2017

Optimization Parameters for Cavitation Erosion and Corrosion using Robotic FSP of NABs.

Azman Ahmad

University of Wollongong

Recommended Citation


Research Online is the open access institutional repository for the University of Wollongong. For further information contact the UOW Library:
research-pubs@uow.edu.au
Optimization Parameters for Cavitation Erosion and Corrosion using Robotic FSP of NABs.

By
Azman Ahmad

A thesis submitted in fulfilment of the requirements for the award of the degree:
Degree of Doctor of Philosophy

The University of Wollongong
School of Mechanical, Materials & Mechatronic Engineering
March 2017
ABSTRACT

Background: The wide-spread usage of Nickle Aluminium Bronze alloy is observed for casting of components in the environmental applications. The reflection of endurance, corrosion resistance and extreme strength is emerged from an oceanic environment. The factors of nickel aluminium bronze are effective in demonstrating fracture toughness at high and low temperature along with a tensile strength. Friction stir process is an innovative technique used to enhance the performance of metals including aluminium, titanium, copper, nickel and steel-based alloys. Friction stir process is effective in modifying and controlling the microstructure in the surface region. It also assists in experiencing an enhanced mechanical property on the basis of rotating friction stir process tool. Objective: The main objective of this study is to explore the significance of optimization parameters for robotic friction stir process using Computer Numerical Control friction stir process. The study further aimed to investigate the hardness of microstructure elements of nickel aluminium bronze. By considering vibratory waves and corrosive medium, cavitation erosion and corrosion of Nickle Aluminium Bronze alloys were also analysed. Methodology: A Friction Stir Process has been used in order to analyse the influence of cavitation erosion and corrosion of nickel aluminium bronze. The robotic system was used to represent the low force technique on the basis of optimization parameters. The samples were acquired for mounting and polishing hardness, microstructural hardness, corrosion and cavitation testing. The samples were examined by using the optical microscope and SEM. The images obtained from these tools were used for analysing. Hardness testing was further computed through the acquired samples. The calculation and graph plotting were obtained by using a potentiostat apparatus. Nickel aluminium bronze specimens were used for the experiments with different chemical compositions. Results: The results for the microstructure hardness and cavitation erosion have been examined from the optimization parameters. The higher range of rotation speed (RPM) with low traverse speed (mm/s) leads to the highest surface hardness. A rotation speed of 3000 RPM with a traverse speed of 0.5 mm/s and 1 mm/s provide the highest hardness readings of $HV_{0.2} = 290$ and $299$ respectively. It has been examined from the results that acceptable parameters for CNC and articulated robot ranges between 3000 RPM to 4000 RPM. In the FSP, the average grain size lies between $1\mu m$ to $5\mu m$. The second zone exhibits a banded primary $\alpha$ and $\beta$ phase. In the third zone, there is the presence of $\alpha$ phase and $\beta$ phase with an equiaxed size of average $5 \mu m$. It has been examined that the higher range of rotation speed (3000 RPM-4000 RPM) reduces the mass loss.
of 54 percent while the lower range reduces to 35 percent of mass loss relative to as cast Nickle Aluminium Bronze alloy during the cavitation experiment. Conclusion: The microstructural evolution of FSP was analysed significantly through the optimization parameters. The cavitation erosion and corrosion attributes were associated with hardness of the specimens. The layers of cast nickel aluminium bronze were associated with the microstructures of FSP. The specimens used for the FSP significantly increases the properties of hardness, fatigue and tensile strength. It has been suggested that more parameters must be used for analysis of Nickle Aluminium Bronze alloy specimens.
ACKNOWLEDGEMENTS

First and foremost, I give gratitude to the Almighty Lord for His mercy and blessing on me to accomplish this study in spite of the numerous constraints that I encountered. I would like to express my deepest gratitude to my principal supervisor Professor Huijun Li, for giving me the opportunity to embark on this PhD study with his intellectual guidance, invaluable support, continual encouragement, and patience throughout this work. His inspirational knowledge and understanding of metallurgy was accompanied by continuous support of any idea or suggestion always finding the best solution in my rambling situation. To be your student has been a real pleasure for me. I also would like to express my deepest gratitude to my co-supervisors, Dr Stephen Pan and Dr Stephen van Duin for their unending invaluable advice and supervision and for providing me with the guidance and encouragement to finish this thesis. I thank you for your patience and kindness and, most importantly, your willingness to share your knowledge during my PhD voyage. In addition, I have been very privileged to have the opportunity to collaborate with many other great scientists such as Dr Dominic Cuiri, Professor Zhue, Nathan Larkin, Dr Sina Jamali and others. I really appreciate the sacrifice of time out of your incredibly busy schedules to perform experiments with me, analyse data and to correct numerous manuscripts as well as this thesis. I would also like to thank the following support staff who have given me unending help between their busy schedule such as Greg Tillman, Ron Marshall and Stuart Rodd.

I would also like to acknowledge the support that has been given to me by Tony Romeo and Dr Mitch Nancarrow and for the use of equipment on the UOW innovation campus. I also acknowledge the support and service that was given by Dr Denis Whitfield to proofread my thesis.

I would like to thank the Malaysian Government, especially the Majlis Amanah Rakyat (MARA) for the scholarship support during my studies at the University of Wollongong. I am also grateful to the University of Kuala Lumpur, Malaysia for granting me study leave and a generous stipend for my family during my doctoral studies in Australia. I am also grateful to University of Wollongong for providing me a High Degree Research (HDR) top-up scholarship. Many thanks to my friends in Wollongong such as Fathul, Mad Ithnin, Faiz Din, Wani, Nizam, Hairuddin, Sameha, Norzizi and Bayu. For those I have forgotten, it’s only on paper but not in my heart.
Lastly and most importantly, I wish to express my gratitude thanks to my lovely wife, Hasnita Hashim and my children (Hafis, Nasir, Aisyah, Maman and Anis), my beloved parents Sophiah Dalib and my late Father (Hj Ahmad Ali), Hamimah Yasin and Hashim Hamzah for their prayers and moral support. Their patience, encouragement, and understanding enabled me to consistently concentrate on my PhD study and was greatly appreciated.
DECLARATION

I, AUTHOR, declare that this thesis submitted in fulfilment of the requirements for the conferral of the Degree of Doctor of Philosophy, from the University of Wollongong, is wholly my own work unless otherwise referenced or acknowledged. This document has not been submitted for qualifications at any other academic institution.

Author
August 2016
# TABLE OF CONTENTS

ABSTRACT ............................................................................................................................... i
ACKNOWLEDGEMENTS ........................................................................................................ iii
DECLARATION ........................................................................................................................... v

CHAPTER 1: INTRODUCTION ...................................................................................................... 1
  1.1 Background ..................................................................................................................... 1
  1.2 Problem Statement ....................................................................................................... 3
  1.3 Research Scope ............................................................................................................ 4
  1.3.1 Research Objectives ................................................................................................. 4
  1.4 Outline of Thesis ........................................................................................................... 5

CHAPTER 2: LITERATURE REVIEW .......................................................................................... 7
  2.1 Background ................................................................................................................... 7
  2.2 Constructions of Nickle Aluminium Bronze alloy .......................................................... 7
  2.3 Kappa Phases (K) ......................................................................................................... 11
    2.3.1 Kappa I Phase ($K_1$) ............................................................................................. 12
    2.3.2 Kappa II Phase ($K_{ii}$) ......................................................................................... 12
    2.3.3 Kappa III Phase ($K_{iii}$) ....................................................................................... 12
    2.3.4 Kappa IV Phase ($K_{iv}$) ....................................................................................... 13
    2.3.5 Martensite Beta ($\beta'$) ....................................................................................... 13
  2.4 Concept of Friction Stir Process (FSP) ........................................................................... 14
  2.5 Process sequence and tools .......................................................................................... 17
    2.5.1 First Phase (Plunging Phase) ................................................................................ 18
    2.5.2 Second Phase (Dwelling Phase) ........................................................................... 18
    2.5.3 Third Phase (Processing) ..................................................................................... 18
    2.5.4 Fourth phase (Retracting) .................................................................................... 19
  2.6 Advancing and Retreating Side ..................................................................................... 20
  2.7 Advantages of Using FSP ............................................................................................. 20
  2.8 FSW/FSP Tools ............................................................................................................. 21
  2.9 Mechanical Properties Enhancement and Grain Refinement of NAB alloys ............ 22
  2.10 Robotic FSP ................................................................................................................. 23
    2.10.1 Robot Constraints ................................................................................................. 25
<table>
<thead>
<tr>
<th>Section/Title</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.11 Failure Mechanism</td>
<td>26</td>
</tr>
<tr>
<td>2.11.1 Background</td>
<td>26</td>
</tr>
<tr>
<td>2.11.2 Corrosion</td>
<td>26</td>
</tr>
<tr>
<td>2.11.3 Electrochemical and Crevice Corrosion</td>
<td>27</td>
</tr>
<tr>
<td>2.12 Corrosion Measurements</td>
<td>28</td>
</tr>
<tr>
<td>2.12.1 Weight loss measurement</td>
<td>28</td>
</tr>
<tr>
<td>2.12.2 TAFEL Plots</td>
<td>29</td>
</tr>
<tr>
<td>2.13 Cavitation</td>
<td>30</td>
</tr>
<tr>
<td>2.13.1 Erosion Rate</td>
<td>32</td>
</tr>
<tr>
<td>2.13.2 Estimation Method for Cavitation</td>
<td>33</td>
</tr>
<tr>
<td>2.14 Discussion and Summary of literature review</td>
<td>33</td>
</tr>
<tr>
<td>CHAPTER 3: FSP SETUP AND METHODOLOGY</td>
<td>34</td>
</tr>
<tr>
<td>3.1 Background</td>
<td>34</td>
</tr>
<tr>
<td>3.2 ABB IRB 6660 Robot</td>
<td>34</td>
</tr>
<tr>
<td>3.2.1 Force/Torque Sensor</td>
<td>35</td>
</tr>
<tr>
<td>3.2.2 Motor Spindle System</td>
<td>35</td>
</tr>
<tr>
<td>3.2.3 Robotic FSP Test-bed</td>
<td>36</td>
</tr>
<tr>
<td>3.2.4 FSP Tool pin</td>
<td>36</td>
</tr>
<tr>
<td>3.2.5 LabVIEW Program</td>
<td>37</td>
</tr>
<tr>
<td>3.2.6 Program Structure</td>
<td>37</td>
</tr>
<tr>
<td>3.2.7 Operator Interface</td>
<td>38</td>
</tr>
<tr>
<td>3.2.8 Design of experiment (DOE)</td>
<td>38</td>
</tr>
<tr>
<td>3.2.9 Robotic Endurance Test</td>
<td>39</td>
</tr>
<tr>
<td>3.2.10 Types of FSP Runs</td>
<td>41</td>
</tr>
<tr>
<td>3.3 FSP CNC Milling Setup</td>
<td>42</td>
</tr>
<tr>
<td>3.3.1 CNC FSP Runs</td>
<td>43</td>
</tr>
<tr>
<td>3.4 Summary and Conclusion</td>
<td>45</td>
</tr>
<tr>
<td>CHAPTER 4: EXPERIMENTATIONAL METHODOLOGY</td>
<td>46</td>
</tr>
<tr>
<td>4.1 Introduction</td>
<td>46</td>
</tr>
<tr>
<td>4.2 Material Used</td>
<td>46</td>
</tr>
<tr>
<td>4.3 Samples Preparation</td>
<td>47</td>
</tr>
<tr>
<td>4.3.1 Polishing</td>
<td>47</td>
</tr>
</tbody>
</table>
6.3 Summary of Hardness ........................................................................................................ 110
6.4 Summary of Cavitation ..................................................................................................... 110
6.5 Summary of Corrosion ...................................................................................................... 111
REFERENCES ......................................................................................................................... 112
APPENDIX .............................................................................................................................. 119
LIST OF FIGURES

Figure 1: Copper-Rich End of Cu-Al Phase Diagram (Source: Cuevas et al., 2002 pg.14)...............................................................7
Figure 2: Vertical Section through Cu-9% Al-5% Ni- 5% Phase diagram (Source: Cuevas et al. 2002 pg.15)...............................................................8
Figure 3: Schematic Presentation of β Phase breakdown of NAB Alloy (Source: Cuevas et al., 2002 pg.16)...............................................................10
Figure 4: Optical Microscope of Cast NAB (Source: Cuevas et al., 2002 pg.17).................................10
Figure 5: Schematic formation of Martensite (Source: Cuevas et al., 2002 pg.17).................................11
Figure 6: Schematic of Phase Transformation Diagram (Source: Cuevas et al., 2002 pg.19).........................13
Figure 7: Lattice Structures of NAB Phases (a) BCC β (b) Kiii phase B2 (NiAl) and c) Ki, Kii, Kiv phase DO3 (Fe3Al) Structures (Source: Cuevas et al., 2006 pg 18).................................................14
Figure 8: Method of FSP Processing (Source: William R.A., 2004 pg. 20).........................................................14
Figure 9: FSP Areas of SZ, TMAZ and HAZ (Source: Choi el et al, 2007, pg. 6).................................16
Figure 10: FSP/FSW Overall Phases (Source: Khairuddin et al, 2012, Welding Processes)..17
Figure 11: Schematic of Tool Processing Phase (Source: Shultz, 2010, pg. 3).................................19
Figure 12: Schematic diagram of Shoulder shape (a) spiral (b) dome (c) linear (d) scoop (e) concentric circle (Source: Zhang et al, 2012, pg. 252)...............................................................19
Figure 13: Sample of FS Tool pin........................................................................................................22
Figure 14: FSW/FSP Machine (Source: CASC, 2012).............................................................................23
Figure 15: ABB IRB 6660 Robot Used for FSP/FSW ...........................................................................24
Figure 16: Excitation Waveform for Polarization Resistance (Source: Princeton Applied Research, 2010, pg. 8)..................................29
Figure 17: Tafel Plots (from Princeton Applied Research, 2010, pg. 8) .................................................30
Figure 18: Effect of cavitation of ship propeller (Source: Johneck, 2011, pg. 1).................................31
Figure 19: Shock waves produced by collapse and rebound of a cavity (Source: Hammitt, 1980, pg. 68) ........................................................................................................................................31
Figure 20: Impingement of liquid micro-jet (Source: Soyama, 2005, pg. 1)........................................31
Figure 21: Schematic Representation of Erosion Rate with Exposure Time.................................32
Figure 22: ATI Force/Torque Sensor...................................................................................................35
Figure 23: Assembly of ATI Force/Torque Sensor and Shuner spindle With Test bed..............35
Figure 24: Schematic of Signal flow for Robot.................................................................................36
Figure 25: Dimension and FSP Tools With Pin .................................................................37
Figure 26: Outlook of the LabView Program .................................................................38
Figure 27: LabVIEW Graphic User Interface .................................................................38
Figure 28: Parameter 4000 RPM, Force 1750 N and Traverse speed 0.5 mm ..............40
Figure 29: Parameter 3200 RPM, Force 1750 N and Traverse speed 0.5 mm ..............40
Figure 30: Parameter 4000 RPM, Force 1750 N and Traverse Speed 0.5 mm ..........41
Figure 31: (a) Acceptable Runs (b) Reject Runs .............................................................42
Figure 32: Multiple Runs ...............................................................................................42
Figure 33: Schematic of CNC Sensor Attachment .........................................................43
Figure 34: Assembly of Test Plate, Insulator, Holding Plate and Force Sensor ..........44
Figure 35: CNC FSP Test Runs .....................................................................................45
Figure 36: Common Area of Hardness Measurement ..................................................49
Figure 37: Hardness Indentation Grid ............................................................................49
Figure 38: Schematic Corrosion Test Cell (Source: AR Tuncdemir, 2013) .................51
Figure 39: Schematic Ultrasonic Processor With Probe ..............................................53
Figure 40: AFM Feedback loop .....................................................................................53
Figure 41: Scope Trace with Optimized Realtime Parameters (Source: R. Robbins, 2010) ...54
Figure 42: Sample of Surface Profile after Polishing ..................................................55
Figure 43: SEM Schematic (Source: J. Witkke, 2008) ..................................................56
Figure 44: CNC FSP ......................................................................................................63
Figure 45: Graph of Axial Force Vs RPM .....................................................................64
Figure 46: Graph of Axial Force Vs Traverse Speed ....................................................65
Figure 47: Montage of FSP Cross Section Indicating the Details of the Zones .........71
Figure 48: SEM Illustration of As-Cast NAB ...............................................................71
Figure 49: OM Illustration of Zone 1 ............................................................................71
Figure 50: OM Illustration of Zone 2 ............................................................................72
Figure 51: OM Illustration of Zone 3 ............................................................................72
Figure 52: OM Illustration of Zone 4 ............................................................................72
Figure 53: Transformation Sequence During β Cooling (Cuevas, 2002, pg. 16) .........73
Figure 54: Image of OM and Microstructure with Peak Temperature Plotted against Depth 76
Figure 55: Graph Of Hardness Vs Rotation Speed ......................................................77
Figure 56: Rotation Speed 3000 RPM .......................................................................79
Figure 57: Rotation Speed 1000 RPM

Figure 58: As-cast NAB Cavitation Surface

Figure 59: Sample of Specimen after Polishing (0 Hour)

Figure 60: Cast NAB during Cavitation Erosion

Figure 61: Cast NAB after 12 Hours of Cavitation

Figure 62: RFSP Surface after 12 Hours of Cavitation

Figure 63: Micro Cavity and Wormhole

Figure 64: Optimum Cavitation Graph

Figure 65: Potential OCP For CNC and RFSP

Figure 66: Potentiodynamic Graph

Figure 67: Ductile tearing

Figure 68: EDS corrosion

Figure 69: XRD Results for the Samples

Figure 70: XRD Results for Samples and Compound Database

Figure 71: Percentage of Compound (S-Q)

Figure 72: XRD Results for Compound Database

Figure 73: LabView Mapping of the Circuit
LIST OF TABLES

Table 1: Comparison between Area of Studies and Experimentation ........................................4
Table 2: Table of Parameter........................................................................................................44
Table 3: Cavitation Table...........................................................................................................45
Table 4: Details of Potential and Current ..................................................................................94
Table 5: Polishing and Grinding Table......................................................................................119
<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Full Form</th>
</tr>
</thead>
<tbody>
<tr>
<td>AFM</td>
<td>Atomic-Force Microscopy</td>
</tr>
<tr>
<td>ANSI</td>
<td>American National Standards Institute</td>
</tr>
<tr>
<td>ASTM</td>
<td>American Society for Testing and Material</td>
</tr>
<tr>
<td>BCC</td>
<td>Body Centre Cubic</td>
</tr>
<tr>
<td>BM</td>
<td>Base Metal</td>
</tr>
<tr>
<td><strong>CNC FSP</strong></td>
<td>Computer Numerical Control FSP</td>
</tr>
<tr>
<td>DOE</td>
<td>Design of Experiment</td>
</tr>
<tr>
<td>DOF</td>
<td>Degree Of Freedom</td>
</tr>
<tr>
<td>EDS</td>
<td>Energy Dispersive Spectrometer</td>
</tr>
<tr>
<td>F/T</td>
<td>Force/Torque</td>
</tr>
<tr>
<td>FCC</td>
<td>Face Center Cubic</td>
</tr>
<tr>
<td><strong>FSP</strong></td>
<td>Friction Stir Process</td>
</tr>
<tr>
<td>FSW</td>
<td>Friction Stir Welding</td>
</tr>
<tr>
<td>GUI</td>
<td>Graphical User Interface</td>
</tr>
<tr>
<td>HAZ</td>
<td>Heat Affecting Zone</td>
</tr>
<tr>
<td>LSV</td>
<td>Linear Sweep Voltammetry</td>
</tr>
<tr>
<td>MMI</td>
<td>Man and Machine Interface</td>
</tr>
<tr>
<td>MPY</td>
<td>Mili per Year</td>
</tr>
<tr>
<td>NAB</td>
<td>Nickle Aluminium Bronze alloy</td>
</tr>
<tr>
<td>OM</td>
<td>Optical Microscope</td>
</tr>
<tr>
<td>PFZ</td>
<td>Precipitate Free Zone</td>
</tr>
<tr>
<td>Ra</td>
<td>Roughness Average</td>
</tr>
<tr>
<td><strong>RFSP</strong></td>
<td>Robotic FSP</td>
</tr>
<tr>
<td>RPM</td>
<td>Revolution Per Minute</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning Electron Microscope</td>
</tr>
<tr>
<td>SZ</td>
<td>Stir Zone</td>
</tr>
<tr>
<td><strong>TMAZ</strong></td>
<td>Thermo Mechanical Affecting Zone</td>
</tr>
<tr>
<td>UTS</td>
<td>Ultimate Tensile Strength</td>
</tr>
<tr>
<td>YS</td>
<td>Yield Strength</td>
</tr>
<tr>
<td>ZRA</td>
<td>Zero Resistance Ammeter</td>
</tr>
</tbody>
</table>
CHAPTER 1: INTRODUCTION

1.1 Background

Nickel-aluminium bronze (NAB) are copper base alloys that are extensively used for casting of components in the oceanic environment applications. Such a vigorous environment reflects the extreme strength, endurance and corrosion resistance exhibits by these alloys. NAB’s also display some exceptional mechanical properties. Among these attributes, few of them are demonstrated such as superior fracture toughness at high and low temperature with a moderate tensile strength. It also shows a good resistance against cavitation erosion effect and low coefficient of friction with exceptional wearability (Mishra & Ma, 2005). These factors lead to most suitable alloys against marine environmental deterioration which are widely accepted for components such as pumps, valves, gears and propellers.

The cast Nickel Aluminium Bronze alloy (NAB) alloy are normally custom made for propellers assigned as UNS C 95800, which are recognised as alpha nickel aluminium bronze or propeller bronze (Mishra & Ma, 2005). The NAB propellers are usually sand casted and slow cooled according to the American Society for Testing and Material (ASTM) B148 (Thomas et al., 1995) of NAB specification and sand casting technique. The complex microstructure and phases transformation of NAB is reflected upon the gradual cooling, which contributes significantly to the material properties (Thomas et al., 1995). The difference in NAB microstructures is due to the dissimilarity of the cooling rate during the casting process. Existing defects such as porosity and cracks were also detected, affecting the components strength, cavitation and corrosion resistance.

The occurrences of selective phase corrosion such as de-alloying and de-aluminification have been detected during the corrosion test on NAB specimens after a long period in seawater immersion test. It has been detected that fusion welding of cast NAB during propeller repair works induces defects and promotes non-equilibrium condition in thermal stresses, which stimulate the corrosion attack to the metal (Mishra & Ma, 2005). Annealing heat treatment is an effective process to mitigate the affect, which is applied after the fusion weld alters the alloy’s microstructure and obtains more suitable mechanical properties (Dawes, 1995).

The cast NAB propellers attributes have low cooling rates which causes a large grains size microstructures hence large components will shows a lesser strength relative to a smaller casting component (Thomas et al., 1995). However, the propellers are required to endure repetition of high force at the surface as it turns and moves through the water. Compression,
shear and stress are the forces that allow the surface to withstand and turns and moves through the water. In addition, the propeller also experiences a continuous effect of corrosion and cavitation that causes a major failure to the structure. Hence, it is necessary to introduce a new process that is able to reinforce the required area at the surface without altering the dimensions of the propeller blades. The new process must be able to reconstruct the microstructure and changes the mechanical properties to generate a better ductile, hard, tensile strength, corrosion and cavitation resistance. The parameters of Friction Stir Process (FSP) can fundamentally assist in improving surface properties regardless of transforming into chemical composition. Additionally, FSP can persuade grain refinement that consequently enhances the surface properties; such as corrosion and wear. On the contrary, the chemical composition of the surface is changed by FSP to instigate reinforcements within the fabrication of the surface metal matrix nano composites and surface metal matrix composites. Thus, FSP grain refinement is used to increase surface energy as compared with the coarse grained unprocessed samples.

FSP is emerged as a branch of surface engineering technology that can be adapted to metals such as aluminium, titanium, copper, steel and nickel–based alloys (Mishra & Ma, 2005). FSP is an innovative concept extracted from Friction Stir Welding (FSW); a solid-state joining process founded by Wayne Thomas from Thames Welding Institute in 1991 (Mahoney et al., 2003). A particular function of FSP is its ability to create localized modifications and control of the microstructure in the surface region without altering the overall shape and dimensions of the components.

