INVESTIGATION OF PROCESS PARAMETERS IN THERMAL DEBINDING OF 3D-PRINTED 17-4PH STAINLESS STEEL SAMPLES

JOEHAN ARIFF BIN ISMAIL

MASTER OF MANUFACTURING ENGINEERING (INDUSTRIAL ENGINEERING)

2025

Faculty of Industrial and Manufacturing Technology and Engineering

INVESTIGATION OF PROCESS PARAMETERS IN THERMAL DEBINDING OF 3D-PRINTED 17-4PH STAINLESS STEEL SAMPLES

Joehan Ariff Bin Ismail

Master of Manufacturing Engineering (Industrial Engineering)

2025

INVESTIGATION OF PROCESS PARAMETERS IN THERMAL DEBINDING OF 3D-PRINTED 17-4PH STAINLESS STEEL SAMPLES

JOEHAN ARIFF BIN ISMAIL

A report submitted
in fulfillment of the requirements for the Master of Manufacturing Engineering (Industrial Engineering)

Faculty of Industrial and Manufacturing Technology and Engineering

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

2025

DECLARATION

I declare that this master project entitled "Investigation of Process Parameters in Thermal Debinding of 3D-Printed 17-4PH Stainless Steel Samples" is the result of my own research except as cited in the references. The master project has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature :..

Name : Joehan Ariff Bin Ismail

Date :..

APPROVAL

I hereby declare that I have read this master project and in my opinion this master project is sufficient in terms of scope and quality as a partial fulfillment of Master of Manufacturing Engineering (Industrial Engineering)

Signature : ....

Supervisor Name : Associate Prof. Ir. Ts. Dr. Shajahan Bin Maidin

Date : ....

DEDICATION

To my beloved mother and father

Wife and daughters

Siblings and frineds

Respectful supervisor and lecturers

ABSTRACT

The thermal debinding process is a key stage in metal additive manufacturing, significantly influencing the quality and properties of 17-4PH stainless steel components produced using Fused Deposition Modeling (FDM). This study investigates optimal thermal debinding parameters aimed at effectively removing binder materials while maintaining the structural integrity of printed components prior to sintering. Samples were fabricated using Ultrafuse® 17-4PH stainless steel filament on an Ultimaker S5 3D printer, with precise control of printing conditions. The research specifically explored the influence of varying heating rates which were 1.5°C/min, 2.0°C/min and 2.5°C/min and different peak temperatures of 400°C, 500°C, and 600°C, all conducted under a consistent nitrogen atmosphere and without an isothermal holding period. After debinding, samples underwent comprehensive analysis including visual inspection, Scanning Electron Microscopy (SEM), and Energy Dispersive X-ray Spectroscopy (EDX). These methods provided valuable information about surface conditions, internal microstructural features, and elemental distributions. Results revealed at heating rates of 1.5°C/min, combined with peak temperatures of 500°C provided optimal conditions for thermal debinding. SEM analysis demonstrated that samples processed under these conditions exhibited minimal structural defects, such as micro-cracks or bloating, preserving the integrity of the component structure. Furthermore, the SEM images confirmed a homogeneous and well-developed porosity network, essential for efficient binder removal during the subsequent sintering process. In contrast, samples at heating rates 2.5°C/min were found to have a higher incidence of structural irregularities, particularly internal voids and minor cracks. Additionally, EDX compositional analysis highlighted a notable increase in residual carbon content within these samples. Specifically, the EDX results revealed higher carbon concentrations, indicating incomplete removal of binder material and possible accumulation of carbonaceous residues at faster heating conditions which was 2.5°C/min. This could adversely affect the mechanical properties and overall quality of the final sintered components. Overall, the findings underscore the critical importance of carefully balancing thermal debinding parameters. Proper control of heating rates and peak temperatures is essential to optimize both the physical structure and chemical composition of the 17-4PH stainless steel components. This study provides valuable insights and practical guidance for emphasizing that meticulous process control.

