

Effect of annealing time on residual stress in selective laser melted Co-Cr-Mo components

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Dissertation accepted in fulfilment of the requirements for the degree *Master of Engineering in Mechanical Engineering* at the North-West University

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Graduation: November 2023

ACKNOWLEDGEMENTS

I would firstly like to thank the Collaborative Programme for Additive Manufacturing for funding my studies by providing me with a bursary for two years. I would also like to thank the following people, without whom I would not have been able to complete this research:

The North West University, especially my supervisor Mr. CP Kloppers, whose insight and knowledge into the subject matter steered me through this research. I would also like to give special thanks to Dr. Jaundrie Fourie, whose support as co-supervisor allowed my studies to go the extra mile

The team at NECSA, especially Dr. Deon Marais for performing the Neutron Diffraction Analysis and providing detailed feedback and support.

My colleagues at the Additive Manufacturing Facility, who supported and encouraged me through the past three years of study.

And my biggest thanks to my parents and my husband for all the support through this research, without which I would not have been able to successfully complete my studies. You have been amazing.

ABSTRACT

Keywords: *Additive Manufacturing, Removable Partial Dentures, Heat-Treatment, Selective Laser Melting, Co-Cr-Mo, Simulation*

Additive manufacturing (AM) is a three-dimensional (3D) manufacturing technique that allows the user to manufacture designs with complex geometries. Selective Laser Melting (SLM) is an AM technique where a layer-wise process of melting metal powder is used to manufacture 3D objects. A major disadvantage of SLM is the deformation of parts during the print cycle or after the part is removed from the build plate. The thermal inconsistencies inherent in this layer-wise manufacturing process cause residual stress build-up that may exceed the yield stress of the material and lead to print failures.

The residual stress in AM parts manufactured with metal powder is similar to the residual stress that builds up in cold-worked metal parts due to the applied stresses stored within the material. The residual stress is stored in the form of dislocations and stress-relief annealing is commonly used to minimize this residual stress. Several studies have investigated the effect of stress-relief annealing on the microstructure, mechanical properties and residual stress in AM components. The research indicates that stress-relief annealing can change the microstructure, improve mechanical properties and reduce residual stress.

Limited knowledge is available on the effect stress-relief annealing hold time at maximum temperature has on the residual stress in AM parts. This study aimed to investigate the effect of stress-relief anneal time on the residual stress in Co-Cr-Mo components manufactured with SLM with the use of neutron diffraction techniques and Simufact Additive software. Neutron diffraction is a non-destructive method for determining residual stress in a material. Simufact Additive software is simulation-based software that uses thermo-mechanical simulations to analyse the AM process. Six Co-Cr-Mo samples were manufactured on the ORLAS Creator with SLM and five of the samples were stress-relief annealed using varying time profiles. The annealing times and process parameters were chosen from the literature to provide comparable results. The samples were annealed at 1065°C with annealing times of 15 minutes, 1 hour, 2 hours, and 3 hours. One sample was annealed at 750°C for 1 hour.

The results obtained through simulation and neutron diffraction analysis showed good correlation. The samples heat treated to 1065°C showed more residual relief than 750°C indicating that the heat treatment temperature had a significant effect on the degree of residual stress relief. The results related to samples heat treated to 1065°C indicated that shorter annealing times resulted in near- identical residual stress values than longer annealing times. This discovery indicated that

residual stress relief occurred in a shorter time frame than anticipated and that heat treatment duration longer than 15 minutes did not result in improved residual stress relief results.

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ACRONYMS AND ABBREVIATIONS

0.2% YS	Offset Yield Strength
3D	Three-Dimensional
AM	Additive Manufacturing
ASTM	American Society for Testing and Materials
BCC	Body-Centred Cubic
CAD	Computer Aided Design
CLSM	Confocal Laser Scanning Microscopy
DED-L	Laser Beam Directed Energy Deposition Systems
EDM	Wire Electro Discharge Machining
EEF	Exposure Energy Fraction
FCC	Face-Centred Cubic
FDM	Fused Deposition Modelling
FEM	Finite Element Modelling
HCP	Hexagonal Close-Packed
MPISI	Materials Probe for Internal Strain Investigation
Necsa	The South African Nuclear Energy Corporation State-Owned Companies Limited
OM	Optical Metallography
OR LASER	OR Lasertechnologie GmbH
PBF	Powder Bed Fusion
PBF-L	Laser Beam Powder Bed Fusion Systems
RP	Rapid Prototyping
RPD	Removable Partial Denture
SEM	Scanning Electron Microscopy
SLM	Selective Laser Melting
SLS	Selective Laser Sintering
STL	Stereolithography File-Type
UTS	Ultimate Tensile Strength
VEF	Volumetric Expansion Factor
XRD	X-Ray Diffraction

CHAPTER 1 INTRODUCTION

1.1 Background

The global dental market is expected to grow at a compound annual growth rate of 7.6% from 2018 to 2023 [1]. Due to the global ageing of the population there is an increased demand for cosmetic dentistry and for general dental procedures [1, 2]. The ageing population requires dental prosthodontics such as implants and dentures to replace missing teeth. Most designed dentures are removable partial dentures (RPD) although there are various designs on the market that require dentures to be fixed to existing teeth or to implants [2].