FSP is able to produce extreme plastic deformation of an alloy and homogenize the microstructure and grain refinement. Therefore, the alloys experience newly improved mechanical properties (Oh-ishi, Zhilyaev & McNelley, 2006). The method comprises of rotating FSP tool, which plunges the surface and traverse across the metal surface, generating severe plastic deformation due to the stirring action in the metal under of the tool (Song et al., 2013). The FSP tool is manufactured with a conical pin protruding at the centre of bottom surface and leaving behind a few millimetres of flat area known as the shoulder. The pin is completely plunged during the rotation about the cylindrical axis.

The FSP is a solid-state process which means the parent metal maintains a solid form throughout the whole process. The material plasticizes due to the adiabatic heating, resulting in a very high distortion rates relative to the tool motion. During the extreme plastic
deformation, the metal could be displaced to the other side of the tool. The soft metal is then forged by the pressured surface in contact with the tool shoulder and the pin profile (Mishra & Ma, 2005).

Earlier studies regarding the effect of FSP on industrial aluminium alloys have exposed that the process was able to modify and refine the grain structure (Culpan & Rose, 1978; Fuller, 2006). The processed samples exhibit improvement for mechanical properties such as increment of static strength and increase ductility.

1.2 Problem Statement

FSP can make significant improvements to the surface properties of cast NAB; therefore, attention must then be given to the engineering aspects of applying this process industrially. According to (Mishra, Ma & Charit, 2003), the plunge forces required for FSP/FSW varies from 5000 N to 8000 N. These values are acceptable if using a dedicated FSW machine; however, these machines are typically restricted to flat surfaces over a limited area. For marine components, feature contours and other complex geometry are unsuitable for FSW machines. Industrial robotic manipulators could be utilised in these situations to carry out FSP on non-planar surfaces. The high number of movable Degree of Freedom (DOF) of a manipulator and its relatively large work volume can allow it to support a FSP tool along a contoured surface.

One related drawback to this proposal is that robotic arms cannot supply the force specified to carry out the process (Mishra, Ma & Charit, 2003). The vibration generated from the FSP is also an area of concern as robot manipulators do not have the rigidity of dedicated FSP equipment. Therefore, the process must be conducted under a relatively low force situation. Oh-Ishi & McNelley (2004) have conducted an analysis using a robot for the process but the study was mostly in the tensile properties area. There is paucity of researches that relates to cavitation erosion and corrosion with regards to robotic FSP (rf, using low force to conduct the stirring process. Hence, there is a need to analyse whether using a low force technique would be able to produce the same outcome (or better) as the dedicated FSP machine.

There is no specific research on the acceptable parameters of the robotic FSP and FS machine that could produce optimum mechanical improvement especially in the cavitation erosion and corrosion findings (Table 1). No researcher has stated that they follow certain guidelines to choose the parameters before conducting their experiment to produce the results as expected. Thus, it is crucial that defined parameters are required for researchers or industrialists to use as a future reference. Choosing the proposed parameters will definitely produce the finest results.
The conclusion is made based on the paucity of researches and finest applications of optimization parameters specifically in the cavitation erosion process. Furthermore, the assistance of FSP can consequently provide optimum mechanical improvement and finest results in the cavitation erosion and corrosion findings.

Table 1: Comparison between Area of Studies and Experimentation

<table>
<thead>
<tr>
<th>Areas of Studies</th>
<th>Mishra, Murray, Mahoney</th>
<th>NI, Xie, Ma, Cuevas</th>
<th>Schussler, Exner</th>
<th>Al-Hasheem, Riad, Hasan</th>
<th>William McNelley</th>
<th>This Thesis</th>
</tr>
</thead>
<tbody>
<tr>
<td>FSP machine</td>
<td>√</td>
<td>√</td>
<td>X</td>
<td>√</td>
<td>X</td>
<td>√</td>
</tr>
<tr>
<td>Robot</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>√</td>
<td>√</td>
</tr>
<tr>
<td>Cavitation</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>√</td>
<td>X</td>
<td>√</td>
</tr>
<tr>
<td>Corrosion</td>
<td>X</td>
<td>X</td>
<td>√</td>
<td>√</td>
<td>X</td>
<td>√</td>
</tr>
<tr>
<td>Optimum parameters</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>√</td>
</tr>
<tr>
<td>Hardness</td>
<td>√</td>
<td>√</td>
<td>X</td>
<td>√</td>
<td>X</td>
<td>√</td>
</tr>
<tr>
<td>Microstructure</td>
<td>√</td>
<td>√</td>
<td>X</td>
<td>X</td>
<td>√</td>
<td>√</td>
</tr>
</tbody>
</table>

1.3 Research Scope
1.3.1 Research Objectives

It has been shown that Friction Stir is a solid-state joining process for localised metal surface improvement. As-cast NAB is inseparable with the marine components, particular interest regarding the destructive effect of the environment to the alloys is necessary. Most of the components exhibit a contour surface; therefore, robotic FS is the most suitable technique to be applied. Thus, the first objective of the study is to find acceptable FSP parameters for the robot FSP and CNC application in terms of tensile strength, hardness and microstructure process. The analysis will include a series of variation parameter of plunging force, traverse speed and rotation speed.

Continuation of the analysis is the second objectives to analyse the relation of the variation parameters for both Robotic FSP and Computer Numerical Control FSP (CNC FSP), which affects the hardness and microstructure of the process NAB alloys.
The penultimate objectives are to analyse the effect of cavitation erosion and corrosion of the alloys when subject to continuous vibratory waves and corrosive medium respectively. The study would spearhead the direction of the optimum parameters from both techniques. The final objective is to justify the optimum parameters for both techniques, which examines the production of parameters against the environmental attack. The results will be concluded on the basis of optimum parameters when FSP is concerned.

1.4 Outline of Thesis

The first chapter is prepared to provide an introduction to this thesis. It discusses the usage of cast NAB and its importance to the marine industry. In addition, some information regarding the destructive effect of the environment to the NAB alloys structures is provided. Information related to the surface modification is also discussed in this section. The importance of the research and the objectives of the research are also focused. The section pinpoint the critical issues that need to be solve and hence the needs to carry out the study. Literature review is prepared to introduce a background of the constituents of the microstructure and phases in section 2.1 that presents the cast NAB alloys. A detailed transformation of different phases and grains formation during the cooling of the alloys was presented in this chapter. Section 2.2 includes the detail information of FSP and all the relevant process. Furthermore, it involves some discussion related to the tool used and different types of tool available. Literature is added in this section regarding the CNC and the articulate robotic arm chosen to implement low force for the non-symmetrical surface. Section 2.3 discusses about the failure mechanism faced by the cast NAB alloy when subjected to the environmental attack. Two types of failure are critical to propeller endurance that is the corrosion and cavitation erosion attack. A detailed discussion of the surface attack and mass loss were discussed to understand the gradual process that leads toward failure. A more comprehensive understanding of the studies that have been conducted and formulated to accomplish the objectives of this thesis is also discussed.

In chapter 3, the experimental equipment setup for the FSP is demonstrated in this thesis. The two mechatronic systems have been used to evaluate the thesis objectives. The first system is to articulate robot; therefore, the purpose is to process the samples with a series of different parameters. The second system comprises of three axis industrial HAAS CNC machine that is
used to verify the obtained results. For each system, the mechanical system parameters are characterised and are subsequently used in the subsequent chapters.

In section 3.2, a discussion of the experiments conducted after the samples for FSP is presented. The discussion includes, cutting, polishing techniques, cavitation experiment and the corrosion experiments undertaken. All the results of the experiment were recorded and analysed.

In chapter 4, section 4.1 discusses about the preparation done to conduct the FSP robotic process and related preparation to conduct the FSP CNC process. As the articulate robot is prone to high vibration subjected with heavy duty task, a minimum accepted parameter is used for testing and documenting. The purpose is to verify the safe working parameter for the continuation experiment. As CNC machine is very rigid than a robot, a vibrational effect is not a disturbing factor while conducting the test. A series of experiments were designed and conducted with a variation of interchangeable parameters for both CNC and robotic process. The information obtained is used to categorise the parameters and hence towards getting the optimum results.

Finally, all the variants were recognised and a new fixture to hold the samples was ready; therefore, the experiments were ready to be conducted.

Chapter 5 discusses about evaluating the results obtained in chapter 3. The results of experiments conducted are discussed and evaluated to provide a comparison between the CNC FSP and the robotic FSP results.

Chapter 5 presents the results obtained and a discussion about their relevance. The chapter includes a comparison of CNC and robotic techniques to obtain the optimum parameters. It also discusses the robotic capability, cavitation and corrosion experiments and compares selected parameters. A conclusion discusses the selected parameters that produce optimum results.
CHAPTER 2: LITERATURE REVIEW

2.1 Background
This section contains background information of the cast NAB alloy structure and phases together with its phase transformations. In order to understand the microstructure changes, a typical cast NAB material was chosen as sample. The variation of properties is used as an advantage to appreciate the cast NAB microstructure of constituent parts such as valves and propellers.

The second part is a detailed description of the FSP and its outstanding ability to enhance mechanical properties of metals. It also includes some evidence of the types of FSP tool and its schematic motion to have a clearer understanding regarding the process.

The last part is about implementing a low force technique for the FS process. An ABB IRB 6660 articulate robot was chosen due to its ability to manoeuvre un-flat surface, which is very suitable for marine components. Some information related to CNC machine was also stated for the purpose of analysing the robotic process.

2.2 Constructions of NAB
For a better understanding of the cast NAB alloy, related literature of the phase transformation during the cooling of NAB is discussed. It involves the formation of different phases occurred at various temperatures until the full phase is established at solidification temperature.

Figure 1 presents the copper-rich end of the Cu-Al binary equilibrium phase diagram.

![Figure 1: Copper-Rich End of Cu-Al Phase Diagram (Source: Cuevas et al., 2002 pg.14)](image)
The γ phase is considered to damage the corrosion resistance and the mechanical properties of the NAB alloys. Due to the high aluminium content, the γ phase shows a lower electrochemical potential than its neighbouring copper-rich α phase and consequently it has a very high potential to corrode (Liu et al., 1997). The corrosion of aluminium is denoted as dealuminization, which is similar to the dezincification of brass. Thus, countermeasures must be taken to minimise the formation and growth of the γ phase without affecting and reducing the mechanical properties. The alloys that have a lot of the γ phase will be less strong and less ductile compared to the NAB alloys (Field et al., 2001). The addition of other alloying element has been proven to minimise the retardation of γ phase formations. A destructive effect of γ phase is revealed on the polymeric materials and consequently deteriorate the mechanical, physical and chemical properties. Additionally, the effects of γ phase is shown in reducing tensile strength and elongation that indicate that the radiation is making even more inelastic.

The addition of iron and nickel to the Cu-Al alloys results in widening α phase area and invites a bigger addition of Al without developing the Y phase formation (Field et al., 2001) (Figure 2). The Fe and Ni alloying results in the precipitation of complex intermetallic K phases from the β and α phases has significantly increased the NAB’s mechanical properties (Mahoney, 2005).

![Phase Diagram](image)

**Figure 2:** Vertical Section through Cu-9% Al-5% Ni- 5% Phase diagram (Source: Cuevas et al., 2002, pg. 15)

By increasing the aluminium contents of NAB alloys to around 8.8 wt % to 10 wt %, studies have shown that the strength, hardness and the corrosion resistance is improved. Results have shown a reduction in elongation, which is related to the presence of the lamellar $K_{iii}$ phases
within the grain boundaries (Mahoney, 2005). The alloys with 9.5 wt % of Al produce optimum property mixture for the manufacturing of propellers following the addition of other alloys such as 5 wt % nickel, 4 wt % iron and 0.5 wt % manganese.

Alloys that contain more than 3 wt % Fe will display a reduction of grain size and restricted higher temperature grain growth and; therefore, solidification ranges also decline. The compositions comprising of 3 wt % to 5 wt % Fe have shown increased strength and strength retention at high temperatures. This combination results in enhanced attributes of wear, abrasion resistance and higher fatigue endurance limits. The maximum value of 4 wt % Fe has been designated to give the best mixture of properties (Mahoney, 2005).

With an addition of 0 to 0.5 wt %, Ni has increased the strength and refined the grain size as compared to Fe. On the other hand, the addition of Ni minimises the transformation of β phase while cooling increases the hardness simultaneously. An alloy with the concentration of Nickel less than the iron concentration may become susceptible to rigorous second phase attack in seawater. For the optimum corrosion resistance, it has been established that the Nickel content should be more than the iron content and preferably 5 wt % Ni with 4 wt % Fe (Mahoney, 2005).

Nickel-Aluminium Bronze alloys (NAB) are copper-based alloys with a chemical composition of 81 wt % Cu, 9 wt % Al, 5 wt % Ni and 4 wt % Fe. A number of research projects has been done to categorise the chemistry, microstructure and crystallographic characteristic of the cast NAB alloy (Mahoney, 2005; Hovanski et al., 2015). As seen in Figure 2 at a nominal 9 wt% Al content, the NAB starts to solidify and form a single phase β body centred cubic structure body-centre cubic (BCC) in solid solution. As the cooling continues, the β phase disintegrates to a new formation of face centred cubic (FCC) α phase and consequently by continuous transformation to α, which consist of various multi K phases. Cuevas (2002) has categorised the K phases into four significantly distinct types which are, $K_i, K_{ii}, K_{iii}$ and $K_{iv}$ with different morphologies observed using the (OM). The categorization is also related to the sequence of phase formation during the cooling stage. The schematic in Figure 3 and 4 shows typical phases present in the microstructures of NAB in standard conditions and Figure 5 shows the schematic of the formation of martensite. Due to non-conformity of thickness during the casting of the propeller, sudden cooling or quenching of the β phase region will form a martensite structure.
**Figure 3:** Schematic Presentation of β Phase breakdown of NAB Alloy (Source: Cuevas et al., 2002, pg. 16)

**Figure 4:** Optical Microscope of Cast NAB (Source: Cuevas et al., 2002, pg. 17)
Figure 5: Schematic formation of Martensite (Source: Cuevas et al., 2002, pg. 17)

The phase is the copper-rich area, which dominates the microstructure with light grey colour areas. α phase is the equilibrium terminal of solid solution, which exhibits an FCC structure with a lattice parameter of \(a=3.64\,\text{Å}\). In the phase, there is also the possibility of the presence of Windmanstätten morphology. During the cooling period, a pro-eutectoid α phase forms the β phase starting at about \(900^0\,\text{C}\) and at about \(580^0\,\text{C}\). A eutectoid reaction produces an outcome with a mixture of α phase and \(K_{iii}\) phases. Furthermore, \(K_{iv}\) particle presents precipitations between α phase matrixes while cooling (Cook et al., 2003).

Jahanfrooz et al. (1983) have stated that during slow cooling in the β phase, a Windmanstätten pro-eutectoid α phase precipitation from both inter-granularly and intra-granularly was observed. The intra-granular precipitation was found to be more dominant in coarse-grained alloys and happens in large castings such as propellers and; therefore, shows results of slow cooling rates.

2.3 Kappa Phases (K)

While cooling, the Ni-Fe-Al K-phases precipitate from α and β phases. According to Jahanfrooz et al. (1983), K phases have four different phases known as \(K_i, K_{ii}, K_{iii}\) and \(K_{iv}\) with different morphologies.
2.3.1 Kappa 1 Phase ($K_i$)
The $K_i$ phase is observed in the NAB microstructure with the presence of iron more than 5 wt%
Fe and is spotted as a large rosette-shape. The iron-rich $K_i$ precipitates with an estimated
grain sizes ranging from 20 µm to 50 µm and the locations are at the centre of the grains (Figure
6). According to Jahanafrooz et al. (1983), the $K_i$ phase exhibits a $DO_3$ lattice structure with a
chemical composition similar to Fe$_3$Al. The $DO_3$ structure is considered to be a BCC super-
lattice defined by eight cells as shown in Figure 7.
It has been found that in the NAB alloys with a high iron content, the $K_i$ precipitates formed
from the $\beta$ phase during the initial stage of the cooling and it grows to become large for the $\alpha$
phases formation. This is a proof that the $\beta + K$ region in Figure 6 extends upwards and moves
to the left in the alloys. Hence, the cast microstructure represents the large $K_i$ precipitates
bounded by the phase.

2.3.2 Kappa II Phase ($K_{ii}$)
The composition and structure of $K_{ii}$ is quite similar to the $K_i$ composition and structure, but
the $K_{ii}$ structure is an iron-rich dendritic particle, which is also referred as a globular or rosette
in shape. The particles are relatively small; about 5 µm to 10 µm in diameter. Unfortunately it
is very easy to misjudge the large particles of $K_{ii}$ as $K_i$ particles. The $K_{ii}$ precipitates also
exhibits some $DO_3$ lattice structures with a parameter of = 5.71 Å (Cook et al., 2003). In Figure
7, the $K_{ii}$ shows an “ordered version” of the disordered lattice with a high temperature $\beta$ phase
and; therefore, resulting in a dendritic morphology. The $K_{ii}$ precipitates have been found to
exist in the lamellar eutectoid region of the microstructure and formed within the temperature
range as the Windmastätten $\alpha$ phase.

2.3.3 Kappa III Phase ($K_{iii}$)
A globular or a nickel-rich lamellar eutectoid form of the K phase, i.e. $K_{iii}$ precipitates found
from the $\beta$ phase and ranging the formation around 580$^0$C. Hasan et al. (1982) have found that
the $K_{iii}$ phase has an NiAl composition and is related to the B2 structure with a lattice parameter
of = 2.88 Å . The lattice parameter is roughly half the lattice parameter of the $K_i$, $K_{ii}$ and $K_{iv}$
phases. It can be seen in Figure 8 that B2 structure for the NiAl is an ordered structure, which
is comparable to the $K_{ii}$ phases in its similarity to the “ordered version” of the $\beta$ phase. Thus,
it is expected that the $K_{ii}$ particles will act as a substrate for the formation of the $K_{iii}$ particles
(Cook et al., 2003).
Bell (1994) has suggested that the pro-eutectoid α is considered as an “active nucleus” for the nucleation of the β eutectoid decomposition into the α + K_{iii}. Hence, the precipitation of K_{iii} on the eutectoid α/β interface is the prime nucleation needed for the formation of the eutectoidal colony but the re-nucleation of α may not be essential for shaping the K_{iii} particles.

### 2.3.4 Kappa IV Phase (K_{iv})

The iron rich K_{iv} phase structure is labelled as the ‘Tee’ shape with fine equiaxed particles segregated all over α grains. The K_{iv} phase exhibiting the same Fe_{3}Al composition as presented in K_{i} and K_{ii} phases. The K_{iv} phase further presents a DO_{3} crystal structure with a lattice parameter of = 5.77 Å (Cook et al., 2003). As can be seen in Figure 8, a precipitate free zone (PFZ) is found at the boundary of α grains.

### 2.3.5 Martensite Beta (β’)

As the transformation temperature declines, the decomposition rate of the β phase also declines at a lower temperature. Thus, there is the possibility that the β phase decomposition is partially finished during normal cooling rates. As the decomposition slows and finishes, the balance of the β phase undergoes diffusion-less transformation to become martensite β (Oh-ishi, Zhilyaev & McNeilley, 2006).

---

**Figure 6**: Schematic of Phase Transformation Diagram (Source: Cuevas et al., 2002, pg. 19)
2.4 Concept of Friction Stir Process (FSP)

FSP is a relatively new technique that is able to alter the local surface microstructure, resulting in a fine grain microstructure and improvement in the material properties. Instead of joining material with FSW, the FSP technique processes the surface area of a particular material by using a stirring tool inserted in the surface that moves across the surface (Ni et al., 2010). In FSP, a wear resistant cylindrical tool with a small diameter pin protruding on a larger diameter shoulder shank is rotated at high speed and tilted at a controlled angle. Because FSP involves high forces in multiple directions, the sample is rigidly locked to the working table to restrain any movement. The tool is plunged into the metal surface slowly so that the tip generates enough heat to soften the contacted surface and allow the pin to penetrate. After the pin penetrates, the shoulder starts to contact with the surface and a larger heated area is created. The tool is then moved in a transverse direction across the metal surface and produces the required area of processing. There are two types of surface processing that are very popular among manufacturers and seldom used for FSP maintenance; spiral and square raster (Shafiei-Zarghani, Kashani-Bozorg & Zarei-Hanzaki, 2009) (Figure 8).

Figure 7: Lattice Structures of NAB Phases (a) BCC β (b) Kiii phase B2 (NiAl) and c) Ki, Kii, Kiv phase DO₃ (Fe₃Al) Structures (Source: Cuevas et al., 2006, pg. 18)
When the tool traverses the surface for single or multiple run, the friction between the tool pin and the metal surface generates instantaneous heat that softens and plasticises the allocated area. This softening reduces the resistance and increases the mobility of the pin to move more easily. During the stirring motion, a softened but yet solid state region of metal is created and stirred vigorously by the pin. Touching of the shoulder to the metal surface generates additional heat and; therefore, plasticises a larger cylindrical column around the inserted tool pin. The depth of penetration is controlled by referencing the touching shoulder to the bottom of the pin. The shoulder surface also serves as a restraining surface that the plasticised metal does not move in the upward motions (Shafiei-Zarghani, Kashani-Bozorg & Zarei-Hanzaki, 2009).

The stirring actions coupled with the downward force are able to generate a highly deformed area with a very fine microstructure. With the correct parameters in the deformed area, porosity or defects in the microstructure may be avoided. Hence, a defect free region with a fine-grained re-crystallized microstructure is formed using the FSP techniques. The active stirring area with an oval shape in the centre is known as the Stir Zone (SZ) and the middle is known as the Nugget (Fuller, 2006) (Figure 9). The recrystallised zone generated from the FSP was observed by Os-ishi et al (2004) who stated that the nugget zone microstructure and grain size comprises of equiaxed, fine, dynamically re-crystallized grains.
A severe distortion of the grain structure can be observed at the border of the SZ known as the Thermo-mechanical–affected Zone (TMAZ). Observation shows that the grain size of original and sub-grain boundaries seem to replace the fine equiaxed re-crystallization grains in the FSP nugget. The TMAZ grains were observed to be highly disoriented sub-grains relative to the grains of the NAB parent metal (Ni et al., 2010). Liu et al. (1997) stated that the FSW development was governed through slip deformation and the re-crystallization progress by the dynamic continuous re-crystallization, which induces a continuous increase in disorientation. The FSP and FSW exhibit many similarities that encompass intense shear deformation by the rotating tool pin and it is expected that they have comparable properties.

Field et al. (2001) have stated that FSW identified the existence of a severe and identifiable texture gradient through the nugget and the bordering region of the FSW weld. A significant shear direction aligned with the tangent of the rotating tool was detected.

By inspecting the micro structure using an optical microscope (OM), it can be seen that the grain size distribution is not uniform for any particular processing parameters. The grain sizes vary from the surface to the bottom as well as from the advancing side to the retreating sides of the tool. The variations of the grain size are expected to relate both peak temperatures and cooling periods experienced by the grains themselves.

The heat affected zone (HAZ) in the area adjacent to the TMAZ represents the material that is not plastically deformed but has been exposed to a thermal cycle during the process. This causes modifications of properties such as ductility, strength and corrodibility and can lead to grain growth.
The FSP surface experiences a very high strain rate and a very high temperature gradient and grain size, which differs in a short period and distance similarly to the HAZ of the fusion weld. The TMAZ in the FSP metal does not show any loss of strength or unwanted mechanical properties, which typically results from the high temperatures of a fusion welding process. When the melting temperatures are achieved during fusion welding, grain growth occurs and alloy distribution in the HAZ subsequently reduces its mechanical properties and corrosion resistance. On the other hand, since FSP is a solid-state process and the temperatures do not reach melting temperatures, grain growth is minimised in the specified area. Thus, the FSP affected area exhibits a finer microstructure than the parent metal with an increase in mechanical properties and subsequently improves the corrosion properties (Mahoney, 2005). The capacity to refine the surface microstructure of any material results in higher strength; therefore, the FSP also demonstrates other mechanical advantages. As the process uses a non-consumable tool that is reasonably wear resistant, it does not release any hazardous gas or produce any hazardous by-product. Being a solid state process (i.e. the material does not melt during the process), no grain growth decreases in strength or increases corrosion susceptibility as properties are usually occurred during fusion welding. There is limited noise emitted during the process and; therefore, the tool can be reused (Smith, Hinrichs, & Crusan, 2003). In general cases, no special preparatory requirement is needed (such as cleaning of the material before or after the process) and hence a greener environment is achieved.