PENYIASATAN PARAMETER PROSES DALAM PENYAHIKATAN TERMA BAGI SAMPEL KELULI TAHAN KARAT 17-4PH YANG DICETAK SECARA 3D

ABSTRAK

Proses penyahikatan terma merupakan peringkat penting dalam pembuatan aditif logam yang mempengaruhi secara signifikan kualiti dan sifat komponen keluli tahan karat 17-4PH yang dihasilkan melalui kaedah Pemodelan Pemendapan Berfused (FDM). Kajian ini menyiasat parameter optimum dalam proses penyahikatan terma bertujuan untuk menyingkirkan bahan pengikat dengan berkesan sambil mengekalkan integriti struktur komponen tercetak sebelum proses pensinteran. Sampel dihasilkan menggunakan filamen keluli tahan karat Ultrafuse® 17-4PH pada mesin pencetak 3D Ultimaker S5 dengan kawalan teliti terhadap keadaan pencetakan. Penyelidikan ini secara khusus mengkaji pengaruh pelbagai kadar pemanasan iaitu 1.5°C/min, 2.0°C/min dan 2.5°C/min serta suhu maksimum berbeza iaitu 400°C, 500°C dan 600°C, kesemuanya dilakukan dalam suasana nitrogen yang konsisten tanpa sebarang tempoh tahanan isoterma. Selepas proses penyahikatan selesai, sampel menjalani analisis menyeluruh termasuk pemeriksaan visual, Mikroskop Elektron Imbasan (SEM) dan Spektroskopi Sinar-X Pancaran Tenaga (EDX). Kaedah ini memberikan maklumat penting tentang keadaan permukaan, ciri mikrostruktur dalaman serta taburan unsur dalam sampel. Hasil kajian menunjukkan bahawa kadar pemanasan 1.5°C/min yang digabungkan dengan suhu puncak 500°C memberikan keadaan paling optimum untuk proses penyahikatan terma. Analisis SEM membuktikan bahawa sampel yang diproses pada keadaan ini mempunyai kecacatan struktur yang sangat minimum, seperti retakan mikro atau penggelembungan, sekali gus mengekalkan integriti struktur komponen. Selain itu, imej SEM mengesahkan kewujudan jaringan keliangan dalaman yang homogen dan teratur, penting untuk penyingkiran bahan pengikat secara efektif semasa proses pensinteran yang berikutnya. Sebaliknya, sampel yang diproses pada kadar pemanasan 2.5°C/min menunjukkan kadar kecacatan struktur lebih tinggi seperti rongga dalaman dan keretakan kecil. Tambahan pula, analisis EDX menunjukkan peningkatan ketara kandungan karbon sisa dalam sampel ini. Secara khususnya, keputusan EDX mendapati kepekatan karbon yang tinggi, menandakan penyingkiran bahan pengikat tidak lengkap serta kemungkinan berlakunya pengumpulan sisa karbon pada kadar pemanasan yang lebih pantas iaitu 2.5°C/min. Ini berpotensi menjejaskan sifat mekanikal dan kualiti keseluruhan komponen yang disinter kelak. Secara keseluruhan, dapatan kajian ini menegaskan betapa pentingnya mengimbangi parameter penyahikatan terma dengan teliti. Kawalan rapi terhadap kadar pemanasan serta suhu maksimum amat kritikal bagi mengoptimumkan struktur fizikal serta komposisi kimia komponen keluli tahan karat 17-4PH. Kajian ini memberikan panduan praktikal yang berharga serta menekankan pentingnya kawalan proses yang teliti untuk aplikasi industri.

ACKNOWLEDGEMENT

In the Name of Allah, the Most Gracious, the Most Merciful.First and foremost, I would like to take this opportunity to express my sincere acknowledgement to numerous individuals including my supervisor Associate Prof Ir. Ts. Dr. Shajahan Bin Maidin, my colleagues and my pals. Their ideas, views, and knowledge aided my research and comprehension in the Investigation of Process Parameters in Thermal Debinding of 3D-Printed 17-4PH Stainless Steel Samples. I would also like to express my appreciation to everyone named above who has contributed information, collaboration, guidance, and creative thought to the successful completion of the projects. Without their assistance and collaboration, this study would not have been conducted as it is described here. I am also indebted to University Teknikal Malaysia Melaka (UTeM), for providing the essential literatures and knowledge. My gratitude also extends to all of my colleagues and those who have assisted me on various occasions and boosted my spirits. Their perspective and advice were very helpful. I am grateful to all my family members especially to my wife Nur Atikah Binti Ab Rahman for her support and cooperation which lead me to work harder in order to complete this project.

