 304 SS/6TiN a) at room temperature; b) at 400 °C and c) at 600 °C.

Figure 5.9 compares the SEM micrograph of composites at 600 °C. From this Figure, it could be seen that the materials were dominantly damaged by brittle fracture and plastic deformation. Large fragments of wear debris could be observed in all specimen and plastic deformation in the form of ploughing, misplaced material (Figure 5.9b and Figure 5.9d) and a few grooves (Figure 5.9a and Figure 5.9c) are also observed. The results found in this study are in line with those conducted by Laguna-Camacho et.al (2013). 304L showed an unconventional wear mechanism which was characterized by large fragments on the surface of the eroded surface, and this was an indication that the removal mechanism was through brittle fracture. Also, pitting behaviour was observed.
Elsewhere, Bahri et.al (2016) on their study of the erosive wear of 304L stainless steel caused by olive seed particles impact supported the study reported by Laguna-Camacho et.al (2013). Their analysis revealed that the materials from eroded surface of 304L SS had been removed due to ploughing, pitting and wear debris were also detected.

![Figure 5.9 SEM micrographs of a) 304 ASS, b) 304 ASS/2TiN, c) 304 ASS/4TiN and d) 304 ASS/6TiN at 600 °C.](image)

**5.2.3. XRD analysis of eroded materials.**

The XRD pattern of the eroded 304 ASS, 304 ASS/2TiN, 304 ASS/4TiN and 304 ASS/6TiN at room temperature (25 °C), 400 °C and 600 °C are given in Figure 5.10, 5.11 and 5.12, respectively. The Cu Kα radiation source at 40 kV and 30 mA
set up in order to collect the diffractograms. At room temperature (25 °C), the XRD pattern is recorded between the $2\theta = 5$ and $90^\circ$. For 400 and 600 °C, the XRD pattern is recorded between $2\theta$ values of 15 and $90^\circ$. A slight change in relative intensity has also been encountered. At room temperature, the austenite peaks slightly changed to lower values at peak positions $2\theta = 43.771^\circ$, $50.939^\circ$ and $74.867^\circ$. This shift in peaks could also be as a result of the stress on lattice parameter after the erosion study when alumina impacted on the nanocomposites (Li et.al., 2017). Ideally, a shift in peaks to a lower 2-theta angle values results from compressive stress while a shift to a higher values of 2-theta angle results from tensile stress (Sawabe et.al., 2017). XRD analyses also reveal the formation of new Al-Ni (B2) peak 400 °C and 600 °C temperatures. Furthermore, at 400 °C and 600 °C, new alpha-Ti peaks were observed on the XRD graph of 304 ASS + 6TiN.
Figure 5.2 XRD patterns for 304 ASS composites at room temperature.
Figure 5.3 XRD spectra of 304 ASS matrix composites at 400 °C.
Figure 5.4 XRD spectra of 304 ASS matrix composites at 600 °C.
CHAPTER SIX

6. CONCLUSION AND RECOMMENDATIONS

6.1. CONCLUSIONS

In this study, solid particle erosion wear behaviour of the spark plasma sintered 304 ASS, 304 ASS/2TiN, 304 ASS/4TiN and ASS 304/6TiN was conducted under 25 °C, 400 °C and 600 °C conditions at 90° and a constant impact velocity of 18 m/s. The study included hardness tests before and after erosion tests. XRD and SEM/EDS were used to identify phase formed and microstructural analysis of eroded specimen, respectively. Based on the results obtained, the following conclusion could be drawn;

- SPS results given in terms of relative densities show a decrease in relative density with increasing content of TiN.

- An increase in TiN value in the matrix increased the hardness values before and after erosion studies. An increase in temperature results in decreased hardness properties of the composites.

- The erosion wear rate indicates that there is a proportional relationship between the decreasing percentage of hardness and the erosion rate, as the percentage of TiN in the material increases (meaning increased hardness), the material wastage decreases.

- Macrographs of the eroded specimens show three layers in which the first (central) layer represents high material loss, the second layer where lesser amount of material wastage is experienced and the third (outer) layer where the least amount of material was removed.
Microstructures indicate that the erosion damage occurs mainly by plastic deformation and brittle fracture. Large fragments in the form of wear debris, pitting, ploughing with slip on the side, misplaced material and a few grooves are observed.

- 304 SS shows abrasive materials being embedded in their structure, especially at higher temperature. At 6 wt% of TiN, material has been removed from the surface in the form of flakes and some parts were flattened.

- XRD reveal that there is a new phase (B2: AlNi) formed after erosion at 400 °C and 600 °C and the formation of alpha-Ti on 304 ASS + 6TiN.

**6.2. RECOMMENDATIONS**

The erosion-wear is experienced in boiler-tubes in power plant generations where high temperatures are employed. In this type of environments, both erosion and corrosion exist. It is recommended to conduct studies on erosion, corrosion and the synergistic effect of erosion and corrosion at 25 °C, 400 °C and 600 °C.
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