### 2.5 Process sequence and tools

The process flow of the FSP was adapted from Mahoney et al (2005) and is illustrated in Figure 10, which comprises of four simple phase processes.

![Figure 10: FSP/FSW Overall Phases (Source: Khairuddin et al, 2012, Welding Processes)](image-url)
2.5.1 First Phase (Plunging Phase)
In the first stage of the process, FS tool is rotated to the required speed and slowly moved towards the surface of the metal until it touches. The plunging of the tool is achieved via a predefined feed rate or by using force control. As the tool pin touches the surface the force starts to increase at the tips of the tool and together with the tool rotation it generates extreme frictional heat that begins to plasticise the metal surface; thereby, allowing the tool to penetrate creates a high viscous metal at the specific area (Smith, 2000).

2.5.2 Second Phase (Dwelling Phase)
Depending on the type of metal, it is necessary to stop the plunging spot for a period (idling time) so that the required temperature of the surface and the tool is achieved. This action will reduce the yield strength of the metal and less resistance for travel speed (Smith, 2000).

2.5.3 Third Phase (Processing)
The main processing activity happens during the third phase. The heated tool travels along the surface of the metal while applying a certain amount of plunge force to soften the metal. The rotation of the tool creates a complex material flow of plasticised material from the advancing side to the retracting side. Thus, the pin’s incision as it passes is refilled with material (Smith, 2000).

To have a clearer view of the processing phase, the schematic process is presented (Figure 11).

- Traverse speed $v_s$
- Revolution speed $v_{rot}$ or $n$
- Tilt angle $\alpha$
- Plunge depth $E_t$ (or $E_{t,c}$ referred to tool centre point) in the controlled position
- Downward or axial controlled force $F_A$.

The traverse speed and tool revolution decisively determines the heat input to the material. Figure 12 shows that the tool is tilted at an angle from $1^0$ to $3^0$ to curb the workpiece material at the front side of the tool shoulder. Consequently, it will automatically increase the forging force at the plasticised metal and at the rear of the tool, which leads to improve mechanical properties of the processed metal (Mishra, Ma & Charit, 2003; Oh-Ishi & McNelley, 2004).
2.5.4 Fourth phase (Retracting)

Finally, at the end of the whole process, the tool starts to retract away from the work-piece, which shows a pin hole at the surface (Figure 12). The pin hole can be minimised if the tool is lifted from the material gradually (Smith, 2000). The method applies a dummy plate at the end of the run. When the tool retracts, the dummy plate will be cut off and the surface edge will be grinded.

Figure 12: Schematic diagram of Shoulder shape (a) spiral (b) dome (c) linear (d) scoop (e) concentric circle (Source: Zhang et al, 2012, pg. 252)
2.6 Advancing and Retreating Side

When the tool is rotating at a consistent speed, the processing path is divided into two halves that are known as the advancing and retracting sides (Figure 11). The advancing side is defined as the side that demonstrates the position of tool circumferential speed is in the same direction as the travel speed. The retreating side is the opposite travel velocity vector (Hovanski et al., 2015).

In order to attain a homogeneous fine grain microstructure over a large area, the tool pin must move over the metal surface in a predetermined pattern. According to Oh-ishi et al. (2006), the action will increase the refinement and hence improves the mechanical properties due to the additional exposure to strain and the thermo-mechanical cycle. A well-known technique for the rastering approach is to move the tool pin by half a pin diameter in the direction of the advancing side after each pass (Smith, 2000).

2.7 Advantages of Using FSP

The main purpose of using the FSP technique is to modify the microstructure at the specific area relative to the component’s applications. Thus, the component’s dimensional geometry will remain unchanged (Mishra & Ma, 2005).

Due to the high degree of plastic deformation in the localised area, the coarse and imperfect microstructure in the specific area is processed to a very fine grained, defect free and porosity free area. Also, the particles from the secondary phases are dispersed evenly throughout the metals. Hence, the mechanical properties such as strength, ductility, and fatigue failure and corrosion resistance are improved (Mahoney, 2005).

According to Mishra et al. (2005), FS processes can be characterised by the following attributes:

• Considerable material flow around the tool
• Low heat input into work-piece
• Refinement of the microstructure in processed metal
• Random rearrangement of grain borders in the stir zone
• Blending of surface material with subsurface metal
• Elimination of surface and near surface imperfections and porosity in castings
2.8 FSW/FSP Tools

FSW and FSP share the same type of tools. There are two types of tool: pin and pin-less types. A pin tool consists of a shank and shoulder with a concentric pin in the centre. A pin-less tool only has a shank without the pin (De Backer et al., 2012).

The tool geometry and the material of tool will establish the process quality, tool durability and tool cost. The tool material must exhibit abrasion resistance, adhesive and chemical wear and be inert to the environment of the material. The tool must also demonstrate high fracture toughness, particularly when the plunge force is applied to the surface and during the period of dwelling. During the FS process, the tool must exhibit dimensional stability as the temperature of the tool surrounds to the melting temperature of the work-piece. Moreover, high shear and compressive strength are needed to sustain at high temperature (Smith, 2000).

There are few functions of the shoulder in the friction stir pin as outlined by Misra et al (2005).

- Absorbing the heat generated through the extreme material deformation and friction.
- Protecting the process zone in order to maintain the plasticised material.
- Compacting the material flow at the rear surface and supporting the material consolidation.
- Supporting material flow.

Usually the shoulder will produce a large amount of heat mainly concentrated at a thin layer underneath the surface.

Basically, there are three types of shoulders.

- Flat shoulder
- Concave shoulder
- Convex shoulder

To maximise the material flow, the tool shoulders are designed with extra features such as grooves or nubs (Figure 13).
The pin is basically reflected as a conical shape as it assists the penetration stage during the plunging phase. Similar to the shoulder design, the pin exhibits features such as a thread to support the material flow (Figure 14). The pin diameter must be large enough to resist the traverse load but small enough to make the incision in the material. If the application of the FS can lead to a relatively serious wear or a possible broken pin then a threadless pin can be used (Mahoney, 2005).

2.9 Mechanical Properties Enhancement and Grain Refinement of NAB alloys

When the cast NAB alloys are subjected to FSP, subsequently the initial coarse microstructure of the cast NAB with grain size of 100 µm -150 µm is transformed to a finer microstructure and the presence of defects are eliminated. Nevertheless, the stir zones (SZ) are categorised by inhomogeneous structures and can be differentiated into four different sections: the fine Widmanstätten primary α phase in the surface layer, banded primary α and β' phases in the subsurface layer, equiaxed α and β' phases in the centre, and stream like α and β' phases at the bottom. The heterogeneous microstructure can be improved by altering the FSP parameters but can be totally eradicated with the investigated FSP parameters. The finished processed samples show significant improvement in hardness, tensile strength and ductility relative to the base metal (BM) (Soron, 2007).

With regard to the mechanical properties of the FSP NAB prepared with different parameters, Ni et al. (2010) reveals that there are five significant ways: the enhancement of the tensile strength and ductility relative to the BM, improvement of the Yield Strength (YS) and the
Ultimate Tensile Strength (UTS), and the elongation of the samples was improved. All the properties experience enhancement in the range of 20% to 31% from the BM alloys.

A similar report comes from Choksi (1989) using the Hall-Petch equation: \( \sigma = \sigma_0 + f d^{-1/2} \) who discovered that the yield strength and the hardness increases as the grain size reduces. This strengthens the finding of Ni et al (2010) that the mechanical properties are enhanced using the FSP techniques.

### 2.10 Robotic FSP

Currently, the FSP is dominated by a dedicated FSP machine as seen in Figure 14. The custom built machines are limited to certain assigned tasks for which they are devised. Due to this restriction, the FS machines are not suitable for a greater range of tasks typically required by industry where high productivity is needed (Culpan, & Rose, 1979).

A CNC or milling machine that exhibits three to five axes for the FSP process can be adapted, but they are also limited to a certain path of movement (i.e. in a linear line or circular path) and are typically incapable of tilting the FS tool. Even if the machine has the ability to exhibit 3D processes, the 5 axes machines still face a major disadvantage with special sophisticated jigs required to hold the work-pieces in its positions and to ensure that a clear and accurate penetration is achieved (Cuevas, 2002). To expand the usage of FSP, researchers have investigated a more flexible method of processing that is robot-based. Robotically applied FSP is effective for processing complex compound surfaces (Hasan et al., 1982) as well as having an unconstrained work volume when coupled with a moveable platform such as a linear bed.

![Figure 14: FSW/FSP Machine (Source: CASC, 2012)](image_url)
Industrial robots are more flexible and able to perform multi-tasking tasks of the industry (Figure 15). Another main benefit of the Robotic FS is the workspace setup and the ability to be integrated in the production line that permits changes of application and geometries without modifying the hardware (Jahanafrooz et al., 1983).

With the ability to manipulate six DOF, the robot arms are able to access more locations including difficult locations inside the working space. So, unlike gantry or milling machines, the industrial robots are more versatile and capable of producing complicated three-dimensional paths, which is essential for production of complex industrial components with 3D geometries (Bell, 1994).

The jointed arm axes of the robot are designed with an ability to rotate 360°, which is an added advantage of the robot to execute multiple processes in different workstations. For instance, a series of components could be arranged at different location in the workstations and time will be saved from loading and unloading the production of the plant (Cuevas, 2002).

As robots are widely used in industry, the investment and the training cost of the robotic operators should be reduced to make the whole process much more affordable. With the extensive usage of varieties of industrial robots, the developments of man with machine interfaces (MMI) are expanding rapidly and allow rapid familiarisation for more easier and user-friendly job execution. Furthermore, path programming is easily generated allowing optimisation of the operation time for the machine and; thus, increase productivity with robotic FS processes (Jahanafrooz et al., 1983).
2.10.1 Robot Constraints

To generate an economical and reliable manufacturing environment for robotic FSP, few obstacles must be overcome, including contact stability and the compliance issues. First and foremost is the lack of stiffness exhibited by the system relative to a conventional friction stir machine. The restricted stiffness is the outcome of the complexity of the robot configurations itself. In all the robot arm joints there is a servomotor, transmission axes and gearbox that account for the system’s compliance. The degree of flexion for each of these aspects is dependent on the robot’s positions, orientation and magnitude of force. At the typical process forces, it is inevitable that there will be slight deflections in the system when pressure is engaged during the process (Cuevas, 2002). The high pressure and forces during the process coupled with a relative lack of damping features in the robot arm leads to high and fluctuating deviations.

In FSP, the work-piece is clamped on top of the working table and hence the robot arm will exert a very large force vertically relative to the metal surface. Thus, it produces significant errors in the axial direction if no force control is used. Similarly, the accuracy in the traverse directions will be affected due to the high pressure force opposing the traverse directions (Bell, 1994).

The static deviations possess constant high force as lack of stiffness causes a resonance in the robot, particularly at the early stage when the tool initially engages at the surface. It can produce vigorous oscillations to the whole system and consequently the robot can be overloaded (Jahanafrooz et al., 1983).

The deflections have a detrimental effect on the positional precision of the process. The matter is an essential consideration if the operator wishes to minimise the surface defects due to inadequate heat input and inappropriate tool penetration depth (Bell, 1994).

A vital issue that is faced by the industrial robots is the low force as they are capable of producing force during the process. Because of this, the tool is unable to generate enough heat. This is usually compensated with lower travel speeds and by increasing the rotation speed. However, the lowered forces limit the feasible stir zone depth (Oh-Ishi & McNelley, 2004).

To establish a consistent process and desired FSP results, large spindle-motor torques and high axial forces are vital prerequisites. Spindle motors that can deliver the required torque may have a large structure and are sometimes difficult to adapt to the robot’s tool attachment. This can lead to a greater distance between the robot’s joint and the FSP tool and results in an
upsurge of torque, robot arm deflection and an increased potential for resonance (Jahanafrooz et al., 1983).

2.11 Failure Mechanism
2.11.1 Background
As cast NAB is widely used in the oceanic environment, two critical factors have a severe impact in the service life. These factors entail corrosion and cavitation erosion effect. Hence, the usage of the components studies must be conducted in order to optimize the material loss from these factors that could be minimise and controlled.

In this section, the literature is prepared to explain the basics of corrosion and cavitation degeneration mechanism. The discussion of corrosion will include the chemical effect that happens during the oxidation process as well as the mass loss calculation. Similarly in the cavitation section, explanation relating to the shockwave impact and its relation to the mass loss at the NAB surface is presented.

2.11.2 Corrosion
Corrosion of metals and non-metals takes place because of gradual environmental interaction on the material surface. The structures and facilities of different materials are affected by this interaction. Even the ambient air laden with moisture and oxygen can start this process and known as rusting on steel surfaces (Field et al., 2001). Corrosion affects the microstructure, mechanical properties and the physical appearance of the materials. Rusting and other types of deterioration reduce the capacity of pipelines and equipment, resulting in loss of output as well as loss of equipment or even life. Hence, anti-corrosive coatings are used to combat the corrosion damage to critical structures and equipment.

The NAB displays excellent corrosion resistance characteristics, which make it a preferred material for marine components such as impellers, propellers and valves. However, it still suffers from various corrosion and erosion as it affects the surfaces badly especially near the weld runs and submerged regions. Corrosion, crevice corrosion, selective phase corrosion, corrosion erosion, stress cracking, cavitation and fatigue corrosion all contribute to the failure of NAB in the vigorous sea applications (ASTM_G133, 2010). Therefore, more research should be done in this area to further reduce the effect of failure at the alloys.
2.11.3 Electrochemical and Crevice Corrosion

All metals exposed to the environment will exhibit different thicknesses of oxide layers that define the level of corrosion resistance (Schüssler & Exner, 1993). This layer varies and sometimes determines the rate at which corrosion can occur. For NAB, a rapid formation of its oxide film is not disposed to localised breakdown and consequent pitting. This is due to the presence of high aluminium contents in NAB alloys, which induces a layer of aluminium oxide ($Al_2O_3$) covering the surface and preventing further oxidation. Thereby, NAB alloys exhibit a high corrosion resistance in either flowing or stagnant seawater.

Nevertheless, all metals are subjected to corrosion except gold, especially in an acidic environment and even with a high corrosion resistance. NAB alloys will also be affected by the environment. Many researchers have documented the corrosion behaviour of cast NAB in seawater (Takaloo et al., 2011; Kear et al., 2004; Klassen, Roberge & Hyatt, 2001). Kear et al. (2004) have reviewed the corrosion of copper in chloride media and shows that the corrosion process is complicated by the formation of a surface film or oxide layer. Similarly, with copper, it can be determined that the electrode process of NAB is also anodic dissolution of copper and it may form cuprous dichloride anion simplified as in equation 1 and the oxygen reduction as a cathodic process as in equation 2:

\[
\begin{align*}
Cu + 2Cl^- - e^- & \rightarrow CuCl & (1) \\
O_2 + 2H_2O + 4e^- & \rightarrow 4OH^- & (2)
\end{align*}
\]

Cuprous oxide ($Cu_2O$) is formed by a dissolution process [38] as follows:

\[
2CuCl_2^- + 2H_2O \rightarrow Cu_2O + H_2O + 4Cl^- & (3)
\]

The presence of aluminium increases the corrosion resistance of copper in seawater and also promotes the formation of an oxide layer. The layer is then recognised by the oxide colour (whitish colour) of Al reacted with chloride and followed by hydrolysis to produce an aluminium hydroxide layer (Wharton, & Stokes, 2008):

\[
\begin{align*}
Al + 4Cl^- & \rightarrow AlCl_4^- + 3e^- & (4) \\
AlCl_4^- + 3H_2O & \rightarrow Al(OH)_3 + 3H^+ + 4Cl^- & (5)
\end{align*}
\]

Schussler et al (1993) report that the proactive oxide layer is comprised of Al and Cu oxides ($Cu_2O$ and $Al_2O_3$), which contributes to the good corrosion resistance of the NAB samples. The oxide layer consists of Cu in the outer region and Al is rich in the region adjacent to the BM. The oxide layer acts as a barricade and hinders the movement of ions across the corrosion product, especially the sustainable proactive layer that forms quickly on the NAB surface.
Wharton et al. (2005) describe the corrosion characteristics of cast, wrought and heat treated NAB alloys in seawater. Wrought NAB contains very little β and K_{III} phases. Applying sufficiently high temperatures during heat treatment minimises the corrodible β phase and increases the density of the fine K phase precipitates in the phase region. Their report describes how the corrosion rate is influenced by metallurgical composition, processing history and surface roughness.

In another report, Wharton et al. (2008) claim that NAB experiences crevice corrosion, which occurs in confined areas where fluid can access but cannot readily flow. For NAB, crevice corrosion starts occur near the eutectoid regions with only a small attack of the Cu rich α phase within the region of the α + K_{III} eutectoid. Due to the nature of the K_{III} phase, it was possible for crevice corrosion to reach a depth of 80 µm in 30 days of testing. α grains showed minimal attack because of the presence of a large portion of K_{III} phase near the eutectoid region.

2.12 Corrosion Measurements

2.12.1 Weight loss measurement

The easiest method to calculate the corrosion rate of a particular metal is by subjecting the metal to a corrosive medium such as sea water, or liquid NaCl₂, and evaluating the weight loss of material as a function of time. Even though it is a very simple analysis, there is no easy way to extrapolate the outcome to forecast the lifespan of the material (which is a subject for research). Furthermore, some corrosion happens with no significant mass loss. In a pitting corrosion, the loss is so minute that it is very hard to distinguish the loss by gravimetric techniques (Klassen, Roberge & Hyatt, 2001).

The conventional measuring technique includes the electrochemical process, which is an alternative method to determine the corrosion rate. Quantitative and direct measurement of the corrosion rates can be obtained from a simple electrochemical calculation such as a linear sweep voltammeter (LSV). As stated, most of the corrosion activities are electrochemical in nature and comprise the reaction at the surface of the corroding metal. Hence, the electrochemical experiments techniques can be used to determine the corrosion mechanism and forecast the corrosion rates. Using the ASTM G31 standard, calculations can be made for the corrosion rates and equivalent weights (ASTM_G31_REV1, 2012).
2.12.2 TAFEL Plots

Another option for weight loss calculation could be done using a Tafel polarisation graph. The equation is related to the electrochemical kinetics with the rate of an electrochemical reaction to the over potential. The graph will directly stimulate the corrosion current relative to the corrosion rates. This procedure is quite precise to determine the weight loss within the process. The Tafel constant $\beta_A$ and $\beta_C$ can be obtained from the Tafel plots, and can be used together with the Polarization Resistance data to compute the corrosion rates (Figure 16, Figure 17).

The values obtained from the graph can be very useful for further study of corrosion inhibitors (Baboian 2005).

A Tafel graph is constructed on a metal sample by polarizing the sample to about 300 mV anodically (positive going potential) and cathodically (negative going potential) known as corrosive potential. The potential is not necessarily needed to be scanned but also can be ‘stepped’ if required. The outcome is plotted on a logarithmic scale. The corrosion current, $i_{corr}$, is acquired from the Tafel graph by extrapolating the linear part of the curve to $E_{corr}$. Thus, the corrosion rates can be computed from the $i_{corr}$ by using the equation 6:

$$\text{Corrosion Rate} \ (\text{mpy}) = \frac{0.13 \ i_{corr}(E.W)}{d}$$ \hspace{1cm} (6)

Where E.W. is the Equivalent Weight (g) of the corroding species, $d$ is density (g/cm$^2$) and $i_{corr}$ is corrosion current density (µA/cm$^2$)

![Figure 16: Excitation Waveform for Polarization Resistance (Source: Princeton Applied Research, 2010, pg. 8)](image-url)
2.13 Cavitation

Reduction of performance has always been a topic widely discussed in the world of engineers because it directly affects the cost of maintenance and productions. For marine components, cavitation and erosions have been one of the major factors contributing to lack of performance. One of the main reasons is due to the cavitation erosion effect; especially on components that are placed in a corrosive media such as the marine environment (Levy, 1995).

Cavitation erosion is defined as degradation of mass at the surface without the presence of abrasive particles in a liquid. It is often caused by an impinging liquid, abrasion by slurry, bubbles or droplets (Levy, 1995). Figure 18 shows the effect of cavitation and erosion at a service propeller.

The mechanism of cavitation erosion includes repeated vibratory shock waves generated by the collapse and rebound (Figure 19) and impingement of micro-jets of liquid through the destructions of a non-symmetrical cavity (Figure 20) (Mills, 1987).

The first form of liquid erosion is the high velocity of water droplets impinging on the surface continuously and eventually removing the surface material as well. The combination of cavitation erosion and liquid impingement has the same hydrodynamic forces on the metal surface and; thus, a continuous degradation of material from the surface happens by the repetitive impulse of water jets bombarding the surface area. Therefore, the metal surface experiences both effects, which leads to a visible cavitation erosion in the short term and eventual catastrophic effect in the longer term. Figure 16 shows the effect of cavitation in early stage.
**Figure 18:** Effect of cavitation of ship propeller (Source: Johneck, 2011, pg. 1)

**Figure 19:** Shock waves produced by collapse and rebound of a cavity (Source: Hammitt, 1980, pg. 68)

**Figure 20:** Impingement of liquid micro-jet (Source: Soyama, 2005, pg. 1)
The second type is the liquid impingement. The repetitions of millions of vacuum bubbles collapsing on the components surface will induce high pressure micro-jets striking to surface and thus resulting in cavitation wear. These cavities can disperse the energy at each individual collapse. When the bubbles crumble in the liquid, the process will transmit the momentum to the surface causing significantly high pressures around the crumble areas. Thus, the cavities that explode closest to the surface will cause severe damage to the metal surface. Then a non-condensable gas in the bubble will endure and again the compression causes a rebound and some oscillatory nature for some of the cavities (Margulis, 1995).

2.13.1 Erosion Rate

Erosion rate is defined as a continuous loss of material from a solid surface due to the exposure of cavitation effect over a period of time (Wharton et al., 2005). Figure 21 shows a typical erosion rate graph of most metals. In the initial stage, there is no weight loss. This stage is labelled as the incubation period. During the incubation period, there is only random pitting and mostly removal of oxides at the surface of the metal. The second stage is known as the acceleration stage. In this period, the mass loss is significant and easily detected. The erosion rate accelerates to a maximum with fracture and pitting apparent. In this stage, the metal surface experiences a significant bombardment of shockwave or micro-jet impingement activity results a coarse surface, which can be easily seen by the normal eye. As the experiments continue, the erosion rate will be consistent and it is labelled as a maximum rate stage. The lessening of the weight loss happens because the rough surface traps the liquid between the edges and prevents the vibratory impact. The erosion rate is dependent on several factors such as intensity of the vibratory wave, the mechanical properties of the material and the surface conditions (Margulis, 1995).

![Figure 21: Schematic Representation of Erosion Rate with Exposure Time](image)
2.13.2 Estimation Method for Cavitation

The cavitation erosion method of assessment was adapted from the ASTM G32-10 (Standard Test Method for Cavitation Erosion Using Vibratory apparatus) (ASTM G32-10, 2010), which covers the method of setting up the experiment, material loss calculation and graph plotting method. The mass loss against time curves can be plotted by measuring the weight of the sample with an accuracy of 0.001 grams at the start of the experiment and over a staggered period of 2 hours separation for the whole experiment. The standards also specify the temperature, the amplitude, and the frequency of the vibratory waves, volume of liquid and the immersion depth. Two types of liquid that are usually used are distilled water and 3.5% Sodium chloride (NaCl) solutions.

2.14 Discussion and Summary of literature review

The literature review has shown that cavitation erosion and corrosion effect is an inevitable effect as faced by marine component that could lead towards total failure of marine components. Mishra et al (2005) have found a new technique known as FSP and proved that the method was able to improve the surface properties and thus improving the service life of the alloys.

For a dedicated FSW machine and symmetrical shape components, the properties attributes are easily achieved. To apply the process at non-symmetrical shape and at different angles, the machine is not applicable at all. Thus, an alternative method by applying articulate robot is essential to finish off the given tasks. Robotic arms are widely used in the industries because of its ability to manipulate many processes at different angles required by the user. The downside of the robotic arm in the axial force that could be supplied is limited.