TABLE OF CONTENTS

		PAGES
DECLARATION		
APPROVAL		
DEDICATION		
ABSTRACT		**i**
ABSTRAK		**ii**
ACKNOWLEDGEMENT		**iii**
TABLE OF CONTENTS		**iv**
LIST OF TABLES		**vi**
LIST OF FIGURES		**vii**
LIST OF ABBREVIATIONS		**ix**
LIST OF SYMBOLS		**x**
CHAPTER		
1.	**INTRODUCTION**	**11**
	1.1 Background	11
	1.2 Problem Statement	14
	1.3 Project Objectives	15
	1.4 Project Scope	16
	1.5 Master Project Outline	17
2.	**LITERATURE REVIEW**	**19**
	2.1 Introduction	19
	2.2 AM Techniques	20
	2.2.1 Binder Jetting (BJT)	20
	2.2.2 Directed Energy Deposition (DED)	22
	2.2.3 Material Extrusion (MEX)	23
	2.2.4 Material Jetting (MJT)	26
	2.2.5 Powder Bed Fusion (PBF)	27
	2.2.6 Sheet Lamination (SHL)	29
	2.2.7 Vat Photopolymerization (VPP)	30
	2.3 AM Process Flow	31
	2.4 AM Advantages	35
	2.5 AM Disdvantages	36
	2.6 Debinding	37
	2.7 Summary	42
3.	**METHODOLOGY**	**43**
	3.1 Introduction	43
	3.2 Flow Chart	43
	3.3 Experiment Set Up	44
	3.3.1 Material For Specimen	44
	3.3.2 Printing Process	46
	3.3.3 Thermal Debinding	47
	3.3.4 Design of Experiment (DoE) Using Design Expert	48

3.3.5 Microstructure Analysis 49
3.4 Summary 51

4. Result and discussion 53
4.1 Introduction 53
4.2 17-4 PH Stainless Steel 3D Printed Samples 54
4.3 Thermal Debinding on 17-4 PH Stainless Steel 3D Printed Samples 56
4.4 Compositional Analysis 59
4.4.1 Carbon Content Reduction and Binder Removal Efficiency 60
4.4.2 Composition Level After Debinding 61
4.5 Microstructural Analysis of Thermally Debinding 17-4PH Samples 69
4.5.1 Microstructural Analysis 70

5. conclusion and recommendation 77
5.1 Conclusion 77
5.2 Recommendation 80

LIST OF TABLES

TABLE	TITLE	PAGE
Table 2.1:	Sample of literature review for thermal debinding on stainless steel (SS) material	40
Table 2.2:	Summary of debinding process	42
Table 3.1:	Manufacturer Recommendation Set Up	45
Table 3.2:	Experiment parameter combination based on Full Factorial design	49
Table 4.1:	Thermal debinding parameter (S indicated as sample)	57
Table 4.2:	Composition after thermal debinding	60