Co-Cr-Mo alloys are highly valued in the medical and dental industries for their biocompatibility, superior mechanical properties, outstanding abrasive wear resistance, and exceptional corrosion resistance [3–5]. The traditional manufacturing method for metal dental components is creating a digital or physical impression of the patient's mouth; designing a metal alloy frame to fit into the patient's mouth; making a mould of the framework, casting the metal alloy framework; and finally veneering with acrylics or ceramics [6–8]. The process requires various intermitted testing procedures to ensure perfect fit and comfortability for the patient. Any changes that must be made require a repeat of the entire process, which is time consuming and requires additional consumable materials. The dental industry is continuously seeking improved methods to efficiently create and produce dental components [4, 6, 7].

AM is a 3D manufacturing technique that allows the user to create designs with complex geometries and to manufacture these complex parts faster than traditional manufacturing methods [7, 9, 10]. This manufacturing method has gained interest in the dental field due to the complex geometry and patient specific nature of dental components. The AM field has improved tremendously since its conception in the 1970s and various materials have been added to the AM database including dental materials such as dental polymers and dental metal alloys [6, 8, 10].

Metal AM techniques are classified into four groups: laser beam powder bed fusion systems (PBF-L), laser beam directed energy deposition systems (DED-L), electron beam powder bed fusion systems and 3D metal printing with arc welding systems [9]. SLM is a metal AM technique categorised as PBF-L and the process starts with a chosen fine metal powder with specific particle sizes. The powder is contained within the build plate area where a laser melts the first layer of powder to the solid build plate. The build plate is then lowered after each layer is melted for the roller and scraper to apply the correct layer thickness of the material to the print area. After the process is completed, the excess powder is carefully removed from the part. SLM has improved

significantly since inception and offers the possibility to produce parts with near 100% density [6, 8].

One of the disadvantages of SLM is the deformation of printed parts after removal from the build plate [11]. During printing operations, the first layers start to cool off while the consecutive layers are being printed. The thermal inconsistencies create a residual tensile or compressive stress in the part. As soon as the part is removed from the build plate the residual stress could exceed the yield stress of the component and severe deformation, warping, cracks and delamination may occur. These failures can also occur during the print cycle, obstructing the recoater blade and resulting in build failure, which result in wastage of material and manufacturing time [11–13]. This is especially critical to dental components such as RPDs as these components have complex thin structures, which are specifically designed to fit perfectly within the patient's mouth. Thin-walled components are prone to deformation and therefore limiting deformation in AM dental components is essential [4].

Researchers are continuously working on possible solutions to minimize build failures by improving support structure design, scanning patterns and post-process heat treatments [12–15]. The residual stress seen in metal AM parts is similar to the residual stress that builds up in cold-worked metal parts due to the applied stresses stored within the material. The residual stress is stored in the form of dislocations and a heat treatment procedure, known as stress-relief annealing is commonly used to minimize residual stress [16, 17].

Several studies have investigated the effect of stress-relief annealing on the microstructure and mechanical properties of metal AM parts [11, 14, 18–23]. Research shows that stress-relief annealing leads to significant improvement in the microstructure and mechanical properties, resulting in increased ductility and enhanced wear resistance, albeit with a slight sacrifice on tensile and yield strength. Studies measuring residual stress before and after stress-relief annealing found that the heat treatment significantly reduced the residual stress. The findings of these studies suggest that stress-relief annealing should significantly reduce the deformation of printed parts.

1.2 Problem statement

With the innovation and research in the field of medical technologies and dentistry the development of patient specific medical devices has become more popular practice. Removable partial dentures have been manufactured by means of centrifugal casting for a number of years but with the advent of metal additive manufacturing this technology is replacing the current production methods.

Additive manufacturing can produce high quality fully dense parts, however the factors influencing the dimensional accuracy of these parts are still in development. Removable partial dentures produced by additive manufacturing have significant residual stresses inside the components due to the thermal history of the manufacturing process. An effective stress relief anneal heat treatment can drastically reduce the residual stress and thus also the distortion of the part. Thus, the problem is to develop a effective stress relief anneal cycle to reduce the residual stress in removable partial dentures produced by additive manufacturing.

1.3 Aims and objectives

This study aims to investigate the effect of stress-relief holding time on the residual stress in Co-Cr-Mo alloy components manufactured with SLM. The aim will be achieved by completing the following objectives:

1. Manufacture Co-Cr-Mo samples with the use of SLM on ORLAS Creator and stress-relief anneal the samples with variation in annealing hold time.
2. Obtain residual stress state prediction by simulating the manufacturing process using Simufact Additive.
3. Determine the inherent strain in samples using Neutron Diffraction.
4. Calculate the residual stress by using the measured inherent strain.
5. Compare predicted residual stress with neutron diffraction data for verification purposes.