A CNC machine was selected to be operated at a low force and to obtain comparable results to validate the outcome and to confirm its validity. The selected CNC machine needs to carry out the task at similar parameters as the articulate robot and the results obtained were analysed.

Three set of parameters were needed to be varied that is the plunge force, rotation speed and the traverse speed. By varying these parameters, different entities of samples were obtained. The samples were sent to the micro Vickers tester for hardness testing to the vibratory apparatus for cavitation erosion testing and potentiostat analyser for the corrosion testing.
CHAPTER 3: FSP SETUP AND METHODOLOGY

3.1 Background

Multi-tasking is a very important aspect in the manufacturing process. The ability to perform a particular task in eccentric condition is a primary requirement. Performing a typical FSP will generate high friction and also need a high plunging force at different angles or uneven surface making the FSW/FSP machine impractical. The ability of undertaking varieties of jobs could be done by an articulate robot but with certain limitations such as the inability to produce high plunging force. Therefore, the articulated robot is considered as low force equipment due to its ability to generate around 2000 N.

As this study is related to the use of low force FSP, an articulate robot will be used for the purpose. With the inability of a proper FSW machine, a CNC machine is chosen to carry out the task. The function is to compare and; thus, validate the results of the robotic FSP. For the comparative purpose, the same parameter will be applied to both machines.

The FSP experimental setup was divided into two sections. Section 3.2 is the robotic FSP to stimulate the uneven surface of cast NAB component. For the purpose of experimentation, a flat surface sample was used. The processing of the surface was conducted with an industrial robot (ABB IRB 6660) in the Manufacturing Research Laboratory (MRL). A detailed preparation and setup of the robot are described including the detail of the software, the tools, and runs.

Section 3.3 explains the FSP setup by using a CNC Milling Machine. Both setups are prepared and carried out at the University of Wollongong (UOW), Australia. A detailed review is given regarding the fixture, the CNC adaptation technique, the software and the runs.

3.2 ABB IRB 6660 Robot

The ABB IRB 6660 robot was chosen to carry out the FSP on the samples. Figure 22 has shown the robot arm with a serial manipulator with six DOF. The robot design was quite robust in welding, machining, packing, handling and other uses. Hence, the structure is relatively rigid, stiff and heavy in order to cater for both high and low-frequency vibrations. The robot was equipped with an IRC robot controller. The programming can be conducted offline using the IRB’s attendant software ABB Robot Studio 5.14.01. This utilizes a high-level programming language called RAPID and is the manufacturer-specific recommendation (Kurt, Uygur & Cete, 2011).
3.2.1 Force/Torque Sensor
In order to measure the forces acting on the system, a Force/Torque (F/T) sensor was mounted on the last joint of the robot arm. The signal was recorded by an ATI F/T sensor and saved in the ABB Test Signal Viewer Version 1.4, which comes together with the ABB Robot Studio 5.14.01 software. Figure 23 shows the image of the force sensor and the second image (Figure 24) show the assembly of the sensor and motor to the robot arm. The sensor was able to record a maximum force up to 2500 N and torques up to 400 Nm (ABB, 2014).

![Figure 22: ATI Force/Torque Sensor](image)

![Figure 23: Assembly of ATI F/T Sensor and Shuner spindle With Test bed](image)

3.2.2 Motor Spindle System
To provide spinning motion to the tool bits, a powerful motor was required for the robotic FSP task. Therefore, the motor-spindle-system used a standard ISO 3 kW power rated SUHNER. The motor spindle system, called a BEX 35 model, is illustrated in Figure 23. The rotational
movement was transmitted via a belt-drive to a high-precision turning spindle with a spindle concentricity of 0.01 mm. The latter was located at the end of the robot joint (ATI, 2014). Because of lower requirements for rotational, the FSP tool is mounted using an ISO 40 tool holder and the matching collet chucks.

For the spindle speed, the control of the motor and gearbox was powered by a Zener MSC3 3-phase AC drive. The drive has an analog input, which can be configured to control the rotational speed from 0-4000 Rpm. By adjusting the speed controller, the required speed was achieved easily and counterchecked using a tachometer.

### 3.2.3 Robotic FSP Test-bed

The robotic experimental setup was very simple because it only uses a table vice to clamp the sample (Figure 24). During the FSP cycle, LabVIEW software was used for data collection of the underlying variables of a parameter such as force, temperature and traverse speed. Figure 24 shows the schematics and how the system was connected via various signal paths.

![Schematic of Signal flow for Robot]

**Figure 24:** Schematic of Signal flow for Robot

### 3.2.4 FSP Tool pin

A set of tools from Beijing FSW Company was used for the experiments. The tool can be used for FSW processes as well as FSP processes. The tool could be used for FSW/FSP ferrous and nonferrous alloys and can withstand temperatures of 900-1000°C at high x- and z-axis loads, produce consistent weld properties, and maintain a high abrasion-resistance. The tool consists of a polycrystalline cubic boron nitride (PCBN) tip and a tungsten carbide shank (Ahmad et al., 2014).
Tipless tools were used in the feasibility experiments, which produced some impressive results (Ahmad et al., 2014). As observed from the schematics, the pin is 2.52 mm wide and 2.2 mm deep; therefore, the stirring area was relatively small and multiple side-by-side runs were needed to obtain a larger surface for the related experiments (Figure 25).

![Figure 25: Dimension and FSP Tools With Pin](image)

### 3.2.5 LabVIEW Program

LabVIEW software was used to monitor the variable parameters during the experiments i.e. the lateral forces, the plunge forces and the traverse speed. A LabVIEW program is able to execute custom control strategies in the program and stipulate real-time feedback about the system to the user.

### 3.2.6 Program Structure

The software was designed using parallel loops that were timed depending on the refresh rate. All the main tasks run in a different loop but allow tuning for the priority structure of the overall program. There were seven different segments: force sensor, plunge offset, traverse speed, spindle speed, control system, data logging and robot network connection. Each segment was divided into two parts, Graphical User Interface (GUI) components, and processing
components. The GUI loops were updated every 200 ms while the critical loops were updated every 10 ms (Figure 26).

![Figure 26: Outlook of the LabView Program](image)

### 3.2.7 Operator Interface

GUI was designed to be an efficient data collection medium while the experiment was ongoing. Figure 27 illustrates the outlook of the GUI data controls. The setup of the inputs is located on the left, commissioning for outputs is located in the middle while the log file outputs, robot connection and control loop interface on the right.

![Figure 27: LabVIEW Graphic User Interface](image)

### 3.2.8 Design of experiment (DOE)

To achieve a systematic investigation of the impact of different process parameters, the surrogation functions for the objective variables must be approximated. Utilisation of the Design of Experiments (DOE) for the optimisation is described in this section. The DOE defines the sampling plan for the process parameters. A starting set of parameters and a range
of their values were selected based on the experience gathered during the preliminary studies. Even for a moderate number of parameters, a full factorial design quickly leads to a large number of experiments, as observed in Equation 7 (Simpson, 2005).

\[ n_{exp} = l^{n_{para}} \]  

(7)

where

- \( n_{exp} \): Number of required experiments
- \( l \): Number of levels of a parameter
- \( n_{para} \): Number of parameters

\[ n_{exp} = l^{n_{para}} \]

\[ n_{exp} = 4^2 = 16 \]

No of experiments required =16.

The evaluation of the test results is very time-consuming, as it requires preparation and examination of samples; therefore, a fractional factorial design was applied. For the DOE, orthogonal arrays (OA) were utilised. Orthogonal arrays are strongly recommended by Taguchi et al. (1987) as they are designed to minimise the interrelation of responses and to focus on the main effects.

### 3.2.9 Robotic Endurance Test

Before conducting the designated experiment, the articulated robot needs to be tested in order to ensure its ability to cater the high rotation and high force induced during the FS process. A set of parameters was designed with a descending value starting from a maximum 4000 RPM, 3200 RPM, 2500 RPM and finally 1000 RPM. In order to maintain other parameters such as traverse speed, plunging force, and tilting angle at a constant value, the maximum value was chosen to refer to the study done in which significant improvement of properties was achieved (Suhner, 2014).

The GUI was programmed to record all the related parameter. With the assistance of ATI Omega 160 force sensor, the axial force produced by the robotic head was able to be detected. At 4000 RPM, the force sensor detected minor fluctuation of force but it is considered to be in the acceptable zone (Figure 28). When the speed reduces to 3200 RPM, a larger force fluctuation graph was observed but still considered to be in the safe working condition (Figure 29). But at 2500 RPM, the force sensor pick up a significant fluctuation of force exerted by the robot head and considered to be severe (Figure 30).
Finally, the plunging force was set at 1750 N, the speed of rotation was 1000 RPM and the traverse speed was 0.5 mm/s, the force sensor picked up very significant and vigorous vibrations during the penetration and the early stage of the friction stir causing the joints to shake severely. The experiment was stopped immediately for the safety of the machine and the operators.

Taking into account the force fluctuation and safety consideration, the robotic FSP proposed safe working zone in the area of 3000 RPM to 4000 RPM. Lowering the rotation speed could jeopardize the functionality and accuracy of the articulated robot.

Figure 28: Parameter 4000 RPM, Force 1750 N and Traverse speed 0.5 mm

Figure 29: Parameter 3200 RPM, Force 1750 N and Traverse speed 0.5 mm
3.2.10 Types of FSP Runs

Using the RAPID software, a program was prepared for the robot to start the execution by rotating the tool and hence approached the initial point of the sample surface. The plunging starts at the same spot and; thus, elevating the tool that surrounds temperature to plasticise level. This process enables the tool to travel in traverse direction without any major resistance and hence eliminating the possibility of damaging the tips.

Few sets of runs and visual observation were accomplished as the acceptable runs exhibit a full penetration of the pin and the shoulder rubbing against the surface. If lack of penetration and the run does not exhibit full shoulder engaging the surface, then the run will be rejected (Figure 31). Few selected parameters were selected for the cavitation erosion and corrosion tests. To maintain the parameters, multiple runs were done by overlapping the runs with a 5 mm shift. Figure 32 illustrates an example of multiple runs. The multiple runs will then be measured and cut into the segment for cavitation erosion and corrosion test.

![Figure 30: Parameter 4000 RPM, Force 1750 N and Traverse Speed 0.5 mm](image)
Due to the unavailability of an authentic FSW machine, a HAAS VF 2 with 3-axis vertical CNC milling machine was used for the replacement and experimental setup for the FSP. The CNC was chosen because the rigidity of the machine is 60 times better relative to the articulate robot (Veem, 2011). The variable experimental parameters could be programmed using the G-Code routine in the machine. Using the linear interpolation G coding, the tool was set to travel in a 3 degree slanting motion relative to traverse motion of the table.

The limitation of the CNC mill was reflected from the tool as it could not be tilted relative to the work table axis. To accommodate this limitation, the work-piece was mounted at an inclination of $3^\circ$ and the travel path of the tool was modified to follow this inclination. This
simulated the same inclination effect of tilting the tool. The CNC milling machine was interconnected with LabVIEW, the Spindle Motor and 3 Axis force sensor so that all the required data could be accessed and recorded. The schematic of the sensors connections is given in Figure 33.

3.3.1 CNC FSP Runs

A CNC mill G code program was prepared following the parameter set (Figure 33). Each run was set to 40 mm long and 20 apart and was done on each variable parameter. Opposite to the Robot FSP, the force inserted to the cast NAB during the CNC FSP was unable to be detected. The reason behind this limitation is that there is no force control in the CNC machine. A force sensor was placed underneath the backing plate and its function was only to detect how much force was plunged to the cast NAB. The signal from the sensor was detected via a LabVIEW program connected to the force sensor and hence a graph of force vs time was able to be plotted.

Figure 34 shows the assembly of the backing plate and the force sensor.

Figure 35 shows the finished runs done by the CNC. The runs were then cut using a CNC Wire-cut for microstructural analysis and corrosion testing. For cavitation erosion test, multiple runs were done and the parameter selected as shown in Tables 2 and 3.
Figure 34: Assembly of Test Plate, Insulator, Holding Plate and Force Sensor

Table 2: Table of Parameter

<table>
<thead>
<tr>
<th>Spindle speed (RPM)</th>
<th>1000</th>
<th>1000</th>
<th>1000</th>
<th>1000</th>
<th>Robot Application</th>
<th>CNC Application</th>
</tr>
</thead>
<tbody>
<tr>
<td>Traverse speed (mm/s)</td>
<td>0.5</td>
<td>1</td>
<td>1.5</td>
<td>2</td>
<td>N</td>
<td>Y</td>
</tr>
<tr>
<td>Traverse speed (mm/s)</td>
<td>0.5</td>
<td>1</td>
<td>1.5</td>
<td>2</td>
<td>N</td>
<td>Y</td>
</tr>
<tr>
<td>Spindle speed (RPM)</td>
<td>3000</td>
<td>3000</td>
<td>3000</td>
<td>3000</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Traverse speed (mm/s)</td>
<td>0.5</td>
<td>1</td>
<td>1.5</td>
<td>2</td>
<td>Y</td>
<td>Y</td>
</tr>
</tbody>
</table>
The FSP equipment in this thesis is explained in this section. The robotic system represents the low force technique along with uneven surface and CNC machine has been used for the evaluation of results. The study of the results will be conducted in chapter 5.

After friction stirring the samples, the samples will be cut to the required size. The samples were then mounted, polished, hardness tested, micro-structural examine, corrosion tested, cavitation tested, and examined with Atomic-Force Microscopy (AFM) and SEM.

3.4 Summary and Conclusion

Table 3: Cavitation Table

<table>
<thead>
<tr>
<th>Spindle speed (RPM)</th>
<th>1000</th>
<th>2000</th>
<th>3000</th>
<th>4000</th>
</tr>
</thead>
<tbody>
<tr>
<td>Traverse speed (mm/s)</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
</tbody>
</table>

Figure 35: CNC FSP Test Runs
CHAPTER 4: EXPERIMENTAL METHODOLOGY

4.1 Introduction
As cavitation erosion and corrosion plays a major role in component degeneration, preventive measure must be taken so that the effect can be controlled or minimised. In order to have this knowledge, thorough studies must be undertaken so that industrialist would use this platform to improve the service life of components.

To achieve the objectives, overall methodology of testing and the NAB process was described. The discussion starts with the description of the material used for robotic and CNC process. Furthermore, the method of cutting, grinding and polishing the specimens for the hardness test, cavitation erosion test and corrosion test is discussed. The samples were prepared independently for each related test.

The experiment consists of hardness testing, cavitation testing and corrosion testing was explained. After testing, the samples were sent to the instruments such as SEM, AFM and Energy Dispersive Spectrometer (EDS) for further analyses.

The first step is the cross section segments in which samples were sent for hot mounting and polishing. The mounted samples are examined using the (OM) and SEM. Images are captured and used for analysing where the samples were sent for hardness testing.

A detailed description of the corrosion experiment was described. The experiment was done using a NaCl₂ stimulate sea water. A potentiostat apparatus was used to obtain the necessary information and software was used to do the necessary calculation and graph plotting.

A detailed description of the cavitation erosion experiment was featured. The same solution of NaCl₂ was used again to stimulate the sea water. The experiment was divided into several sections to enable the progress of the cavitation erosion for monitoring and recording. For the gradual roughness progression, an AFM machine was used for the weight loss and accurate digital weighing scale was used. This chapter also explains the usage of AFM with a detailed methodology of SEM.

4.2 Material Used
A cast NAB specimen with the chemical composition of 80% Cu, 8.8% to 9.5.0% Al, 4.0% to 5.5% Fe, 0.75% to 1.3% Mn, 4.5% to 5.5% Ni (which follows the DSTAN 2000 Australia defence standards for marine applications) was used for the experiments. The chemical analysis was confirmed by Veem Ltd, WA, and Australia (2011). With the assembly (Figure 36), the
cast NAB was processed with the robotic arm and the CNC machine. The finished NAB slab was then metallurgically analysed.

4.3 Samples Preparation

The FSP NAB sample was cut using a CNC wire cut to the preferred size using an Accustom 50 precision cutter for numerous tests. The cutter uses a non-ferrous 5 inch Silicon Carbide (SiC) with continuous flow of coolant to carry out the task. In order to reduce the risk of cutting wheel, the speed was set to 4000 RPM and the traverse was set to 0.5 mm/s.

For hot mounting, the samples were cut cross-sectional and cleaned with ethanol and dried. Stuers Citopress-20 machine is used for the hot mounting task. A releasing agent powder was placed on top of the ram and the samples were placed in the centre of the ram. Two pieces of sample must be placed facing each other to minimise waviness at the surface. In order to do some checking with SEM, the mounted samples must exhibit electrical conductivity. Thus, combination of Polyfast and Durofast is suitable for edge preservation. The samples and resin is then locked inside the housing chamber. A temperature of around 180°C and a force of about 250 bars are applied during the embedment of the specimen. Water cooling is used to obtain the shortest possible mounting time. After 5 minutes, the samples are ready to be polished (Al-Hashem & Riad, 2002).

4.3.1 Polishing

Mechanical polishing is the most common method for preparing materialographic specimens and for microscopic examination. The specific requirement of the prepared surface is determined by the particular type of analysis or examination.

In order to capture the desired image, scratches must be removed from the sample surface. The hot mounted samples are sent to be ground and polished using a Stuers Tegra system, which comprises a TegraPol-21 grinding and polishing table, a Tegraforce-5 specimen mover and a TegraDoser-5 for automatic dosing. The equipment was set to run for 5 minutes, rotational speed of 150 RPM and axial force of 30 N for each polishing process.

A maximum of 6 samples can be mounted on the specimen holder at one time. The initial process is to ground the samples using 220 SiC paper with water lubrication. The second stage uses a MD-Largo 9 µm cloth 0 with Stuers DiaDuo-2 lubricant. The third stage uses a MD-Dac 3 µm cloth with Stuers DiaDuo-2 lubricant. The fourth stage uses a MD-Plus 1 µm with Stuers DP-Suspension as lubricant. Finally the samples were polished with MD-Chem 0.5 µm
and with a mixture of Ammonia and Hydrogen Peroxide solution. Details of the polishing steps are given in Appendix 1.

To preserve the surface from contamination and oxidation, the surface need to be etched after polishing. A solution of 5g FeCl$_3$ + 2 ml HCl + 95 ml C$_2$H$_5$O was prepared to use the etching of the samples. The surface was dipped inside the solution and shakes for a few second until the surface turn to a slightly pale colour and straight away rinse with water and dried. The purpose of etching enhances the contrast on surfaces in order to visualize the micro or macrostructure. Etching in materialography exerts a controlled influence on the surface profile or optical properties at grain boundaries, phases or grain surfaces; thus, enabling microscopic inspection and additional use of optical filters in the microscope.

4.4 Hardness

After etching, the mounted samples clearly show the overall shape of the FSP area. The SZ regions of interest show the hardness measurement. An overall surface hardness measurement was carried out to achieve more accurate results. Thus, a Durascan Automatic Micro Hardness tester with Vicker indenter was used to measure the hardness of the cast NAB and processed samples (Preece, 1979). The tester is equipped with eco-flow software for the computational calculation. A test load of 200 gram force (gf) was selected and a grid of 9 points was used at the nugget surface and other allocated surfaces.

The polished sample was placed on the movable stage and secure with a blue tag to prevent it during the testing. The head is lowered very closely to the surface to obtain the reference point before testing. Using the software, a matrix of 9 point (Figure 37) was selected with a clearance of 0.2 mm between each point. Using the camera, the surface was focused to obtain a sharp image. The cursor was set at the top left of the box for the first point and automatically the rest of the point will follow. By pressing the start button, indenter will automatically move to the starting point and initialize the indenting. After that the software will generates an excel type of report stating the value of all the point. From the results, the average hardness can be calculated.
4.5 Optical Microscope

In order to analyse the microstructure, a Leica DM 4500 model OM is needed to magnify the surface image. The apparatus automatically detects the grain boundary using contrast method and objective lens. This provides valuable consistency and reproducibility for the research. Hence, the details of the grain structure could be examined carefully.

After starting the observation, the sample was placed between the centre of the microscope stage. The halogen lamp is switch on and adjusts manually to the requirement of the observer. The polarisation contrast light is adjusted using the polarise slit. Using the lowest magnification
objective lens (10 times magnification), the image is focused using the focusing knob and the fine tune knob. When a sharp image visualise then a higher resolution of objectives lens must be used. There are 50 times, 100 times and 200 times magnification of the lens. As the bigger resolution objective lens is used, the glass is getting very close to the sample. Precautions must be taken to ensure that the lens are not touching the samples; thus, scratching the lens. When a sharp image is visualized and all the required details are obtained then grain boundary, grain size and different phases under the parameter are measured through measurement ruler. The image is then captured using a digital camera with Leica Application Suite (LAS) as a part of an integrated system solution.

4.6 Corrosion Experiment

One of a primary failure of cast Nab faced when subjected to sea water is corrosion. With the acidity of 8.1 pH (Al-Hashem & Riad, 2002) and the vigorous current absorbed by the components, the tendency of failure is very high. Thus, a corrosion experiment must be conducted at the FS processed surface to determine the improvement achieved as compared to the as-cast NAB.

4.6.1 Corrosion Sample Preparation

The FSP samples were cut to a size of 20 mm X 30 mm X 5 mm with the FSP run located in the centre. The samples were then hand polished using the American National Standards Institute (ANSI) silicon grit paper of 600 (15.3 µm) then 800 (6.5 µm) and finally 1200 (3 µm) producing a surface roughness (Ra) equivalent to 20 nm (Leica, 2014). The samples were washed with ethanol and rinse using distils water and kept in the desiccators to minimise any contaminations.

4.6.2 Corrosion Experiment

A guideline to conduct the corrosion immersion test was obtained from (ASTM G31-Rev 1) (ASTM_G31_REV1, 2012). A 100 mL solution of 3.5% NaCl was prepared and a CH Instrument (electrochemical analyser 600E) with its own software was used to conduct the corrosion experiment. The NAB sample was defined as the working electrode and the saturated calomel electrode (SCE) were connected as the reference electrode and finally a platinum wire regarded as a counter electrode or the auxiliary electrode. The experiment was conducted at room temperature. A schematic of the corrosion test cell with three electrodes is shown in Figure 38.
The instruments use a potentiostat method of polarization and, like all electrochemical instruments, is a three-electrode configuration measuring the flow of current between the auxiliary and the working electrode. A Zero Resistance Ammeter (ZRA) circuit was used to indicate the current developed during the process and the potential of the couple electrode relative to the reference electrode. The ZRA is also able to measure the high input impedance circuit.

The sample was fully covered with water resistance material and 6mm diameter aperture was prepared facing the referencing electrode for the electron transfer during the reaction. A scan rate of 0.002 Vs\(^{-1}\) and a potentiostat swept area between -1 V and +1 V were selected to produce the Tafel polarisation graph. From the graph, the current densities, Mili Per Year (MPY) and other related results could be determined by means of the software.

4.7 Cavitation Erosion Test

Another major failure of marine components is cavitation erosion. The continuous turning of the propeller causing fluctuation of pressure and hence stream of water hitting the surface contributing to the formation of cavitation at the metal surface. To determine whether the FS processed surface has successfully enhanced, the surface properties cavitation test must be conducted.

4.7.1 Cavitation Sample Preparation

Few selected parameters were chosen for the cavitation erosion test. The sample FSPed with multiple runs obtains a wide surface to cater the vibratory apparatus horn. The size of the horn surface is 19 mm diameter so the multiple runs were prepared with a 30 mm wide and 40 mm
length to cover the full horn tip surface. The sample was cut using a CNC wire cut to the size of 30 mm X 30 mm X 10 mm depth to suit the holder.

The samples were then polished using the same step as the corrosion samples preparations; hence, producing the same surface roughness before storing it inside the desiccators.

4.7.2 Cavitation Experimental Setup

The guidelines for the cavitation erosion experiments using a vibratory analysis were obtained from the ASTM G32-10 (2010). A solution of 3.5% NaCl was prepared in the experiments. An ultrasonic processor (Sonic Vibra Cell VC 505, 500 Watt), which emits vibratory waves at a frequency of 20 kHz and an amplitude of 25 µm was chosen for the experiment (Figure 39). The jig was set to hold the specimen at a constant gap of 1.5 mm using a feeler gauge. The distance as Preece (1979) suggested that a gap of more than 5mm will not be effective for the test. The specimen and the probe emitting end were then held near the bottom of a beaker with 600 mL of liquid. Cooling water was circulated around the beaker to maintain a consistent temperature of $23^\circ \pm 2^\circ$C. To eliminate erroneous results, particular care was taken to ensure that the temperature remained in the allowed range. Each time a new specimen was tested and after every 8 hours of liquid usage a new solution is obtained.