LIST OF FIGURES

FIGURE	TITLE	PAGE
Figure 2.1: Seven Types of AM (Adugna et. al., 2021)		20
Figure 2.2: Illustration of BJT technique process flow (Mostafaei et al.2021)		21
Figure 2.3: a) Powder DED; b) Wire DED (Selema, et. al., 2022)		23
Figure 2.4: Layout of FDM process (Rajan et al.2022)		24
Figure 2.5: DIW schematic process (Mahshid et al. 2023)		25
Figure 2.6: Material jetting method (Macovei and Paleu, 2022)		26
Figure 2.7: SLS method (Zhang & Liou, 2021)		28
Figure 2.8: EBM technique (Singh et al. 2021)		29
Figure 2.9: SHL technique (Jadhav et al, 2022)		30
Figure 2.10: VPP process (Rajan et al.2021)		31
Figure 2.11: Construction of 3D CAD model (Lv et al., 2024)		32
Figure 2.12: Sample of G-Code in AM (Gao et. al., 2018)		33
Figure 2.13: AM machine configuration (Tajik et al., 2022)		33
Figure 2.14: General AM process (Zhang & Liou, 2021)		34
Figure 2.15: Part weight loss relation to time in solvent debinding (Singh et. al., 2021)		38
Figure 3.1: Research flow chart		44
Figure 3.2: Ultrafuse® 17-4 PH filament		45
Figure 3.3: Ultimake S5 printing machine		46
Figure 3.4: MV multi-position tube furnace		48
Figure 3.5: SEM-EDX machine		50
Figure 4.1: 17-4 PH stainless steel 3D printed samples		54

Figure 4.2: Scanning Electron Microscopy (SEM) at 1500X magnification 71

LIST OF ABBREVIATIONS

AM	-	Additive Manufacturing
ASTM	-	American Society for Testing and Materials
BJT	-	Binder Jetting
CAD	-	Computer-Aided Design
DOE	-	Design of Experiment
DED	-	Directed Energy Deposition
DLP	-	Digital Light Processing
EDX	-	Energy Dispersive X-ray Spectroscopy
FDM	-	Fused Deposition Modeling
ISO	-	International Organization for Standardization
MEX	-	Material Extrusion
MJT	-	Material Jetting
PBF	-	Powder Bed Fusion
POM	-	Polyformaldehyde
PPE	-	Personal Protective Equipment
SDS	-	Safety Data Sheet
SEM	-	Scanning Electron Microscopy
SHL	-	Sheet Lamination
SLA	-	Digital Light Processing
SLS	-	Fused Deposition Modeling
VPP	-	Vat Photopolymerization

LIST OF SYMBOLS

°C	-	Degree celcius
%	-	Percentage
C	-	Carbon
Cr	-	Chromium
Cu	-	Copper
Fe	-	Iron
Mn	-	Manganese
Nb	-	Niobium
Ni	-	Nickel
Si	-	Silicon

CHAPTER 1

INTRODUCTION

1.1 Background

Few technologies have drawn as much attention and had as much potential as 3D printing in the context of modern production and innovation. The process of additive manufacturing (AM) involves creating objects layer by layer, using computer-aided design software to guide the printing of each layer. This meticulous approach allows for the production of highly customized and detailed parts, making it a valuable tool in various industries such as aerospace, automotive, healthcare, and consumer goods. Stereolithography (SLA), a technique invented by physicist and inventor Chuck Hull in the 1980s, gave rise to the idea of 3D printing. In order to make three-dimensional sculptures, this approach involves solidifying thin layers of photopolymer resin with a UV laser. His invention laid the foundation for the AM revolution that followed. Other AM techniques followed, including Digital Light Processing (DLP), Fused Deposition Modeling (FDM), and Selective Laser Sintering (SLS). The distinct benefits and uses of each technique added to the adaptability and affordability of 3D printing technology.

The evolution of AM has also seen improvements in the speed and efficiency of the printing process, as well as the development of post-processing techniques to enhance the strength and finish of printed parts. It has experienced significant evolution since its inception, with advancements in materials, processes, and technologies. AM has opened up new possibilities for creating intricate and complex designs that were previously impossible to achieve using traditional manufacturing methods. The ongoing research and development efforts are focused on enhancing the speed and scale of the printing process, making it more efficient and cost-effective. Additionally, there is a growing emphasis on expanding the range of materials that can be used in 3D printing including metals, polymers, ceramics, and composites.

Moreover, the evolution of AM is not only expanding its capabilities but also improving the overall quality of printed parts. Advancements in post-processing are enabling the production of parts with superior mechanical properties, making them suitable for end-use applications, further solidifying the position of AM in the production of high-quality and functional components.