The total testing period for each samples were 12 hours with 2 hours intermittent. At every stop, the samples were clean with ethanol then rinsed with distilled water and finally dried with hot air. The samples were then weighted using an analytical balance with an accuracy of ±0.0001 grams to establish the weight loss as a function of time. The results were properly recorded and calculations were made.

After the samples were sent, the AFM machine records the gradual increment of the average surface roughness (Ra). For the detail of surface integrity, the samples were sent to the scanning electron microscope (SEM). It is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons.
The purpose of using AFM at the surface was to increase gradual increment of the surface roughness and to monitor the crack propagation. The apparatus used was Veeco 3100 model, which is able to detect the surface topography of peak to peak 6 nm and the area of scanning is $3600 \text{ nm}^2$ (JEOL, 2014). The schematic of the AFM feedback loop circuit is given in Figure 40. A contact mode of scanning method is used by dragging the tip over the surface with the tip in contact for the detection of the surface fluctuation.
top of the cantilever substrate. Before starting the scanning, the Scan control panel should be set properly. The scan size to 1 µm and X and Y offset is zero. The integral gain is set to 2.0 and the proportional gain is 3.0 with a scan rate to 2 Hz.

To start off the scanning, the desired area must be chosen and the motor must be set to engage. During the scanning, the Real-time operation graph must be monitored to ensure that the tracing and retracing is working well for the scope tracing (Figure 41).

![Scope Trace with Optimized Realtime Parameters](source: R. Robbins, 2010)

The feedback loop maintains constant oscillation amplitude by maintaining a constant Root Mean Square (RMS) of the oscillation signal acquired by the split photodiode detector. The vertical position of the scanner at each (x, y) data point maintains a constant “set-point” amplitude stored by the computer to form a topographic image of the sample surface. By maintaining constant oscillation amplitude, a constant tip sample interaction is maintained during imaging.

There is a possibility of some disturbance during the scanning, so adjusting the scan rate and maintaining the real-time parameters should give a better image of the topography. The selected sharp image is then captured and saved. A sample of the flatness of the polished sample is shown in Figure 42.
After the AFM, the samples were sent to the SEM JEOL JCM 6000 for a detailed surface morphology. The SEM has the ability to magnify a very high resolution (60000 times) and the image formation could be set up within 3 minutes (Galloway, 2004). Prior to scanning, the sample must be clean with distilled water and dried to eliminate contamination on the surface. The vacuum chamber must be pressurised with air to unlock. The sample should be placed in the centre of the stage and the chamber should be locked to depressurise until total vacuum is achieved in the chamber. Using the monitor control, parameter must be set such as Secondary electron image (SEI) or Backscatter electron image (BEI). The accelerating voltage must be set to 15 KV and the electron gun filament must be switch on. Using the monitor control, the image must be adjusted via adjusting the electron gun and relocating the inner-table. Pushing the auto magnification the image will be corrected automatically.

The electron beam was operated in area mode with a beam diameter less than 0.2 µm. The electron beam hits the sample surface area around 30 nm-50 nm and then undergoing Bragg diffractions. The diffracted electrons imposed the camera screen to produce sample surface images. When the SEM monitor is showing a satisfactory sharp image then the image will be captured and saved. The schematic of the SEM is shown in Figure 43.
Figure 43: SEM Schematic (Source: J. Witkke, 2008)
CHAPTER 5: RESULTS AND DISCUSSION

This chapter experimentally validates the results obtained from all the describe tests. As the objectives of the study is to demonstrate the optimum parameters using FSP, it can be successfully used to improve the mechanical, cavitation erosion and corrosion attributes. The results obtained were carefully analysed and discussed to motivate the objectives. The first session of this chapter is the overall results of the findings related to the conducted test. The second part of the argument is regarding the microstructural evolution when the specimens were subjected to FSP process. The third part is the relation of FSP towards the hardness of the specimens and eventually the discussion relating the cavitation erosion and corrosion.

5.1 Results

The original cast of microstructure framework has been illustrated from several different forms existed in the form of NAB alloy.

- The microstructure case of the alloy comprises of the columnar grains of alpha phase with copper-rich solid solutions.
- Large dendritic to rosette shape precipitate indicates the microstructural phase of NAB alloy.
- The microstructures of friction stir operation function is associated with the layers of cast NAB.
- NAB alloy increases the tensile properties, fatigue strength and hardness.
- The processed surface of high cooling rates and FSP construct highly packed grain structures. The mechanical properties of NAB alloy are modified by these structures.
- Surface mechanical properties are constructed with the closed packed structure of FSP.
- The microstructure surface of NAB alloy influences the heat of scratch process.
- The paucity of time did not allow to constitute a grain boundary and to normalize the minute grain size.
- The significant minimization of the cast nickel NAB anticipates the midst grain size of processed top layer.
- Traditional machines are used to integrate FSP of NAB alloy rather than robotic exploiters (Oh-Ishi, Zhilyaev & McNelley, 2006).
- There was no loss in weight found in the first hour due to the presence of an intact surface oxide in both FSP.
• Different erosion rates were witnessed in the additional time period due to the interaction of microscopic surface destruction at specific point.
• No mass is lost during the first phase of incubation period as vibratory waves are separated into intermetallic bonds and oxide layer among the globular grains.
• In the second phase, oxide layer resistance is short of the requirement.
• When mass is lost in the second phase, surface bonds are damaged and weakened.
• When microstructure cast surface is simply battered, upward metallic bonds are observed in the phase (Jahanafrooz et al., 1983).
• A persistent influence of lost mass is observed within the FSP phase.
• Dark colour indications reveal the presence of heavier surface cavities as evident from the FSP tool and microstructure cast.
• When cavitation erosion testing surpasses 12 hours, the difference in mass loss is evident among microstructure cast of NAB and FSP.
• Rough surface of nickel aluminium exhibits the presence of large cavities.
• The entire surface colour did not reveal pale colour on the cast sample as well as less visual evidence of cavitation is being displayed from FSP sample.
• The cavitation erosion process illustrates the increase in tensile strength.
• Grain size reduction produces the resistance in mechanism to illustrate the surface damage and erosion.

The augmented corrosion resistance of FSP to nickel aluminium bronze is categorized from the following aspects:
• The contribution of finer grain structure shows the improvement of corrosion resistance to the rougher grained microstructure of cast NAB alloy.
• The finer grain particles are associated with the corrosive extent and probable absorbencies to attack the microstructure surface.
• The probable solutions for attacking the intergranular structure are emerged from deeper corrosion paths of grainier microstructure.
• The influence of intergranular damage and the corrosion resistance factor is minimized and increased respectively from the corrosion path intensity.
• The intergranular structure and lower surface are damaged actively by the imperfections of corrosion effect.
To normalize the damage, a concentrated catchment region is integrated during the FSP.

To normalize the eroding damage, lesser catchment areas and less imperfections are associated with FSP of NAB alloy.

The penetration of microstructure process and the corrosive ions requires a lasting period.

The phases of cavitation erosion exhibit the inhomogeneous microstructure and the granular microstructure of cast NAB.

The increase in erosion resistance is resultant from the enhancement of FSP NAB.

FSP increases the revolving speed and number of FSP cycles.

The influence of dissolution particles reduces the influence of available sites for the galvanic connection.

The optimization of microstructure cast has illustrated lower corrosion extent as compared to microstructure cast sample including the improvement of the phase’s isolation, reduction of penetrability and modification of grain size.

Higher intensities are produced in the higher-pressure regions that collapse the cavitation erosion.

Throughout the test, nickel aluminium bronze cast is promptly higher in weight as compared to FSP.

No significant difference has been illustrated from the electrochemical polarization behaviour of the NAB alloy that exhibits the microstructure cast and FSP samples (Fuller et al., 2007).

The rate of entire corrosion process is controlled from the cathode functioning step.

The attributes of the diffusion density are illustrated by the cathode curves that restrict diffusion density to the potential range of FSP.

The cathode minimization reaction extent modifies the corrosion potential in the imperative direction.

The existing density of applied voltage indicates the formation of the deprived surface film.

Microstructure cast illustrates higher corrosion rates and lower corrosion density.

The measurements of FSP were executed in order to determine the consequences of electrochemical polarization behaviour of the nickel aluminium bronze curve.
• The microstructure cast and FSP samples comprise high frequency capacitance and low frequency impedance.
• High frequency capacitance was signified from the aspects of surface illustration at the bottom of the matrix and solution.
• The upward corrosion rate for FSP indicates the cast sample from the FSP samples.
• The microstructure cast of NAB categorizes the complex phases and phase formation sequence.
• The dominant parts of fine and equivalent alpha grain are included in the microstructure cast in the FSP of NAB.
• The deformed alpha matrix and retaining lamellar spheroidization are recovered and recrystallized from the FSP (Li et al., 2004).
• The displacing microstructure phases of FSP are influenced by magnesium alloys.
• The heat treatment solution is influenced by the dissolution of cavitation erosion from the cast of magnesium alloy at higher temperature.
• The form of cathode process and oxygen reduction is observed from the electrode procedure of NAB alloys.

The corrosion extent is influenced from the surface roughness, history and metallurgical composition. Moreover, the eutectoid region was slightly damaged from the crevice corrosion behaviour of the NAB. The intensity of attack in cavitation erosion is being held from the enduring nature of the phases, which made it vulnerable for FSP. It has been assumed that alpha grains revealed very minute damage even though the eutectoid alpha phase was preferentially damaged (Preece, & Hansson, 1981). Therefore, this result was classified from the existence of upward extent of cathode stages throughout the eutectoid region. Following factors have been ascribed from the increment of static involvement corrosion resistance to the FSP of NAB. The finer counterpart is large in the proportion as compared to the coarse microstructure of the nickel aluminium bronze. It has been evaluated that size and distribution of micro constituents as well as the rougher microstructure affected the damage intensity in the microstructure cast of NAB. The grainy microstructure is emerged from the isolation of constituents specifically in the grain boundaries, which might endow the corrosion phase in order to resolve the corroded phases. The decomposition of grainy phases as well as the homogenization of elements results
into the FSP. Therefore, the corrosion resistance is enhanced by eliminating the corrosion phase (Mahoney et al., 2003).

The reduction for cavitation corrosion has been endowed from the cast samples of porosities. On one hand, densified regions are much higher as compared to cast porosities region. On the other hand, the region among the corrosion solution and sample is augmented from the porosities, which additionally produce the process of cavitation corrosion testing. Thereby, the corrosion resistance is augmented significantly by reducing the porosities. The promotion in corrosion phases is exhibited from the inhomogeneous distribution of the nickel aluminium bronze cast (Auret et al., 1993). The segregation and morphology of the kappa phases are improved as well as modified through FSP and; thereby, augmenting the corrosion resistance. Moreover, it has been examined that rotation speed and FSP was passed from the augmented corrosion resistance. The extent of porosities accessible for galvanic connection was minimized from the enhanced extent of corrosion resistance in which the characteristics of degenerate particles were involved. High and low intensities have been adhered from the grain refinement in stir region by considering the extreme plastic deformation. The existence of grain refinement was conducted for entire copper alloys throughout the stir zone (Song et al., 2015).

Massive globular constituents were adhered in the base material of alpha particles to break into minute particles as well as distributed throughout dynamically crystallized zone and mechanically influenced region by considering the grain refinement in the stir zone. The augmenting stir intensity has been enhanced from the recrystallized grains throughout the stir zone. The joints generated for nickel aluminium bronze cast have been illustrated from the strength profiles of FSP (Fontana, 2005). It has been assumed that strength value of the base material along with hardness increment in the stir zone was adhered from overall joints. In addition, number of researchers have addressed the increase in hardness for FSP nickel aluminium bronze. The augmentation in the strength throughout the stir zone might be initially ascribed in the FSP to illustrate the grain refinement regions. On the other hand, the stir intensity is dependent on the extent of strength in the nickel aluminium bronze. This is due to the fact that stir depth is augmented when extent of strength is increased. It has been examined that high energy level of martensitic is associated to be resulted in microstructure casts of nickel aluminium bronze, which might considerably influence the subsequent electrochemical behaviour. It is anticipated to be the higher vulnerability to corrosion as compared to other revealed from heat treated specimens (Schüssler & Exner, 1993).
The mobility of corrosion product is revealed from the restriction of corrosion layers in which sustainable protective layer is constructed on the basis of alloy surface. The corrosion attributes have been studied by examining the short-term electrochemical techniques, longer-term immersion trials and impingement studies. These studies and attributes were aligned with the attributes of heat-treated, wrought and cast NAB. The elimination and reduction of corrodible beta phase as well as augmented extent of alpha phase is resulted from the appropriate heat treatment (Auret et al., 1993).

5.1.1 Discussion of vibration analysis
The computer numerical control machine was employed to execute the FSW process due to the paucity of FSP machine. It has been witnessed that the numerical control machine was not interacted with a force controller and, therefore, it consequently restricts plunge force to be produced. A force sensor was employed below the specimen to control the axial force generated throughout the numerical control machine involvement. Thereby, the acquired axial force can be illustrated from a general view and a plot. The flexibility of numerical control machine is observed to be greater than 60 times as compared to the robot arm. The flexibility of the CNC was deemed to be significant for such experiments. The collection of information was made by connecting force sensor with Labview software throughout the FSP.
Figure 44: CNC FSP

The vibration analysis has been depicted from figure 44, which illustrates similarity of penetration period; whereas, shorter time is required for higher rotation. Furthermore, a longer time for penetration is required for the production of lower rotation speeds.

The results have shown the restriction before the detection of high force and pin penetration tool. Furthermore, higher rotation period is required for the penetration periods on the basis of shorter time. On the contrary, a longer time of penetration is produced by lower rotation speed.

It has been analysed from the results that temperature will increase when rotation will be faster. The execution of the process is based on the required higher rotation traverse force; however, experiments are terminated from the reduction of the forces. A significant vibration is displayed throughout the process while lower speed rotations determine a more stable progress.

5.1.2 Plunge/Axial Force Control

The plunge depth, rotation speed and the traverse speed are the three parameters that determines the control independently. The plunge depth was also demonstrated independently by employing CNC machine for the FSP. The structural integrity and the stiffness of the control machine tends to endow an effective axial force for the process due to the CNC capability. On
the contrary, the controlling of the plunge depth was not undertaken separately as force control technique determines the axial force acquired by the FSP tool. The interaction of the axial force and the plunge depth is associated in an indirect way to possess adjustable and intrinsic stiffness of the experiment. The illustration of axial force has been depicted from the presence of NAB microstructure, which reveals the occurrence of iron more than 5 wt. % Fe and is adhered as a large rosette-shape (Figure 45).

**Figure 45**: Graph of Axial Force Vs RPM

It has been found that there is a slight escalation in the plunging depth during the transient state of FSP, which consequently leads to the considerable expansion in the steady state condition. Furthermore, displaced material is restricted between the shoulder and the pin due to the penetration of surface throughout the plunging phase. The results have shown that movement of the material assists in dissolving the displaced material due to the increase in axial force. The traverse speed and the rotation speed of the FSP tool are directly related with the axial force. These parameters can change the steady state axial force although these parameters have a minute influence on the axial force than the plunging depth. Therefore, it has been identified that the increase in surface contact significantly increases the surface temperature. The illustration of axial force and traverse speed is shown in the figure 46, which shows different traverse speeds and rotation speeds. It has been depicted from the figure that these speeds will consequently differ in terms of hardness.
5.1.3 Robotic Friction

The implementation of robotic friction towards the control of temperature and axial force of NAB have been depicted from the results. A constant spindle speed, a constant travel speed of 300 mm/min, and modification in commanded vertical tool position were used to measure the interface temperature. These temperatures were kept constant at the probe location throughout a weld position. The dimensions of the process were captured effectively from the temperature measurement approach as direct contact was developed between the thermocouple sheaths along with the work piece of aluminium. It has been evaluated that there was no constant measured temperature, which vary under the conditions of operating process for robotic FSW (Farmer et al., 2013).

The extent of spindle rotation is observed to match the extent of oscillations. The amplitude of the probe and shoulder interface temperature vary in regards to the weld position. Prior studies have found that measurement strategy was implemented on the basis of captured temperature dynamics. Furthermore, the calibration of specific measurements was adhered from the experimental results of past studies (Akinlabi Andrews, & Akinlabi, 2014).

The amalgamation of work piece with the amplitudes of the oscillations were found to be greater in the welding forces as well as in the setup of CNC mill, indicating higher flexibility of the robotic system as compared to stiffer CNC mill. The process of closed-loop control system is enabled throughout the weld traverse; therefore, the results are associated with the weld traverse for effortlessness. The implementation of command tracking experiments was effective to regulate and identify the performance of control system. The frequency and
amplitude vary during the process for identifying the pre-requisite temperature of probe. The limits of spindle speed vary from 1500 rpm and 2500 rpm; whereas, the limits for tool position ranging between 5 mm and 16 mm. The controller was used to adjust the speed of spindle to measure the probe interface temperature. It can be adhered that the probe interface temperature varies with equivalent frequency (Vikash et al., 2015).

Mungsuntisuk (2013) has shown the significant phase lag of nearly 90 degrees to observe the closed loop bandwidth of the system. The phase lag of the control system executed in the FSP has been reduced due to the existence of higher communication delays shown on the robotic system. Sophisticated controller structures can be used to increase the performance of closed loop bandwidth. On the contrary, the performance of this bandwidth can be judged from the acceptance of numerous applications (Mungsuntisuk, 2013). The suggested extent of the measured temperature for the robotic system was higher as compared to the measured temperature.

The results of past study have showed the properties of disturbance rejection for the temperature control system on the basis of FSP (Mahoney et al., 2003). The measurement of closed loop control of axial force can be examined from the steps of command tracking, which require desired force magnitude of 450 N. The vertical position of the tool is adjusted suitably to allow the system for the execution of commands in sequential approach with insignificant overshoot, closed loop time constant, and steady state error.

The robotic FSP incorporates ramp disturbances along with a vertical offset to execute terminating point of the weld and to assess the properties of disturbance rejection for the axial force system. The escalation or reduction of the axial force can be observed when axial force is not appropriately utilized. The depiction of weld surfaces can be significantly evident to assess the quality of weld. The adjustment of the controller is positioned vertically, leading to develop appropriate shoulder-work piece contact at the time of employing axial force control (Song et al., 2013).

The deployment of ramp disturbance can enhance the weld quality as well as the performance of axial force. The assessment of command tracking was used from the combination of force control system and temperature. A weld travel speed is commanded with a constant axial force. The command of axial force reduces after few seconds to increase in discrete steps over a 7 second interval. The achievement of the desired axial force is observed when the position of controller is slightly changed. The temperature is influenced massively from the modification
in the vertical position of the tool. Thereby, the escalation is made in the desired interface temperature, causing the controller to augment the spindle speed (Culpan & Rose, 1978).

Fuller (2006) has investigated the control system for instantaneous command tracking using force commands and sinusoidal temperature. The movement of desired probe interface was measured at 0.1 Hz with an amplitude of 15 degrees; whereas, the measurement of desired axial force was made at 0.1 Hz and amplitude of 450 N. The performance of combined control system can be observed effectively from the vertical tool position, and adjusting spindle speed in order to capture the force commands and temperature. The implementation of ramp disturbance is also evaluated from the interconnected temperature and force control system. The working of tool shoulder work piece contact is adjusted at the vertical position of the tool, which causes a constant axial force. Furthermore, the adjustment of controller is resulted into the alteration of heat generation obtained from the desired interface temperature (Fuller, 2006).

The application of temperature is used to evaluate the combined control system, which applies a ramp disturbance to the backing plates of several different thermal diffusivities. The performance of welds was measured over a series of different backing plates to simulate a modification in work piece properties. For thermal diffusivities, copper and titanium were used to assess the stiffness of robotic FSP. Thermal management was also utilized by different backing materials throughout welding process to enhance the stiffness of the weld. This enhancement can be made specifically in the heat affected realm. The reduction was observed in the axial force during the comprehensive weld process due to the terminating point of weld performed over the work piece. Therefore, the spindle speed escalated as well as the vertical tool position was increased during the weld. This approach can sustain the desired temperature and control force where quality of weld is improved in the surfaces (Culpan & Rose, 1978).

5.2 Results and Discussion (Microstructure)

5.2.1 Microstructure

A similar microstructure and overall shape were obtained with the CNC and the robotic process. Figure 47 is an illustration demonstrating scanning electron microscopy (SEM) of the FSP cross section. The indistinct diagram includes the Stir Zone (SZ), the Thermomechanical Affected Zone (TMAZ), HAZ and the BM of NAB (BM). The motion of the tool describes the advancement of the page; therefore, the advancing side is on the left (tangential speed of the tool is similar to the movement direction) and the retreating side is to the right of the diagram.
(tangential speed of the tool is opposite to the movement direction). The tool shape is conical with a penetration of only 5 mm deep whereas the bottom width is 2.5 mm.

Figure 47: Montage of FSP Cross Section Indicating the Details of the Zones

The robot and the CNC machine reacted to the plunge force on the tool and moved upwards. This factor combined with the tool wear, produced less stir zone and less penetration. The edge of the advancing side is quite smooth while the retreating side is indistinguishable. The cast NAB grains located near the boundary of the SZ were found to be distorted and the shearing effect was found all over the boundary region. There are dark etching features present in the area, which may be partial reversion of the lamellar α + K_{iii} to form β because of the thermal effect during the FSP and sudden cooling as the tool continued to move. The section outside the SZ exhibits minor distortion and this zone is known as thermomechanical affected zone (TMAZ).

Figure 48 shows the microstructure of BM while Figure 49 to Figure 52 illustrates a higher magnification from the OM exhibiting the approximate zone location along the vertical surface as in Figure 47. Zone 1 is the top section that is rubbed by the tool shoulder during the stirring process and exhibits the most fine grain structure. Zone 2 is located just below the stirring surface region and exhibits a band-like feature. Figure 49 shows the microstructure of zone 1 showing that globular particles of K_{ii} are present near the centre of the image. Also there are two morphologies of the α-Cu phase (that is the light-etching phase in the NAB material). Most of α grains feature a near ellipsoidal figure and are stretched in the perpendicular direction of the axis tool rotation. Elsewhere, the dark-etching region in zone 1 is comprised of
Widmanstätten α with bainite and martensite (dark feature). The band-like structures and coarse globular $K_{ii}$ were also found underneath the zone 2 (Figure 50) and disclosed by the OM. Furthermore, some light-etching of α phase in an elongated form in the direction perpendicular to the tool motion are presented. The dark etching region contains the mixture of Widmanstätten α, bainite and martensite. The results have shown the microstructure testing of the original cast nickel aluminum bronze alloy. Several different types of FSP are shown from the NAB

Columnar grains of α phase were incorporated in the microstructure to identify the copper rich solid solutions. Furthermore, minute extent of lamellar eutectoid phases of β phases were existed in the form of intermetallic and martensitic. K phases are represented in four different phases. The first shape of the K phase is emerged as a large dendritic shape, which is often equivalent to rosette shape precipitate. The second phase of K shape is also similar to first phase; however, it is slightly smaller than the first phase of K shape (Mishra, De & Kumar, 2014). The precipitates of third phase in K shape are revealed as a globular eutectoidal decomposition of lamellar pattern products. Fine particles of different sizes are reflected in the fourth phase of the K shape, which spreads during the grains beside specific crystallographic directions. These four phases are based on iron rich precipitates, forming a base of the nickel aluminium alloys. The size of the second and fourth phase of the K phases are lesser than the rest of the phases. The existence of PFZ is adhered at the grains perimeter.

In zones 1 and 2, the presence of globular $K_{ii}$ is not apparent. The elongated α-Cu grains are not indicated appropriately and the Widmanstätten α is finer in zone 2 relative to zone 1. The lamellar structure related to the $\alpha+K_{iii}$ is not visible in the images. Thus, the findings propose that the lamellar $\alpha+K_{iii}$ present in the BM of NAB has returned to the β phase during the severe deformation and heating of the FSP process. Nevertheless, the primary α phase forms while equilibrium cooling does not manage totally to convert into β phase. Thus, the content of the microstructure during the FSP process comprises of α and β in assorted volume, which depends to the local peak temperatures. Both of the phases change and then the β phase transforms on the following cooling stage leaving the Widmanstätten α or bainite or martensite depending on the cooling rates following the motion of the tool. The cooling rates are estimated to be $10^0 \text{Cs}^{-1}$. The rates are expected to be quicker than equilibrium cooling. The transformation process that takes place the β phase was discussed explicitly by Berzina and is summarised in
the Figure 53 (Arora et al., 2010). The results have shown the layers of cast nickel aluminium bronze, stir zone, and TMAZ from the microstructural analysis of the robotic FSP. The results have shown that the NAB layers from FSP refined the grain structure and reduces the defect present in the porosity. Thereby, the homogenized microstructure augments the tensile properties, fatigue strength and the hardness of NAB.

Highly packed grain structures are constructed from the effects of FSP and high cooling rates, altering the mechanical properties of NAB. It has been investigated that formations of a stable and proactive passive films lead towards a higher grain boundary density. These fine grain structures are attached together from closed packed structure, which generates enhanced surface mechanical properties.

The most critical layer of the surface is the first layer or stir zone, which is toughened due to the abrasion among the NAB surface and FSP tool. The surface area is massively influenced from the heat generated during the abrasion process, which consequently resulted in plastic deformation. There is no ample time to normalize the grain boundary when the abrasion process cools swiftly. However, the sudden drop of temperature recrystallizes the grain boundary with a minute grain size. It has been estimated that the grain size of the processed stir zone is approximately 1 to 5 μm. The cast material compared with the processed stir zone significantly reduces the average grain size of 100 to 150 μm. The results have been revealed from the outcomes of past studies. These studies have implemented robotic FSP for NAB alloy rather than the conventional machines (Küçükömeroğlu et al., 2016).

Coming back to Figure 51, zone 3 is located in the centre of the SZ. The microstructure exhibits very fine and equiaxed grains relative to zone 1. This zone demonstrates a dark-etching region in the location between α grains but the volume of the β phase transformation is lesser compared to zone 2. Lastly, zone 4 (Figure 52) is the lowest zone from the SZ. The grain flow pattern is obvious at the boundary of SZ and TMAZ.
Figure 48: Montage of FSP Cross Section Indicating the Details of the Zones

Figure 49: SEM Illustration of As-Cast NAB

Figure 50: OM Illustration of Zone 1
Figure 51: OM Illustration of Zone 2

Figure 52: OM Illustration of Zone 3

Figure 53: OM Illustration of Zone 4
5.2.2 Discussion of Microstructure

It is obvious that when a cast NAB surface is treated by FSP, the microstructure of the sample will be altered significantly. Studies of the restructuring of grains have been undertaken by many researchers (Lotfollahi, Shamanian & Saatchi, 2014; Auret et al., 1993; Li et al., 2004). Numerous elements of cast NAB, such as band-like features, fine lamellar structures, martensite and very refine grain structures, were detected in different regions and signify fluctuations or changes the thermo-mechanical background from region to region in the stirring zone. There are few studies that shows the impact of concurrent deformation on the kinetics of phase transformation to explain the microstructures found in this present study. As well, the separation of the alloys in the slow cooling of the cast NAB sample will produce assorted microstructures and its own complications. Thus, the local peak temperatures obtained during the FSP could be estimated as the product of local peak temperatures is different at regions and stages during the cooling process. Such approximations are important as exact temperature measurement in separate regions is nearly impossible.

As cast NAB was sent for static annealing in the temperature range of 700°C to 1000°C (0.78 to 0.95 $T_m$), similar microstructures between the CNC and Robot FSP were exhibited after 1 hour of the annealing process in the region of 870°C. The increment of diffusion rates surplus the vacancies while FSP (McNelley, Oh-Ishi & Zhilyaev, 2007) was occupying the place, which sped up the response for the reversal of the lamellar $\alpha + K_{iii} \rightarrow \beta$ because of the heat into the temperature region. Hence, it can be predicted that in the FOP induced microstructure, the modification reveals the local equilibrium due to the achievement of peak temperature. The $\beta$ phase created by the reversion reaction $\alpha + K_{iii} \rightarrow \beta$ predicts the transformation mostly at the
peak temperature and then normalised to room temperature but not in a stable condition (Oh-Ishi, Zhilyaev & McNelley, 2006). The cooling rates obtained during the FSP were faster by a factor of $10^3$ than those obtained during the casting of a bigger sample. The cooling rate is a satisfactory manifest for the β phase created during the transformation at the local peak temperature to convert into Widmanstätten α phase, martensite or bainite. The outcome of the β phase decomposition is obvious as the dark-etching regions observes the OM. The region can easily be observed in Figure 10 and Figure 11. At a higher OM magnification, the Widmanstätten α phase can be seen as light-etching components. The eutectoid reactions of $\beta \rightarrow \alpha + K_{iii}$ during normalisation has been documented as happening at ~800$^0$ C (Jahanafrooz et al., 1983). Hence, the existence of β phase decomposition constituent signifies that the local peak temperatures passed this level during the FSP.

At the second stage, very fine $K_{iv}$ precipitates will reliquefy into the α phase when achieving the peak temperature of 860$^0$ C and as the cooling continues a finer precipitate will be established. In the following stage, a coarse precipitate will liquefy into β phase at the local peak temperature upsurge of more than 930$^0$ C. Lastly, for the cast NAB that has more than 9.4 weight percent (wt pct) of aluminium, the proportion of β phase will increase with a rise in temperature to ~1030$^0$ C. At this temperature a single β phase solid solution will exist. In this present study, the presence of the coarse $K_{ii}$, the existing of Windmanstätten α, bainite and martensite as the result of β phases was extremely helpful in determining the local peak temperature at the Stir Zone. An illustration using OM of the FSP surface related with the peak temperatures was presented in Figure 54.

It is significant that the microstructure of the surface is in contact with the tool shoulder (i.e. in the zone 1 (Figure 49)) as it shows that a very fine microstructure was established in this zone. This shows that extreme strain happened in the SZ owing to friction created between the surfaces. The existence of coarse Windmanstätten α beside the β phase decomposition in zone 1 is evidence that the peak temperature at the surface was higher than the other zone regardless of the cooling effect on the surface and the tools itself. Alternatively, appearance of $K_{ii}$ in all the regions, including near the surface signifies that the peak temperature did not surpass 930$^0$ C. In the region of gradual cooling of cast NAB, the $K_{ii}$ precipitates are larger (average of 20 µm in size), which reveals the high solvus temperature for this component. It is predicted that during the FSP process the $K_{ii}$ precipitates need extra time and strain to liquify the $K_{iv}$ precipitates or the lamellar $\alpha + K_{iii}$ components.
As shown in the zone 2 (Figure 50), it is noticeable that a band-like structure was present. The light-etching element present contains a variety of β phase constituents. Both precipitates present with an elongated shape in the direction perpendicular to the tool axis. The lamellar $\alpha + K_{iii}$ components shaped by the eutectoid decomposition in the zone solidified at the end because of the separation of the Al components. As the alloy was continuously heated, the initial primary shape of the microstructure is the reconstruction of the lamellar eutectoid structure to produce the β phase. β phase will comprise of $K_{ii}$ and will be enclosed with a majority of α phase. When the peak temperature reaches the $\alpha + \beta + K_{ii}$ zone and the volume fraction $\alpha$ and $\beta$ are nearly the same, an extreme deformation outcome is instant causing restructuring of the $\alpha$ and $\beta$ phase and the creation of a band-like structure (McNelley et al., 2009).

At the outer part of SZ, the proximity of the TMAZ is shown in the zone 3 (Figure 51), the microstructure consists of a combination of α phase and martensite that exists as a small portion at the border of SZ and TMAZ. Hence, the lamellar $\alpha + K_{iii}$ components has been able to reproduce β phase at the TMAZ zone just below the tool surface and it seems that it is the preliminary modification while advancing the SZ from the cast NAB. This assists that the lamellar $\alpha + K_{iii}$ components are the initial components to be liquified when the sample was treated with heat. The presence of martensite at both sides of SZ below the tool shoulder signifies that the temperature changes in the vertical direction are bigger underneath the tool pin rather than at the tool shoulder.

In the center of the SZ, at zone 3 (Figure 51), α grains are found to be very fine and equiaxed (opposite to the elongated α grains in zone 2) and dominate the components of the microstructure. The variety of microstructures (including bainite and martensite) is obvious in this zone. The bainite dominate a very fine and the mixed microstructure. Hence, it is predicted that the peak temperature during the FSP process surpassed the eutectoid temperature but fewer than in zone 2. The process also produced a larger amount of β phase in the region. At zone 3, much transformation happened leading to the presence of $\alpha$ phase, β phase, $K_{ii}$ and $K_{iv}$. The formation of β phase will take place at the boundary of $\alpha$ phase and $K_{ii}$, while the $K_{iii}$ will be inclined to form around the coarse $K_{ii}$ precipitates with the cooling process that followed the casting of cast NAB. Throughout the heating, the $K_{iv}$ phase would coarsen as the temperature rose, but afterward liquefy to become $\alpha$ phase at temperatures above 860ºC. In this region, as $K_{iv}$ has fully liquefied, the concentration of Al and Fe will be increased supporting the
development of the β phase. The changes that happen at the temperature of 860° C are supported by the findings of Culpan and Rose (1979).

At the bottom of SZ, in the zone 4 (Figure 52), referring to the fine α grains size in the region. The OM illustrates that there is the presence of some β phase in the area even though the grain boundary at α grains are dominated by the precipitates of the \( K_{iii} \) phase. The formation of the β phase close to the surface in the zone 4 was not observed under the OM, and suggesting that the peak temperature was close to the eutectoid temperature and the prime effect is nearby to the extreme deformation by the FSP. From the illustration, it is obvious that the existence of large number of small particles was operating under the FSP. In this situations, it is proposed that the grain refinement is due to the recrystallization of the particle-stimulated at the area (Humphreys, 1977).

![Figure 55: Image of OM and Microstructure with Peak Temperature Plotted against Depth](image)

**5.3 Result and Discussion (Surface Hardness)**

The surface hardness of the NAB was measured with a Dura scan tester taking an average of 9 points in the centre of the Stirring Zone (SZ) zone and randomly at the cast surface. A 200-gram force was used and the readings obtained show significant differences between the samples. The Vickers hardness \( HV_{0.2} \) of cast NAB = 205 shows the hardness for CNC FSP and Robotic FSP (Figure 55). Different results were shown for different parameter of process. The results vary from a minimum \( HV_{0.2} = 226 \) to \( HV_{0.2} = 299 \) maximum.
The surface hardness caused by the CNC FSP process shows a similar pattern as the robotic FSP. At the initial stage of the spindle, the rotation speed was set at 1000 RPM, and with the traverse speeds of 0.5 mm/s, 1.0 mm/s, 1.5 mm/s and 2.0 mm/s, the Vickers hardness was $HV_{0.2} = 265$, $HV_{0.2} = 264$, $HV_{0.2} = 261$ and $HV_{0.2} = 263$ respectively (Figure 55). An average Vickers hardness of $HV_{0.2} = 263$ was obtained. This is considered quite high relative to the cast NAB which has a Vickers Hardness of $HV_{0.2} = 205$.

A Durascan automatic micro hardness tester was used with Vickers indenter to measure the hardness of the cast NAB. When the parameter was increased to 2000 RPM and the same traverse speeds were applied, the Vickers hardness readings were $HV_{0.2} = 245$, $HV_{0.2} = 234$, $HV_{0.2} = 240$ and $HV_{0.2} = 257$. There was a reduction in hardness of 8 percent. The decrease in hardness is explained by the temperature change, which has a direct impact to the microstructure during the process. As the temperature increased, the hardness reduced because of the grain growth at the higher temperature. Work hardening is described as the strengthening of the material by low temperature plastic deformation. Thus, work hardening contributes to the higher hardness. The larger the grain size, the lower is the hardness according to Nieh & Wadsworth (1991).

By increasing the rotation speed to 3000 RPM and maintaining the same traverse speed variation, an increase of hardness was observed in all the samples. The hardness values have an average of $HV_{0.2} = 288$, which is a significant increase in the hardness of the samples. The observation of Nieh & Wadsworth, (1991) suggests that the hardness increases as the grain size reduces. The hardness obtained in this experiment denotes that lower grain refinement was
accomplished at a lower rotational speed. Figure 55 demonstrates the relationship between translational speed and hardness. The layers of the FSPed nickel alloy bronze were conducted to measure the hardness of the robotic system.

These layers had a significant effect on the measurement of the robotic system hardness. The placement of these layers can be widely viewed from the results along with the Vickers hardness. The surface of the nickel alloy bronze testing plate was also used to measure the hardness of robotic FSP. The illustrations have identified the placement of the hardness measurement. The stir zone measurement of the FSP has been significant in exhibiting the original cast of nickel aluminium bronze. The unprocessed material of the stir zone increases the hardness of FSP; therefore, the resistance to cavitation erosion can be experimented from considerable enhancement. The axial force increases the displaced material to dissolve the material flows out as the tool passes.

The hardness varies as a consequence of different parameters introduced at the processed surfaces; different rotation speed, traverse speed and forces. The different attributes contribute to different plasticising temperatures and; thus, directly affects the rate of cooling. The illustration in Figure 49 signifies that different rotation and traverse speeds will result in different hardness. A higher rotation speed and slower traverse will produce a very high surface temperature and with a sudden drop of temperature, it will result in a distortion in the grain growth. Therefore, it will produce a very small grain and a very hard surface. Figure 56 shows the grain size of the FSP surface with a spindle speed of 3000 RPM. The grain size has an average diameter of 3 µm. Slow spindle rotation will result in a lower surface temperature and, hence, less distortion during the grain growth was shown. Apparently, a relatively larger grain with a lower hardness was achieved. Figure 57 exhibits a grain of average 5 µm size when a lower spindle speed of 1000 RPM was used. The findings strengthen those of Darras (2007), which shows that the hardness of the FS processed area has a direct relationship with the temperature reached in that area.
5.3.1 Discussion Hardness

The hardness profile measured at the SZ regions is a firm signifier that FSP enhanced the hardness properties of the cast NAB in the region of 17 percent to 46 percent. Figure 55 shows a clear understanding for the relationship of hardness with the traverse speed. Therefore, these improvements are ascribed to dual factors. The first is the coarse microstructures of the cast NAB which was extremely refined and compacted; therefore, producing a higher hardness using the Hall-Petch relationship (Nieh & Wadsworth, 1991). Secondly, by using the FSP technique, casting porosity and structural defects were eliminated and; hence, assisted the increase in the hardness at the FSP surface (Shafiei-Zarghani, Kashani-Bozorg & Zarei-
Examining the details in a vertical profile, the FSP surface shows a slight difference in hardness from the top surface moving down to the bottom. This is the outcome of a non-homogenous microstructure present during the process. As mentioned earlier, four zones were established. The first one is the Widmanstätten located at the surface and most stirred regions. The structure at the sub surface is a mixture of α phase structure present at the surface of the SZ. Underneath, there exists banded α phase and β phase. At the center of the SZ, equiaxed α phase and martensitic β phase exists. At the bottom, there is a stream-like deformed and dynamic recrystallization microstructure. Mahoney et al. (2003) discuss different properties of different zones at the FSP surface. Mahoney et al. (2003) report that the Widmanstätten α structure at the SZ exhibits better mechanical properties than the equiaxed and lamellar structures. This explains why the surface layer displays a greater hardness value relative to the central zone of the SZ. Also, the lower most region of the SZ comprises a stream-like fine grain structure and; hence, it demonstrates a better hardness value relative to the equiaxed grains at the center. Therefore, it was justified that the hardness values at the top and bottom are higher than those in the center of the SZ. The hardness of the nickel aluminum bronze alloy has been revealed from the robotic parameters of the FSP.

The robotic parameters of the FSP have massively influenced the stir zone process of the NAB. It has been analyzed that the formation of defects and weld quality were affected from the welding parameters. Every single parameter of the FSP affects the quality of materials as well as the occurrence of multiple sets of parameters. The successful production of weld quality is demonstrated from the amount of heat utilized for the welding joint. Rotational speed, axial force, torque, plunge depth and rotational speed are the major parameters, affecting the extent of heat dependency.

These parameters have assisted significantly in minimizing the extent of welding process. Furthermore, the hardness of welding machine was maintained at the specific point of the heat dissipated in the welding joint. This aspect is accomplished by modifying the consistency of traverse speed and rotational speed. A significant reduction is observed in the performance of welding machine to control the motion of axial force in case the rotational speed is augmented. The reduction in the welding machine is signified from the augmented heat input, causing the softness of the material. Therefore, it has been analysed that robotic deflections in the welding machine significantly eradicates the rotational speed at lower axial force. On the contrary, it is deemed that rotational speed restricts the friction stir coefficient.
Another study was done to determine the hardness using multiple runs of FSP. Apparently, the results show a minor non-significant difference in the hardness. The outcome shows that the microstructure was more homogenous compared to a single run and the multiple runs show more consistence readings in the SZ (Shafiei-Zarghani, Kashani-Bozorg & Zarci-Hanzaki, 2009). Negative influence on the welding machine can be observed in case the rotational speed is not appropriately implemented.

The inappropriateness in the case of rotational speed can minimize the effects of process parameters on the axial force throughout a specific parameter range. The robotic FSP is observed from the implementation of appropriate tool design. It has been investigated that generation of heat input in the axial force, traverse speed, and rotational speed contributed in the performance of FSW. A soft material will be produced from the input of high heat extent throughout the FSP. The performance of rotational speed and low traverse speed reduces the requirement imposed by the axial force. The increased rotational speed of the FSP decreases the axial force and torque in the simulation process. The results of the past study have revealed that the increased control force decreases the axial force and torque (Fuller, 2006).

It has been analyzed that a higher rotation of 3000 RPM and 4000 RPM with a lower traverse speed will exhibits a higher mechanical hardness. This shows that a higher rotation speed produces a better hardness effect compared to the hardness of cast NAB alloys. This is related to the variation in the heat absorbed during the cast NAB plastic deformation period. At the low rotational speed and higher traverse speed, the heat produced was less and not sufficient to achieve plastic deformation. This results in a greater force needed for the process and lower mechanical properties are obtained. Nevertheless, when a faster rotation speed and a slower traverse speed was used, more heat was produced and absorbed by the specimen. Thus, it produces a lower axial force and various dissolutions of K particles. This approach reduces the hardness of both the CNC and Robotic Friction Stir Process (RFSP) samples.

For the lower range of parameter (rotation speed 1000 RPM or 2000 RPM), the samples show a lower Vickers Hardness in the range $HV_{0.2} = 234$ to $HV_{0.2} = 260$. The results signify that the hardness is improved compared to cast NAB but is less hard than that produced by the upper range. The outcome is associated with the microstructure development in the SZ. Figure 57 shows the presence of an inhomogeneous microstructure in the stirring zone as it is not favourable for hardness properties. However, it can be deduced that there is a more
homogenous and more compact microstructures and a wider area of equiaxed grain structure (Figure 56). These factors are favourable for higher hardness properties in the microstructures. The corrosion extent is influenced from the surface roughness, history and metallurgical composition. Moreover, the eutectoid region was slightly damaged from the crevice corrosion behaviour of the NAB. The intensity of attack for cavitation erosion is being held from the enduring nature of the phases, which made it vulnerable for FSP. It has been assumed that alpha grains revealed very minute damage even though the eutectoid alpha phase was preferentially damaged (Song et al., 2016). Therefore, this result was classified from the existence of upward extent of cathode stages throughout the eutectoid region. Following factors has been ascribed from the increment of the static involvement corrosion resistance of the FSP of NAB. The finer counterpart is large in the proportion as compared to the coarse microstructure of the nickel aluminium bronze. It has been evaluated that size and distribution of micro constituents as well as the rougher microstructure affected the intensity of damage in the microstructure cast of NAB. The grainy microstructure is emerged from the isolation of constituents specifically in the grain boundaries, which might endow the corrosion phase resolve the corroded phases. The decomposition of the grainy phases as well as the homogenization of elements results into the FSP. Therefore, the corrosion resistance is enhanced by eliminating the corrosion phase (Qin et al., 2016).

The reduction for cavitation corrosion has been endowed from the cast samples of cast porosities. On one hand, densified regions are much higher as compared to cast porosities region. On the other hand, the region among the corrosion solution and sample is augmented from the porosities, which additionally produces the process of cavitation corrosion testing. Thereby, the corrosion resistance is augmented significantly by reducing the porosities. The promotion in corrosion phases is exhibited from the inhomogeneous distribution of the nickel aluminium bronze cast (Vikash et al., 2015). The segregation and morphology of the kappa phases are improved as well as modified through FSP and; thereby, augmenting the corrosion resistance. Moreover, it has been examined that rotation speed and FSP was passed from the augmented corrosion resistance. The extent of porosities accessible for galvanic connection was minimized from the enhanced extent of corrosion resistance in which the characteristics of degenerate particles were involved. High and low intensities have been adhered from the grain refinement in stir region by considering the extreme plastic deformation. The existence
of grain refinement was conducted for entire copper alloys throughout the stir zone (Thossatheppitak et al., 2013).

Massive globular constituents were adhered in the base material of alpha particles to break into minute particles as well as distributed throughout dynamically crystallized zone and mechanically influenced region by considering the grain refinement in the stir zone. The augmenting stir intensity has been enhanced from the recrystallized grains throughout the stir zone. The joints generated for nickel aluminium bronze cast have been illustrated from the strength profiles of FSP (Mungsuntisuk, 2013). It has been assumed that strength value of the base material along with hardness increment in the stir zone was adhered from overall joints. In addition, number of researchers have focused to address the increase in hardness for FSP to nickel aluminium bronze. The augmentation in the strength throughout the stir zone might be initially ascribed in the FSP in order to illustrate the grain refinement regions. On the other hand, the stir intensity is dependent on the extent of strength in the nickel aluminium bronze. This is due to the fact that stir depth is augmented when extent of strength is increased. It has been examined that high energy level of martensitic is associated to be resulted in microstructure casts of nickel aluminium bronze, which might considerably influence the subsequent electrochemical behaviour. It is anticipated to be the higher vulnerability to corrosion as compared to other revealed from heat treated specimens (Mishra, De, & Kumar, 2014).

5.4 Result and Discussion (Cavitation Erosion)

5.4.1 Surface Damage Characteristic. (Cavitation Erosion Surface)

The NAB and other processed specimens were subjected to the laboratory cavitation experiment. The specimens were polished using 3 µm clothes and cleaned using distilled water before conducting the vibratory process. The new and processed specimens were sent to the AFM machine to determine the Roughness Average (Ra). The initial roughness for the specimens was found to be in the region of 5 nm to 20 nm (Figure 58). Figure 58 has shown the cavitation erosion testing by using robotic FSP. The illustration has revealed that the processed and cast NAB samples incur weight losses throughout the cavitation erosion testing.
An accepted enhancement is revealed from the results, demonstrating the erosion resistance for the FSP material (Figure 58). In the initial phase, there was no detection of the weight loss as the existence of an intact surface oxide layer comprises purely the samples of Cu$_2$O and Al$_2$O$_3$. These samples restrict the development of cavities or pits. At a specific point, the erosion rates were differentiated due to the microscopic surface damage, allowing a constant weight loss rate [10].

In the early stage of cavitation, the mass loss for any sample was not notified after 25 minutes, implying that there is an incubation period in which the bombardment of vibratory waves eliminates the oxide (Preece, & Hansson, 1981). After the incubation period, the mass loss was significant for all the samples. In addition, after 2 hours of continuous cavitation at the attacked area, a light circular shape was observed at the surface (Haosheng and Jiang, 2009), which indicates the cavitation process had begun. After 4 hours of cavitation, a darker colour of the circular shape could be observed and the surface roughness was visible. This signifies the presence of micro pits and cavities, which were detected by the AFM. The roughness of the area detects $Ra$ was 296 nm for NAB, 260 nm for CNC/FSP and 121 nm for RFSP (Figure 59). The surface flaws started to develop and progressed (Haosheng and Jiang, 2009).