According to Nyamekye et al., (2023), AM offers design optimization opportunities that can further enhance environmental sustainability. AM has emerged as a promising approach for achieving environmental sustainability in manufacturing processes. Its ability to reduce waste, lower transportation needs, and produce complex, lightweight designs makes it an attractive option for businesses seeking to improve their environmental footprint while maintaining high-quality production standards. Traditional manufacturing often involves large-scale production runs which can lead to overproduction and excess inventory. This can result in wasted materials, energy, and resources. AM allows for small-scale, on-demand production, reducing the risk of overproduction and minimizing excess inventory. This not only reduces waste but also minimizes the environmental impact associated with large-scale manufacturing and inventory storage.. These factors position AM as a key technology for advancing environmental sustainability in the manufacturing industry.

As AM continues to evolve, its impact is not limited to specific industries but extends across diverse sectors, fueling innovation and opening new possibilities for design and production. The convergence of additive manufacturing with other advanced technologies, such as artificial intelligence and robotics, will further amplify its potential and revolutionize manufacturing processes.

While challenges such as standardization, certification, and scalability persist, ongoing research and development efforts are focused on addressing these hurdles, ensuring the reliability and widespread adoption of AM in industrial applications. the continued evolution of AM is poised to shape the manufacturing landscape, offering greater design freedom, faster production times, reduced material waste, and increased sustainability, making it an indispensable part of the manufacturing industry. As the technology progresses, we can anticipate further breakthroughs and innovations that will continue to redefine the way we conceptualize and produce physical objects in the future.

The mechanical properties of 3D printed parts are critical to their performance and reliability in real-world applications. Factors such as tensile strength, elasticity, toughness, and hardness play key roles in determining the functionality of 3D printed parts under various conditions and stresses. Understanding these properties at a deeper level requires a thorough analysis of the complex interplay of printing parameters, material composition, and post-processing techniques. By optimizing these properties can significantly improve the performance and durability of 3D printed parts. By gaining a comprehensive understanding of these properties, it becomes possible to enhance existing materials and develop entirely new materials and innovative printing techniques. Such advancements can expand the capabilities of 3D printed parts, opening up new applications and industries.

Improper debinding can lead to defects such as cracking, bloating, deformation, or residual contamination, which may compromise the mechanical integrity and final properties of the sintered component. The success of the thermal debinding process depends on carefully controlled parameters, including heating rate, peak temperature, holding time, and atmosphere. Therefore, a deep understanding of these process parameters is essential to optimize the debinding stage and ensure consistent quality in the final parts.

Despite the growing industrial interest in metal-polymer filaments, comprehensive studies on the thermal debinding behavior of 17-4PH 3D printed parts remain limited, especially under varying heating conditions. This research aims to investigate the influence of key thermal debinding parameters on the structural and chemical integrity of 3D printed 17-4PH components. The findings are expected to contribute to the development of more robust processing guidelines for manufacturers seeking to adopt metal FDM technologies in high-performance applications.

1.2 Problem Statement

The adoption of metal additive manufacturing using Fused Filament Fabrication (FDM) has introduced new possibilities for producing complex and cost-effective stainless steel components, particularly with materials like 17-4PH. However, the post-processing stage, specifically the thermal debinding process remains a critical bottleneck in achieving high-quality sintered parts. During thermal debinding, the polymer binder must be completely removed without compromising the structural integrity of the green part. Incomplete binder removal, rapid heating, or inappropriate thermal profiles can lead to serious defects such as internal voids, bloating, cracking, or contamination, which ultimately reduce the density, mechanical strength, and overall reliability of the final product.

Despite the increasing usage of 17-4PH in industry, there is a lack of detailed studies that focus specifically on how different thermal debinding parameters such as heat rate, peak temperature, and atmosphere affect the binder removal efficiency and part quality. Most available process settings are generalized or based on empirical knowledge, which may not be suitable for all furnace systems or geometries. Without a clear understanding and control of these parameters, manufacturers face challenges in standardizing the debinding process, leading to variability in product performance and increased rejection rates.

Therefore, there is a pressing need to systematically investigate the influence of thermal debinding parameters on 3D printed 17-4PH stainless steel samples. By identifying optimal conditions that ensure complete and defect-free binder removal, this research aims to fill a crucial knowledge gap and support the development of reliable, repeatable, and scalable post-processing practices for 3D printed 17-4PH stainless steel.

1.3 Project Objectives

Objectives of the project are:

1. To fabricate 17-4PH stainless steel printed samples.
2. To investigate the optimum process parameters for thermal debinding for 17-4PH stainless steel printed samples.
3. To study the changes in composition and microstructure resulting from variations in thermal debinding parameters.

1.4 Project Scope

This project is focused on the additive manufacturing and post-processing of 17-4PH stainless steel components through the use of Fused Deposition Modeling (FDM), a promising metal 3D printing technology. The scope of this work has been carefully defined to cover each critical step involved in producing high-quality metal parts, beginning from the fabrication stage and extending through to comprehensive analysis and characterization. The project involves fabricating standardized test samples from Ultrafuse® 17-4PH stainless steel filament supplied by BASF. To ensure the accuracy, consistency, and repeatability of the printed samples, an Ultimaker S5 3D printer will be employed. The printing process will adhere strictly to detailed parameters recommended by the filament manufacturer, specifically maintaining a nozzle temperature between 230°C and 250°C, and a heated bed temperature of 70°C to minimize defects such as warping or dimensional inaccuracies. This ensures that all fabricated parts possess uniform geometry and suitable mechanical properties required for subsequent analysis stages. Upon completion of the printing phase, the project will systematically investigate and determine the optimal conditions for the thermal debinding process. Thermal debinding is a critical post-processing step necessary to remove polymeric binder materials from the metal-polymer composite filaments prior to sintering. This phase of the study will be performed using an MV multi-position tube furnace, with experiments methodically planned to explore various controlled heating rates of 1.5°C/min, 2.0°C/min and 2.5°C/min, alongside different peak debinding temperatures of 400°C, 500°C and 600°C. Each experimental condition will be carried out under a consistently regulated nitrogen atmosphere, specifically chosen to protect the samples from oxidation or contamination, without employing an isothermal holding period. The purpose of varying these parameters systematically is to identify the most effective thermal profiles that can thoroughly remove binder material while preserving the structural integrity of the printed components, thereby minimizing defects such as internal voids, cracking, or bloating. Finally, the scope encompasses extensive post-process evaluations that will thoroughly characterize the impact of different debinding parameters on the quality and performance of the processed components. Each sample will undergo detailed visual inspections to assess surface quality and visible defects. More comprehensive evaluations will involve the use of advanced microscopy techniques such as Scanning Electron Microscopy (SEM), coupled with Energy Dispersive X-ray Spectroscopy (EDX), which provide critical insights into the internal microstructural characteristics and elemental composition of the materials. These analyses will specifically highlight the effectiveness of binder removal, the distribution of residual elements, the presence of any microstructural irregularities, and the overall compositional integrity of the samples.in the analytical models.

1.5 Master Project Outline

Based on the objectives previously presented and on the approach proposed before, this master project is made up of five (5) chapters, which contents are summarized as follows:

- Chapter 1. Introduction. This chapter presents the background of the study, research problems, objectives, scopes, contributions and significance of the research.
- Chapter 2. Literature review. This chapter brief overview of AM printing and thermal debinding process based on related studies.

- Chapter 3. This chapter presents the methodology that has been developed to achieve the objectives in Chapter 1 including design of experiment and method for analysis.
- Chapter 4. This chapter presents the key findings from the experiments and discusses it significance in relation to the research objectives. It outlines the results in a clear and structured manner, followed by a thoughtful interpretation of the results. The discussion explores how the observed outcomes relate to the initial problem statement, highlights any trends or unexpected behaviors and connects the findings to previous studies.
- Chapter 5. This chapter sums up the main findings and how they relate to the research objectives. It highlights key takeaways, addresses any limitations, and offers practical recommendations for future work or improvements in the process.