As the cavitation time was increased to 8 hours, the circular area become darker and rougher (Haosheng and Jiang, 2009), indicating that the micro cavities and micro cracks had begun spreading all over the surface and penetration toward the inner surface had increased. Micro cracks of 10 nm to 20 nm in length and 5 nm to 10 nm widths were detected at the surface and as initiating from the bottom cavities. The micro cracks were found to spread from $\alpha$ phase.
boundary and initiating at the boundary opposite to $K_{iii}$ and $K_{iiv}$ precipitates. The K precipitates stayed in the alloy until they were eradicated physically by the cavitation process. The same morphology was observed in an NAB vertical pump by Al-Hashem (2002), describing the premature failure of the component. This implies that a grain boundary attack with selective phase corrosion was the initiator of micro cracks. However, the intermetallic precipitates (like K precipitates) are harder than α phase solid solution due to the influence of cavitation stress, which stimulate that the micro cracks should not be ignored.

As the cavitation time was prolonged to 12 hours the progression of surface roughness observed by the AFM for NAB, CNC, FSP (FSP) and RFSP surfaces were 617 nm, 221 nm and 182 nm respectively (Figure 60 and Figure 61). A circular shape of the cavitation marks (Figure 61) could be observed clearly after the process indicating that the micro cracks and micro pits have started to merge and develop resulting in significant cavities. The surfaces show an increased roughness that is visible without any instrument. The increase in the depth of cavities and grooves has caused surface deformations. Furthermore, ductile tearing was observed (Figure 62) that occurs at the NAB surface and progresses between the grain boundaries towards the inner part of the specimens. The cavitation induced was highly localised indicating that course slip lines developed between α grains. The slips lines develop to become large grooves and eventually becomes worm hole types of deformation as times progresses (Figure 63). The depth observed by AFM varies from 100 µm to 700 µm and the diameters vary from 50 µm to 350 µm. The large surface defect is now spread to the whole attacked area.

The microstructure and phase structure of the metal have an important influence on crack initiation, propagation and mass loss during the cavitation process. As stated previously, the NAB alloys were copper-rich phase, which exhibit different crystal structures. This phase is denoted by FCC, and the phase reflected as a BCC. The FCC structure demonstrates an isotropic material behavior, low sensitivity to strain rate and good ductility (Brezina, 1982). Due to the attributes of the structures, the phase will first experience the slipped planes deformation when bombarded with vibratory pulses. As the pulses continue, more crystalline structures will encounter ductile tearing at the grain boundaries and open up new phases to be cavitated. By referring to Figure 64, it can be seen that the micro cracks and ductile tearing cavities were initiated at the boundary of α and K phases and propagated toward the inner structure, especially at the $K_i$ and $K_{ii}$ regions. In time, a significantly large crater type of cavity,
micro grooves and worm holes will merge and produce a larger defect on the metal surfaces. Figure 63 shows development of the wormhole.

The ductile tearing and grain boundary attacked by corrosion were noted during the electrochemical dissolution experiments and this impact should not be disregarded. Numerous micro cracks 20 µm to 50 µm long were detected at the surface and propagating from the base of the cavities. The micro crack propagation is initiated at the periphery of α phase region, preferring the sites opposite $K_{iii}$ and $K_{iv}$ precipitates. Figure 60 shows the initiation stage of micro crack propagation and the penetration of the ductile crack paths during the process. The morphological characteristic is similar to that found by Darras (2005) who found that oxidation of α phase opposite to the K precipitates was present during the experiments conducted with seawater. The grain boundary attack was experienced on the cavitated specimens, suggesting selective phase corrosion as the gauge of the micro crack activities. Nevertheless, taking into account, the intermetallic (such as the K precipitates) that is harder than the solid solutions influences cavitation stress by inducing the micro defects.

Figure 61 earlier shows the cast NAB specimens, which exhibit an increase of cathodic and anodic current by a small magnitude and the anodic current increases slowly parallel with the increment of the potential without demonstrating an active-passive transition for the processed specimens. The outcome signifies that the alteration of the microstructure grain size has affected the mechanical properties and corrosion erosion characteristics. The reduction of corrosion rate strengthens the view that the CNC FSP and FSP samples with their new properties show better corrosion resistance. The results have shown the corrosion testing for the nickel aluminum bronze alloy using robotic FSP.
Figure 60: Sample of Specimen after Polishing (0 Hour)

Figure 61: Cast NAB during Cavitation Erosion
Figure 62: Cast NAB after 12 Hours of Cavitation

Figure 63: RFSP Surface after 12 Hours of Cavitation

Figure 64: Micro Cavity and Wormhole
In terms of cavitation, the advantage of FSP is evident in enhancing the cavitation erosion of NAB since homogeneous microstructure and fine grains are obtained during friction surfacing. This approach is broadly used to optimize microstructure modification in cast materials, wrought and powder metallurgy in terms of its versatility, energy efficiency and environmentally-friendly features.

5.4.2 Rate of Mass Loss (Cumulative Mass Loss)

The cavitation graph shows the same trend of gradual mass loss for all the samples (Figure 64). The lower spindle speeds of 1000 RPM and 2000 RPM shows a cumulative mass loss of 30.6 mg (2.6 mg cm$^{-2}$ h$^{-1}$) and 30.1 mg (2.6 mg cm$^{-2}$ h$^{-1}$) respectively. The higher rotation speeds of 3000 RPM and 4000 RPM show a lower cumulative mass loss of 14 mg (1.1 mg cm$^{-2}$ h$^{-1}$) and 11.7 mg (0.97 mg cm$^{-2}$ h$^{-1}$).

A clear steady state of erosion has been established in the samples. The lower rotation NAB samples show a greatest mass loss of 54 percent when tested and compared with higher rotation samples, which denotes that cavitation was reduced when the surface was subjected to high speed spindle rotation. The results reflect that a high rotation technique produces better cavitation resistance and better mechanical properties (Culpan & Rose, 1978; Fuller et al., 2007). Nevertheless, Wood and Hutton (1990) have suggested that the cavitation and corrosion is the outcome of a synergy effect which comprise of cavitation erosion and also speeded corrosion. To establish the synergy, Wood and Hutton (1990) established a graph for the ratio of synergistic wear to corrosive wear against the ratio of erosive wear to corrosive wear. An evaluation of the results suggested a relatively greater level of synergism present during the

![Optimum Cumulative Mass Loss](image_url)
process. In these circumstances, the synergy effect has increased the mass loss (erosion rate). Therefore, the instantaneously passivated cavitation endures to be oxidized at a faster rate due to new surface created.

The present study has shown that FSP increases the revolving speed and number of FSP cycles. The influence of dissolution particles reduces the influence of available sites for the galvanic connection. Additionally, the microstructure cast of NAB categorizes the complex phases and phase formation sequence. It has been examined that prior studies have supported the application of FSP in improving the mechanical properties and reducing cavitation (Li et al., 2004). On the contrary, the same improvement can be made by applying other methods, such as cathodic protection to the NAB surface. With cathodic protection, the cast NAB surface is covered with a layer of cathodic gas, which will cushion the vibratory bubbles from giving maximum impact to the surface and eliminates electrochemical dissolution (Preece, 1979). A similar analysis of cavitation under the effect of cathodic protection has been undertaken and the outcome shows that the mass loss of nodular iron was reduced to half (Gouda et al., 1991).

Even though nodular iron is a different metal compared to NAB and the corrosion mechanism is different, it could be presumed that the eradication of the electrochemical corrosion via cathodic protection is not necessarily the cause of mass loss degradation. A study by Auret et al. (1991) seems to indicate that the development of cathodic gas during the process results in less damage at the surface and is dependent on the cathodic current.

The current study shows that the higher rotation speeds of the spindle will produce less mass loss than the lower rotation speeds. When the NAB sample was processed at a speeds of 1000 RPM and 2000 RPM, the surface peak temperature was observed to be around 830 °C. For the speeds of 3000 RPM and 4000 RPM, the surface peak temperature detected was around 1030° C. The higher rotating spindles generates heat by friction and surface of the NAB, which exhibited a fine grained homogeneous microstructure containing lamellar and globular α phase surrounded by quenched β phase. It is easily seen that the grain size at the higher rotation exhibit an average of 3 µm and; therefore, the lower rotation speed exhibits a smaller grain size of an average of 5 µm. When smaller grains were observed, the microstructure became very compressed and hard and the tendency to resist cavitation was much higher. Another inevitable factor is the microstructure refinement that occurs during FSP and manages to eliminate the surface porosity and defect. This produces a much more stable surface and eliminates weak points at the surface. Weak points are a source of cavitation and corrosion initiation.
Temperature variation is closely related to the hardness obtained. As discussed by Preece (1981), grain size recrystallization is independent of the time for cooling. When the surface experiences a surge of temperature, and when the tool moves away from the processed area, the surface will experience a sudden drop of temperature and cause distortion of the grain growth. Thus, a smaller grain is obtained producing a hard surface and a reduction in the mass loss of cavities.

Highly packed grain structures are constructed from the processed surface of high cooling rates that influences FSP and assists in modifying the mechanical properties of NAB. It is deemed that formations of potential and practical inert illustration is formed through upward grain boundary densities. Further, fine grain is merged with the closed packed structure of FSP as well as constructs enhanced surface mechanical properties. The first layer of microstructure surface is regarded as most crucial and emerged complicated from the scratch among NAB surface and FSP tool. The heat of the scratch process is being influenced from the microstructure surface of NAB and; thereby, plastically distorted throughout processing.

5.4.3 Cavitation Erosion

In addition, no mass is lost during the first phase of incubation period as vibratory waves are separated into intermetallic bonds and oxide layer among the globular grains. Extent of the surface bonds have been weakened and damaged throughout the second phase where mass loss is determined and significant. The influence of cavitation erosion emerged to promptly beyond the ultrasonic probe for both FSP and NAB. Therefore, the cavitation erosion was entered deeply into the microstructure surface throughout this phase.

There is no loss observed from the cavitation mass in the incubation stage. The reason behind this loss is the presence of vibratory waves, which diminishes the intermetallic bonds and oxide layers among the grain structure. A significant and consistent cavitation mass loss is observed in the second stage of the accumulation period. This stage has shown that number of surface bonds are deteriorated and fragmented due to resistance of oxide layer resistance.

The difference in mass loss among microstructure cast of nickel aluminium bronze and FSP became more evident when cavitation erosion testing surpasses 12 hours (Ahmad et al., 2014). The Tafel model provides information regarding the corrosion rate, passivity and pitting susceptibility. The corrosion rate for nickel bronze alloy is shown in figure 65 through Tafel plot.
The large existence of large cavities has been exhibited with very rough surface of nickel aluminium bronze as pale colour is produced from yielding scratch. The entire surface colour did not reveal pale colour on the cast sample as well as less visual evidence of cavitation is being displayed from FSP sample. The resistance in mechanism is produced by grain size reduction, which consequently reveals the surface damage and erosion. The augmented corrosion resistance of FSP to nickel aluminium bronze is categorized from the following aspects. Electrochemical corrosion measurements are dependent on the grained microstructure of the nickel bronze alloy, which is illustrated with the help of Tafel equation. The over potential rate of corrosion is shown in figure 66.

Deeper corrosion paths for the ions is endowed from the grainier microstructure, which illustrates the probable solutions to attack the intergranular structure. Cavitation corrosion factor has been ascribed from the catalysts including absorbencies, impurities and imperfections (Song et al., 2013). The surface of microstructure cast is revealed from the imperfections of corrosion effect, which develops easy ways to resolve corrosive effects to actively damage the intergranular structure and lower surface. In this regard, a concentrated catchment region is provided from these imperfections, which normalize the damage occurred during the FSP. If the corrosive ions are highly active, the attack might be much significant and potential at this phase. Less imperfections and lesser catchment areas are closely bounded by FSP of nickel aluminium bronze microstructure in order to normalize the eroding damage (Song et al., 2013).

The effects of cavitation erosion have been shown from the results, showing slightly surface signs for pale and rough colouring. The samples of FSP and NAB alloy are evidently observed from the occurrence of large surface cavities. These samples are shown in the form of dark coloured marking pattern under the probes. Therefore, the cavities were infiltrated into the microstructure surface at the end of this stage.

The cavitation mass loss occurrence and FSP NAB samples became evident after 10 hours of cavitation erosion testing. The occurrence of large cavities in the rough surface exhibits cast NAB as a pale colour, which is generated through ductile tearing. Visual evidence is minutely evident from the cavitation and ductile tearing in the FSP samples and; therefore, the surface color did not show pale color as the cast sample. The results have shown that the average mass loss rate for the cast NAB is greater as compared for the FSP sample. The mass loss rate for
the cast NAB is 490 micrograms/hour whereas, the mass loss rate for FSP is 215 micrograms/hour.

Low dislocation density fine grain microstructure elements are resulted from the generation of dynamic crystallization. The tool pin is used for the stirring and mixing of the material in the stir zone. The root side of stir zone reflects the cavitation testing for the FSP NAB. The occurrence of the cavitation testing was found from optical micrographs. The results have shown the heat generation for specific samples in the FSP. The detection of stir zone in the process was found to be error free from cavitation interface. The improper material flow across the tool pin is circulated at the cavitation process of the stir zone (Mahoney et al., 2003). The production of root side cavitation is emerged from the pin geometry. The results have illustrated the macro and microstructure elements of the samples in the FSP. The adherence of cavitation testing is reflected at the mid stage of the stir zone interface. The microstructure behaviour possessed in the stir zone is similar to the cast NAB structures. The difference of the material flow has resulted in the cavitation testing of the process. In the FSP, cavitation is regarded as a dominant defect, which negatively influence the selection of the tool parameters including traverse speed, tool tilt and rotation speed. The interfaces of the stir zone reflect the placement of the cavitation testing. The FSP was indicated from the inadequate flow of the material. It has been investigated that the process parameters throughout FSP as it depends on the level of the cavitation. Cavitation is generated in the low heat input samples due to the presence of low thermal cycle (Field et al., 2001).

Long-term period is required to instigate and penetrate the FSP microstructure and for the corrosive ions as there is no such factors to commence the corrosion. Thereby, FSP of nickel aluminium bronze is significantly swelled the corrosive resistance from this aspect. The granular microstructure as well as inhomogeneous microstructure of cast NAB is exhibited from the phases of cavitation erosion in which corrosion is promoted from exposed grains. This implication allows corrosion to ease the process of eroding liquid to damage. The morphology of the phases has been modified and isolation is enhanced in FSP of NAB, which consequently results in the increase of erosion resistance. The electrochemical, salt spray corrosion and immersion resistance of alloy in NaCl solution is augmented by FSP. It is being adhered that revolving speed along with number of FSP cycles is augmented when there is increase in the corrosion resistance (Qin et al., 2016). FSP is illustrated through Tafel plot associating with
the extent of electrochemical reaction to the potential rate. Figure 67 has depicted the FSP of nickel bronze aluminium through Tafel plot.

5.5 Corrosion

5.5.1 Electrochemical behavior

The specimens were tested with a 3.5% NaCl aqueous solution and a potentiostat apparatus with its software. A 6 mm diameter aperture was prepared on the surface for the chemical reaction (Figure 68). A potentiodynamic polarisation graph and other relevant details were able to be plotted and tabled for all samples. The software was able to calculate the open circuit potential (OCP), corrosion potential ($E_{corr}$) and the corrosion current ($I_{corr}$). Using the Butler-Volmer equation, the corrosion rate or MPY was able to be calculated. Table 4 shows the details of the Potential, Current and MPY.

<table>
<thead>
<tr>
<th>Table 4: Details of Potential and Current</th>
</tr>
</thead>
<tbody>
<tr>
<td>OCP (V)</td>
</tr>
<tr>
<td>As Cast NAB</td>
</tr>
<tr>
<td>RFSP</td>
</tr>
<tr>
<td>CNC 1000 rpm</td>
</tr>
<tr>
<td>CNC 2000 rpm</td>
</tr>
<tr>
<td>CNC 3000 rpm</td>
</tr>
<tr>
<td>CNC 4000 rpm</td>
</tr>
</tbody>
</table>

Figure 66: Potential OCP For CNC and RFSP
It can be observed that the anodic current increases slowly with the increase of potential without exhibiting an active-passive transition for cast NAB or the other specimens. The RFSP and CNC/FSP process increases both the cathodic and anodic currents by a small magnitude, which results in an active shift of the corrosion potential towards the passivity zone. The corrosion potential for the cast NAB, CNC FSP and RFSP were observed and plotted in the graph illustrated in Figure 66. A shift of -0.032 V for RFSP and -0.0533 for CNC FSP was gained slowly in the noble or cathodic direction. This is an indication of corrosion improvement because as the potential is shifting towards zero potential, the tendency for corrosion will be lessen (Oh-ishii, Zhilyaev & McNelley, 2006).

Corrosion rate is the speed at which metals undergo deterioration within a particular environment. This rate depends on environmental conditions and the condition or type of metal. Using the corrosion current \( I_{corr} \) and the corrosion potential \( E_{corr} \) calculations were made and the corrosion rate MPY obtained. The MPY for NAB was 216 and for RFSP and CNC FSP the corrosion rate was 100 MPY and 108 MPY respectively. The RFSP and the CNC FSP process appears to reduce the corrosion rate significantly. The reduction of the MPY was due to the changes in the microstructure that occurs during the FSP process. As said earlier, FSP eliminates porosity and defects at the metal surface. Because of this, the corrosion concentration area is eliminated resulting in a lower corrosion potential and thus a more passive surface is obtained.

5.5.2 Electrochemical Behavior of Cast NAB, CNC FSP and RFSP NAB
The OCP of the CNC FSP and RFSP specimens show a lesser value of activation compared to the cast NAB sample \( E = -0.427 \text{ mV} \) as seen in Figure 62. This behaviour is expected since

![Figure 67: Potentiodynamic Graph](image-url)
the grains of cast NAB are much larger with an average between 100 µm and 200 µm (Figure 66) while the grain size of the processed specimens was at an average of 2 µm to 15 µm. The Hall-Petch equation denotes that the smaller grain size will result in better mechanical properties (Nieh & Wadsworth, 1991) Similar findings have been reported by Song (2015) who observed the reduction in grain size using the FSP as it gives a better potential for the samples and; thus, better corrosion resistance.

The corrosion testing was investigated in the filtered seawater and depicted through the electrochemical polarization graphs. The results have shown a typical Tafel behaviour for the FSP and cast samples. The frequency of applied potential difference was clearly shown by considering the saturated calomel electrode. The determination of corrosion potential was widely viewed from the minimization of corrosion current. The corrosion potential for FSP is -0.125 V; whereas for cast NAB, it is -0.169 V. At the corrosion potential, the similarity for the measured current jots were measured in the corrosion testing (Mahoney, 2005). It has been revealed that the augmented corrosion resistance of robotic FSP to NAB alloy is categorized from multiple aspects.

The first factor shows the enhancement of corrosion resistance from the contribution of finer grain structure of robotic FSP as compared to the coarser grained microstructure of cast NAB. In order to attack the surface, the potential porosities are closely associated with the finer grain structures as well as corrosive points. The observations acquired from the results are similar to the one witnessed from the experimental results of past study (Mishra, Ma & Charit, 2003; Oh-Ishi & McNelley, 2004).

An inevitable outcome of the immersion of metal in NaCl or seawater solution is the formation of an oxide layer at the surface, which will affect the corrosion potential and the current. Schüssler and Exner (1993) has undertaken extensive study regarding the reaction of NAB alloys in seawater. The oxide film present on the surface consists of $Cu_2O$ layer and the thickness is around 800 nm. The drop-in corrosion current relative to time was expected from the simultaneous reduction in the anodic reaction rate with the increment of the $Cu_2O$ layer and the cathodic reaction for the surface of both samples. Furthermore, the film formation is presumed to be caused mainly by the presence of aluminium infused together with the $Cu_2O$ lattice and hence a decreased rate of oxygen reduction was formed. The oxide layers are comprised of magnesium oxide, aluminium oxide, copper oxide and iron oxide. The $Cu_2O$ layer dominates the outer layer while the aluminium oxide is underneath the $Cu_2O$ layer.
(Figure 65 shows the EDS analysis). As expected, a larger grain will produce more and thicker oxide compared to a small grain and this directly affects the corrosion potential of the samples. The steady increment of the anodic current relative to the increment of the potential can be attributed to a mass transfer occurrence and related to the build-up of soluble oxide product throughout the anodic polarisation and diffusion processes during the oxide film formation. Aluminium is dominant between the oxidation layers; therefore, the formation is mainly based on the aluminium ion diffused in the oxide phase as suggested by Schussler (1983). A larger grain will exhibit a bigger mass transfer due to the large surface area and imperfection of the structure while a small grain with a compact structure will exhibit a lower mass transfer and a reduction of corrosion effect. The results showed that the size and distributions of the micro constituents influence the depth of attack.

The corrosion paths for the ions are provided from the coarser microstructure to outbreak the inter-granular structure. The depth of corrosion path is reduced from the FSP NAB alloy; therefore, it heavily minimizes the effects of inter-granular attack and augments the corrosion resistance factor.

The corrosion effect is regarded as a catalyst due to porosities, scars, impurities and imperfections. The corrosive solutions are easily compared from the larger surface of imperfections, which actively outbreak the inter-granular structure and the lower surface. The outbreak will be investigated through the provision of imperfections in the concentrated catchment for the ions. At this point, the faster and stronger surface will be observed for the outbreak due to highly active corrosion ions. These imperfections are offered by the fine bounded FSP NAB microstructure in order to reset the corrosive outbreak.

In this regard, a longer time will be required to reset as well as penetrate the FSP microstructure for the corrosive ions as there is no active area for the corrosions. Thereby, the corrosive factor of FSP is significantly multiplied from this aspect. The demonstration of an inhomogeneous and coarse microstructure in the K phases significantly supports the corrosion rate on the basis of exposed grains structure. This aspect is easier for FSP to outbreak the corrosion point. The modifications of the K phases can be observed from its morphology, enhancing the segregation as well as the corrosion resistance in FSP (De Backer et al., 2012).

5.5.3 Linear Polarization Graph

Figure 63 shows a potentiodynamic polarization of processed FSP NAB alloys as a function of different parameters measured using the linear polarization technique. The outcome shows a
clear resemblance in the polarization curves of the different processed specimens. It can be observed that for as-cast NAB the potential was -0.4843 V; the polarization resistance was higher than for the other processed specimens. The graph shows the common shift of potential to the left which indicates the improvement of corrosion resistance for all the samples relative to the as-cast NAB. Even though there are no particular studies done to established the effect of microstructure variation between the intermetallic compounds. The K phases and the grain boundaries oxidation behaviour of the as cast NAB with the processed samples shows that the polarization resistance curves of the specimens are directed towards the fact that some phases are more protective to the corrosion effect.

The accelerated cathodic reaction is the outcome of the microstructure with a lower corrosive value determined by the reaction of the Fe and Cu atoms with the other components during the experiments. When samples were subjected to sudden cooling from 900°C, a greater grain density was established resulting in a reduced cathodic reaction rate (Huang, Tsai & Lee, 1996) while the coarse grain with lower grain density will result in a coarse. Widmanstätten phase produces a higher cathodic reaction rate and a lower oxidation resistance. However, the existence of Fe atoms raises the corrosion resistance behaviour of the cast NAB alloys. Kear et al. (2004) have shown that with the increase of Cu content a direct effect on the cathodic polarization attributes that lowers the rate of cathodic reaction during the oxidation process; hence, reducing the corrosion rate. The polarization resistance curves of the processed specimens were higher than of the as-cast NAB. This is due to the higher Al and Cu content present in the specimens providing an enhancement of the phases that effect the corrosion resistance of the processed samples by giving a higher passivity at the surface. It has been investigated that electrochemical, salt spray corrosion, and immersion resistance of NaCl solution are increased through robotic FSP.

A number of FSP is increased as the corrosion resistance increases. Furthermore, this increment reveals the augmentation of FSP rotational speed. The number of sites accessible for the galvanic coupling is reduced from the effects of dissolution particles. The ability to sustain the contact pressure on the basis of axial force is determined as a core requirement of robotic FSP. The results have shown that axial force is capable to endow the welding torque for stirring the weld material. The restriction in the robotic FSW is emerged from the axial force requirements, showing the influence on the hardness of the NAB alloy. The desired axial force can be obtained by simply replacing the rotational tool piece into the work piece. The volume of
machine weld will be judged on the basis of rigidity and precision of the machine tool. However, the rigidity at such extent cannot be assumed with an implemented conventional tool. By considering the polarization graph, it can be seen that the FSP could affect the outcome of the linear polarization graph, which produces a higher electronic and ionic resistance during the experiment. The processed surface signifies that it is more established and protected. The linear polarization curve investigation discloses the active-passive attributes of the samples. After leaving the passive range, a sudden upsurge of current density happens in which a protective layer is destroyed and pitting corrosion started at the surface. During the process, the $i_{corr}$ escalated gradually with the intensifying potential and; therefore, normal active-passive transition was not detected. This could be the effect of selective phase corrosion.

The higher speed range (3000 RPM–4000 RPM) provides better passivation attributes relative to the lower speed range (1000 RPM–2000 RPM). Figure 63 shows that K phases were less attacked signifying that they were more cathodic relative to the Cu-rich α phase. The persistent dissolution of α phase is due to gradual loss of the K intermetallic compounds. A report by Culpan suggests that the corrosion of the as-cast NAB alloys is mainly galvanic in nature. The Cu-rich phase was clearly seen to be attacked at the boundary of the intermetallic phases in the NaCl aqueous solution (Grewal et al., 2013). The effect of cavitation on the surface, such as a rough surface, micro cracks, worm hole and ductile tearing are catalysts to increase and speed up the corrosion process. Micro cracks with an average size of 700 µm exist in the Cu rich phase and were observed opposite to the intermetallic phase. It is predicted that the selective phase corrosion and cavitation stresses were the origin of the micro cracks (Al-Hashem & Riad, 2002). The results derived from the Figure 23 show the potentials of the higher speed range that are compared to the lower range signifying that the higher range provides better corrosion resistance than the lower range. It shows a better combination of lower current density, lower potential and a better passivity. The effect of FSP on the polarization attribute for the cast NAB depends on the constituents of the specimens. The existence of Cu and Al atoms in the NAB microstructure will result in a shift of the polarization curve due to the involvement of Cu and Al atoms and the increment of the Cu atoms in the solid solution.

5.5.4 Corrosion Characterization

The extent of sites accessible for the galvanic connection is reduced from the influence of dissolution particles. The optimization of microstructure cast has illustrated lower corrosion extent as compared to microstructure cast sample including the improvement of the phase’s
isolation, reduction of penetrability and modification of grain size. It is examined that gas filled cavities might be revealed in the form of liquid when liquid access in a low-pressure region. The cavitation erosion might collapse from the activities of irregular collapse uncertainties, which produce higher intensities when these erosions are placed to flow in the regions of higher pressure (Song et al., 2016). The weight loss extent of the microstructure cast and FSP of nickel aluminium bronze is illustrated from three aspects. Throughout the test, nickel aluminium bronze cast is promptly higher in weight as compared to FSP. Moreover, the time of FSP is increased as the sample decreased with the loss rates of microstructure cast. No significant difference has been illustrated from the electrochemical polarization behaviour of the NAB by exhibiting the microstructure cast and FSP samples (Cottam et al., 2013). These microstructures cast and FSP is usually revealed through ductile tearing, which describe the ultimate failure of ductile materials. Figure 67 have shown the ductile tearing fracture of NAB.

![Figure 68: Ductile tearing](image)

In an apparent illustration, the cathode functioning is regarded as a controlling step to control the rate of entire corrosion process. It is deemed that oxygen reduction reaction was mainly the dominant factor in the overall reaction to complete the cathode process. The cathode curves were approximately analogous to the horizontal axis, which illustrates the characteristics of restricting diffusion density on the executed potential range of FSP. This illustration of characteristics was normally relied upon the diffusion of oxygen. It has been observed that corrosion potential in the imperative direction is modified from the cathode minimization reaction extent, which reduces the corrosion potential. The formation of the probable deprived surface film is indicated from the existing density of applied voltage and afterwards it augments the enhancement of the executed voltage. Slightly higher corrosion rates as well as lower
corrosion existing density is illustrated from microstructure cast, which ultimately associates with superior electrochemical corrosion resistance as compared to FSP (Song et al., 2013). The results have suggested that understanding the process parameters, tool geometry, and weld material develops the working process of the robotic more efficiently.

The conditions of the FSW develops the optimum operating aspects to understand the relationships. The instigation of weld pitch can be reflected from the extent of rotational speed subjected to travel speed. The increased extent of the weld pitch can be obtained if rotational speed of the tool is increased or traverse speed is decreased. The experimental results of the axial force are illustrated from the deliverance and consistency of welding parameters.

The measurement of the axial force is associated directly with the mechanical models. The results have shown the increment in the weld pitch from the measurement of axial force. The escalation in the axial force is adhered from the traverse speed and constant rotational speed. On the contrary, there is no significant association between the increased traverse speed and increased axial force. The implementation of robotic FSP is widely understood in demonstrating this relationship. High rotational speeds and low traverse speeds are required from the optimum operating parameters for robotic FSP. Rotational speed at the higher extent is significant in illustrating the implementation of weld material.

The measurements of FSP were executed in order to determine the consequences of electrochemical polarization behaviour of the nickel aluminium bronze curve. In addition, high frequency capacitance and low frequency impedance were comprised as two significant components in the microstructure cast and FSP samples. The attributes of surface illustration was revealed from high frequency capacitance at the edge of solution and the matrix. On the contrary, the cast sample was illustrated broadly from the FSP samples, which indicated that corrosion rate for FSP was upward as compared to microstructure cast sample (Farmer et al., 2013). It has been examined that restriction among low frequency and high frequency impedance were not obvious for the microstructure cast as compared to the FSP. The complex phases and phase formation sequence were categorized from the microstructure cast of nickel aluminium bronze. Furthermore, FSP was initially deployed into the beta phase in which heating and deformation throughout FSP results to recover and recrystallize the deformed alpha matrix and spheroidization of retaining lamellar. The microstructure cast in the FSP of nickel aluminium bronze is comprised of the dominant parts of fine and equivalent alpha grain (Küçükömeroğlu et al., 2016). Energy dispersive spectroscopy (EDS) is the most effective
technique for analysing and investigating the elemental surface component of corrosion-related samples. Figure 68 is illustrated to examine the corrosion samples through EDS. The appropriateness of single application is evidently revealed from the implementation of robotic FSP. The use of force control in the robotic FSW is approached to reveal the plunge depth; whereas, the traverse speed is adjusted on the surface of welding joint. The illustration of forging and consolidation of stirred material ensures the convergent of the axial force. The accomplishment of axial force in the FSP is contacted from the passive control force, showing the effective adaptation of axial force in the nickel aluminum bronze alloy. The performance of the robotic task is showed on the basis of active control force where appropriate control forces are applied to accomplish the FSP controls (Hovanski et al., 2015). The performance of the robotic FSP for NAB alloy is revealed from the deviation of the design tool. The enhancement of the weld quality is observed from the implementation of FSP for robots. The measurement of robot deviations was computed through vision and laser sensors to approach seam-tracking and pre-heating techniques of welding joints. The adjustment of structural robotic manipulator allows the control process of force feedback. It has been assumed that a linear elastic environment is implemented to control the axial force. On the contrary, the functions of the welding parameters are based on non-linear and non-elastic FSP work piece. The increase in the weld pitch can be followed from the volume of axial force. The increase in welding torque will reduce the weld pitch. The exploration of welding materials has focused on the implementation of robotic FSP. An appropriate contribution is observed from the characteristics of FSP on the nickel aluminum bronze alloy (Mishra & Ma, 2005).

Figure 69: EDS corrosion
Magnesium alloys were consequently explored from the influence of displacing microstructure phases of FSP. The dissolution of cavitation erosion from the cast of magnesium alloy has been similarly attributed from the influence of heat treatment solution revealed at higher temperature for longer time. The severe composition segregation is improved through FSP in order to influence the corrosion resistance significantly. The previous study has illustrated significant implication of cavitation corrosion by considering the corrosion of copper in chloride phase (Akinlabi Andrews & Akinlabi, 2014). The complication of corrosion of copper was illustrated from the formation of surface films. It is well judged that number of electrode procedure of NABs are anodic in nature to construct the cuprous dichloride anion in the form of cathode process and oxygen reduction. The mobility of corrosion product is revealed from the restriction of corrosion layers in which sustainable protective layer is constructed on the basis of alloy surface. The corrosion attributes have been studied by examining the short-term electrochemical techniques, longer-term immersion trials and impingement studies. These studies and attributes were aligned with the attributes of heat-treated, wrought and cast NABs. The elimination and reduction of corrodible beta phase as well as augmented extent of fine phases of alpha phase is resulted from the appropriate heat treatment (Vikash et al., 2015).

5.5 X-ray Diffraction Results and Discussion

X-ray diffraction is regarded as a non-destructive analytical technique, which is used to acquire information on the basis of revelation and quantification of assorted crystalline components. The categorization and quality control of structural materials are measured through the use of X-ray diffraction. It has been examined that three-dimensional reciprocal space is formed from the orientational averaging causes. In the diffraction process, the sample orientation is rotated in order to mitigate the effects of texturing.

The atomic electrons and their oscillations are generated through an outgoing wave, which moves the electric field of an incident X-ray in a diffraction experiment. It has been revealed that the positively charged core of the atom is interacted with the charge of incident electron, which generates electron wave-function. Different physical mechanisms are involved in the diffraction processes in order to measure the unpaired electron spins. Figure 69 has shown the X-ray diffraction analysis for the samples in which 3 peaks were detectable.
These components are referred as phases, which are shown in the material to illustrate the versatility of crystalline compounds. In addition, x-ray diffraction is used to classify the structural method associated with crystallographic planes of the structure. It has been notified that diffraction of structural components are dependent on the basis of diffracted radiation intensity and double diffraction angle. X-ray diffraction analysis has been used to determine the compositional phases of nickel aluminium bronze. In the prior study, X-ray diffraction analysis was used to compute the proportional detector with the help of single channel. The samples used for the analysis were copper alloys, which shows the diffraction pattern obtained after annealing from the software (Cottam, & Brandt, 2014).

X-ray diffraction is a process in which radioactive patterns are converted without modifying the wavelengths. This process interferes the assistance of number of reflective lattice in order to observe spatial directions features. In addition, diffraction peak and maximum plan is associated with indexing diffractometry. After quenching heat treatment, X-ray diffraction reveals the diffraction patterns for the nickel aluminium bronze. Furthermore, the NAB samples with certain size were conducted through ambient temperature after applying the heat treatment. The results of X-ray diffraction provided that samples contain minute oscillations after applying the heat treatment. Figure 70 has shown the X-ray diffraction results for the sample and compound database. The analysis has shown that there were no detectable crystalline peaks found for the compounds.
The analysis of X-ray diffraction executed for initial and final phase of the FSP revealed that there was paucity of modifications in the compositions of analysed materials. In this regards, the surface treatment entails the conditions of the phase stability. From the analysis, it has been indicated that the solid solution and inter-metallic phase was present in the FSP. The analysed samples further revealed that there were no diffractions found in the content of nickel aluminium bronze. This measurement was shown from the low detectability threshold of the X-ray analysis. On the contrary, the existence of originated reflections were found from the presence of magnesium oxide. The reason behind this presence is the strong affinity of magnesium, which specifically allow oxide phases to determine lack of gas shield throughout the treatment (Mungsuntisuk, 2013). The reflections from certain phases were intensified from the investigation of stirred material. The texture formed in the material causes the intensification of diffraction reflections. Figures 71 and 72 have shown the percentage of compounds used for the FSP. The results of XRD analysis have shown that the presence of aluminium copper was 57.3% whereas the presence of manganese nickel was 42.7%.

Figure 71: XRD Results for Samples and Compound Database

Figure 72: Percentage of Compound (S-Q)
It has been analysed that the increased temperature effects and increased pressure effects were formed through the specificity of FSP in order to generate structural changes. From these changes, it is clear that the occurrence of preferential material orientation is reflected by the structural conditions. X-ray analysis was used in this study to measure the hardness and resistance of the nickel aluminium bronze for FSP. From the results, it has been signified that there was significant increase in the hardness of the FSP. The augmentation of hardness in the changed layer is observed from the homogenization of the material and microstructural refinement. When a rotational speed is increased, the slight change in the micro-hardness of FSP was also observed. It has been indicated that increase in rotational speed drives to a reduction in the hardness due to the microstructural changes (Grewal et al., 2013).

By considering assorted rotational speeds of the tool, the microstructure hardness is associated with the differences in the grain size. When changing the hardness slightly from the surface, individual samples at different conditions are notified from the results. Throughout the friction modification, this investigation might be associated with the material temperature. The increase in grain growth can be led consequently by high temperature whereas heat zone can be affected from the reduction in hardness.

The patterns of X-ray analysis have revealed the microstructure and mechanical changes in the FSP. These changes were investigated using the samples of NAB. It has been observed that numerous crystalline peaks are emerged for the FSP whereas no detectable crystalline peak was found for the melted part. Due to the low content of aluminium and bronze, the resemblance of nickel aluminium bronze was reflected same as the FSP alloys. In order to
found more strength, the X-ray diffraction patterns were replaced by multiple phases. The increase extent of FSP patterns allow crystalline peaks to decline and broaden from the diffraction surface. On the other hand, the slight increase in the aluminium and nickel contents reveals the variation of hardness values (Sabbaghian et al., 2014).

It has been explored that X-ray diffraction is used to measure the residual stress by deploying measurements of stress measurements and chromium diffraction. The measurement was done to verify the lattice spacing using a linear function by acquiring plane stress linear elastic residual stress model. The integration of diffracted intensity was measured from the samples in order to reduce the impact of the grain size. An automated translation device was used to measure the residual stress distributions. The stress distributions and residual stress measurements were documented through X-ray diffraction analysis. The variation of hardness is revealed by producing HAZ in the FSP, which measures the strength of weld centre (Thapliyal, & Dwivedi, 2016).
CHAPTER 6: CONCLUSION

The study aimed to explore the parameters of FSP for the robotic FSP and computer numerical control application. In this regard, robotic FSP was explored and analysed using NABs. The reason behind using robotic FSP was the significance of technique, which provides enhancement for local metal surface. On the basis of these objectives, variation parameter series is deployed including traverse speed, rotation speed and plunging force. The influence of both robotic FSP and computer numerical control is also investigated to determine the hardness and microstructure of the NABs. The results obtained are effective in concluding the comparison between robotic FSP and CNC FSP.

The study further aimed to investigate the influence of cavitation erosion and corrosion of the NABs by considering continuous corrosive medium and vibratory waves. Thereby, optimization parameters were used to analyse the direction of cavitation erosion and corrosion. The relation of optimum parameters for both FSP and CNC FSP was determined through X-ray diffraction analysis. The results of the analysis reflected that FSP is concerned using the optimization parameters.

The entire focus of this study aimed to enhance the mechanical, corrosion and cavitation erosion features; therefore, the results obtained from the analysis were significant in concluding the objectives. It has been analysed that the specimens subjected towards the FSP considerably analyse the microstructural evolution. Moreover, the hardness of the specimens were related with the cavitation erosion and corrosion attributes. Microstructure hardness of nickel aluminium bronze was illustrated through the precipitates, which comprises of globular eutectoid decomposition commodities. It has been investigated that the microstructures of friction stir operating functioning are associated with the layers of cast NAB. The accomplishment of FSP for nickel aluminium bronze refines the grain structure. Therefore, the process of NAB increases the tensile properties, hardness, and fatigue strength. The microstructure surface of NAB influences the heat of the scratch. The loss of cavitation mass existence throughout distinct phases, which addresses the progression of FSP. The determination and significance of mass loss is weakened and damaged by considering intermetallic bonds and globular grains. In addition, the microstructure surface is associated directly with the grains of cavitation erosion. The restriction of corrosion layers reveals the mobility of corrosion product, which influences the sustainable protective layer of alloy surface. The appropriate heat treatment was used to
eliminate and reduce the corrodbile beta phase and fine phases of alpha phase. Computer numerical control machine was used to conduct the FSP for the vibration analysis. The specimens were controlled in the axial force by using force sensor within numerical control machine. Shorter penetration period is illustrated from the results, which allows vibration analysis to acquire higher rotation for better results.

It has been analysed that deployment of control machine independently analysed the plunge depth for the FSP. The process of axial force requires structural integrity and the stiffness of the control machine. In order to articulate robotic FSP, other optimization parameters should be incorporated for the mitigation of vibration waves.

6.1 Summary of Axial Force
The ABB IRB 6660 articulate robot has a small range of available rotational speeds and traverse speeds for the FSP processes. The allowable range is between 3000 RPM to 4000 RPM. The allowable traverse speed was 0.5 mm/s to 1 mm/s. By using these parameters, a low value force (Robot capability of 2500 N) could be applied to the system to complete the process.

If other parameters were to be applied, a severe vibration will be observed that could damage the articulate robot and be a possible danger to the operators. The CNC machine is not as rigid as a conventional FSW machine and; therefore, the FSP process will cause some vibration to the machine but still the overall FSP graph shape is considered acceptable.

It has been concluded that the higher range of the CNC rotational speed produces a similar axial force in the range of 3000 N for the insertion of the spindle. A smoother graph is observed while a lower range of rotational speeds need a very high axial force for the insertion. It can be observed that around 10000 N force is needed for the process.

It has been examined from the results that acceptable parameters for CNC and articulate robot ranges between 3000 RPM to 4000 RPM. Therefore, the traverse speed is recorded at 0.5mm/s to 1.0 mm/s.

6.2 Summary of Microstructure
The FSP process carried out with the CNC and the robotic arm significantly, which refined the microstructure of the cast NAB. The grain size in the BM lies between 150 µm to 200 µm. In the FSP, the average grain size lies between 1 µm to 5 µm. The SZ was categorized to exhibit an inhomogeneous microstructure and was determined by the setup parameters.
The microstructures of the SZ could be characterized into four important zones from the top to the bottom. The first zone at the top exhibits a Widmanstätten α phase. The second zone exhibits a banded primary α and β phase. In the third zone, there is the presence of α phase and β phase with an equiaxed size of average 5 µm. At the bottom, there is a stream-like α phase and β phase.

The banded α phase in the second zone is the outcome of the inadequate recrystallization during the extreme plastic deformation of FSP. There was no significant difference between the CNC FSP and RFSP in microstructure formation. The size and formation of the phases are nearly the same in zone locations.

6.3 Summary of Hardness

It has been concluded that the hardness of the surface varies. The higher range of rotation speed (RPM) with low traverse speed (mm/s) leads to the highest surface hardness. A rotation speed of 3000 RPM with a traverse speed of 0.5 mm/s and 1 mm/s provide the highest hardness readings of $HV_{0.2} = 290$ and 299 respectively. Similarly, RFSP provides nearly the same value of $HV_{0.2} = 280$.

Similar patterns of microstructure phase distributions were observed with same average size of grain. The higher rotation speeds lead to the smaller grain size of an average 3 µm whilst the lower speeds lead to an average size of 5 µm.

To obtain optimum hardness, the rotation speed should be between 3000 RPM and 4000 RPM and the traverse range should be set between 0.5 mm/s and 1mm/s. The axial force should be set in the range of 2500 N to 4000 N.

6.4 Summary of Cavitation

It has been examined that the higher range of rotation speed (3000 RPM-4000 RPM) reduces the mass loss of 54 percent while the lower range reduces to 35 percent of mass loss relative to as cast NAB during the cavitation experiment.

A significant increase in roughness was observed during the whole cavitation process. The average initial roughness (Ra) of the samples was in the region of 5 nm. The final roughness of the samples was 617 nm, 182 nm and 221 nm for cast NAB, RFSP and CNC FSP respectively.

Cracks were observed to start at the boundary of α grains, penetrating to the inner parts of the grain, and consequently develop worm hole and craters.
From the results obtained, it has been concluded that the cavitation optimum parameter for the FSP technique using the CNC or robot is in the region between the 3000 RPM and 4000 RPM. This is in line with the earlier finding that the optimum parameter for hardness is in the region of 3000 RPM.

6.5 Summary of Corrosion

The potential of the as-cast NAB was observed to be 0.4843 V. For the CNC and the Robot, potential was observed to shift towards the cathodic side by -0.0533 V and -0.032 V respectively. As the samples were more cathodic, they were more corrosion resistant. As-cast NAB exhibited 216 MPY while RFSP and CNC FSP (4000 RPM) exhibited 100 MPY and 108 MPY respectively. This denotes a significant reduction of corrosion loss in a year. The optimum parameters that produce best results were in the region of 3000 RPM to 4000 RPM. The overall optimum parameters for CNC and RFSP produces the accepted results in the region of 3000 RPM and 4000 RPM for the pin and the traverse speed is in the region of 0.5 mm/s to 1 mm/s. For the axial force, the optimum range is about 1500 N to 3500 N. The tilting angle does not have any significant effect in the process. If the stated parameters are used then the results obtained are considered as optimum.
REFERENCES


Darras, B. M. (2005). Experimental and analytical study of FSP.


Leica, (2014). Leica Microscope


**APPENDIX**

**Table 5: Polishing and Grinding Table**

<table>
<thead>
<tr>
<th>Step</th>
<th>Surface</th>
<th>SiC-paper#220</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Suspension</td>
<td>Nil</td>
</tr>
<tr>
<td></td>
<td>Lubricant</td>
<td>Water</td>
</tr>
<tr>
<td>1</td>
<td>Time(minutes)</td>
<td>2</td>
</tr>
<tr>
<td></td>
<td>Disc rotation (RPM)</td>
<td>150</td>
</tr>
<tr>
<td></td>
<td>Sample holder rotation (RPM)</td>
<td>150 (co rotation)</td>
</tr>
<tr>
<td></td>
<td>Force (N/sample)</td>
<td>30</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Step</th>
<th>Surface</th>
<th>MD-Largo (9µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Suspension</td>
<td>DP-Suspension (Level3/6)</td>
</tr>
<tr>
<td></td>
<td>Lubricant</td>
<td>Green (2/6)</td>
</tr>
<tr>
<td>2</td>
<td>Time(minutes)</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>Disc rotation (RPM)</td>
<td>150</td>
</tr>
<tr>
<td></td>
<td>Sample holder rotation (RPM)</td>
<td>150 (co rotation)</td>
</tr>
<tr>
<td></td>
<td>Force (N/sample)</td>
<td>30</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Step</th>
<th>Surface</th>
<th>MD-Dac (3µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Suspension</td>
<td>DP-Suspension (Level3/6)</td>
</tr>
<tr>
<td></td>
<td>Lubricant</td>
<td>Green (2/6)</td>
</tr>
<tr>
<td>3</td>
<td>Time(minutes)</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>Disc rotation (RPM)</td>
<td>150</td>
</tr>
<tr>
<td></td>
<td>Sample holder rotation (RPM)</td>
<td>150 (co rotation)</td>
</tr>
<tr>
<td></td>
<td>Force (N/sample)</td>
<td>30</td>
</tr>
<tr>
<td>Step</td>
<td>Surface</td>
<td>MD-Plus (1µm)</td>
</tr>
<tr>
<td>------</td>
<td>------------------------</td>
<td>---------------------</td>
</tr>
<tr>
<td>4</td>
<td>Suspension</td>
<td>DP-Suspension</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(Level 3/6)</td>
</tr>
<tr>
<td></td>
<td>Lubricant</td>
<td>*Green (Level 10/12)</td>
</tr>
<tr>
<td></td>
<td>Time(minutes)</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>Disc rotation (RPM)</td>
<td>150</td>
</tr>
<tr>
<td></td>
<td>Sample holder rotation (RPM)</td>
<td>150 (co rotation)</td>
</tr>
<tr>
<td></td>
<td>Force (N/sample)</td>
<td>30</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Step</th>
<th>Surface</th>
<th>MD-Chem</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>Suspension</td>
<td>OP-S (Level 0/0)**</td>
</tr>
<tr>
<td></td>
<td>Lubricant</td>
<td>Ammonia (2.5mL) + Hydrogen Peroxide (1.5mL) (Level 0/0**)</td>
</tr>
<tr>
<td></td>
<td>Time(minutes)</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>Disc rotation (RPM)</td>
<td>150</td>
</tr>
<tr>
<td></td>
<td>Sample holder rotation (RPM)</td>
<td>150 (co rotation)</td>
</tr>
<tr>
<td></td>
<td>Force (N/sample)</td>
<td>30</td>
</tr>
</tbody>
</table>
Figure 74: LabView Mapping of the Circuit