1.4 Experimental approach

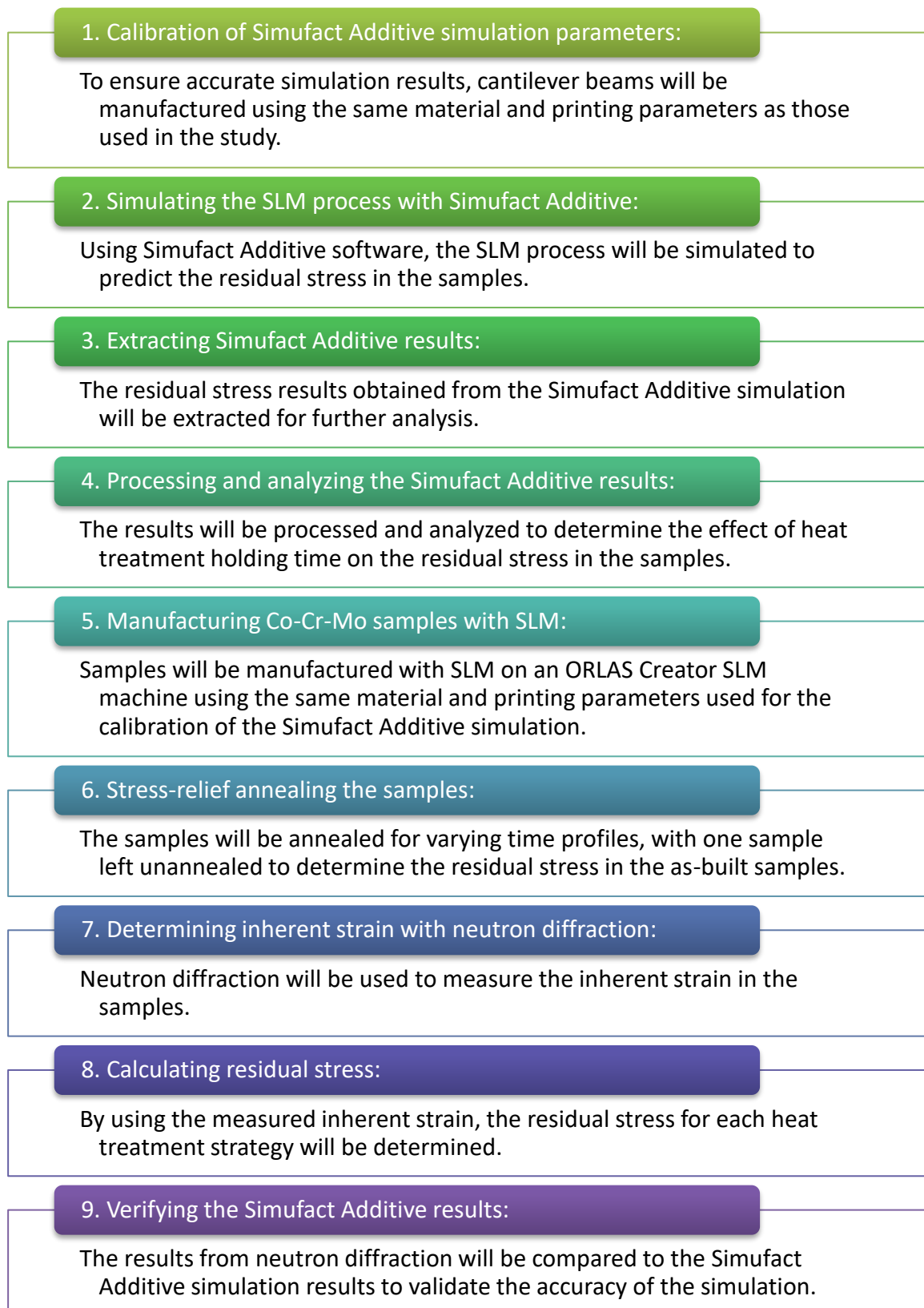


Figure 1.4-1: Experimental approach

CHAPTER 2 LITERATURE REVIEW

2.1 Introduction

Rapid prototyping (RP) is a term used in a wide range of industries to describe the process for the rapid creation of a system or part representation before the final product is produced. The output is a prototype of the final product that acts as the model from which improvements and further models will be derived. In recent years improvements in RP machinery have resulted in higher quality prototypes that closely resemble the final product. Some of the processes have been improved upon enough to enable the direct manufacturing of final products [9, 10].

This chapter focuses on the crucial aspect of residual stress relief annealing in AM. Residual stress is an inherent challenge in the AM process and can affect the mechanical properties and integrity of the manufactured components. The aim of this chapter is to provide a comprehensive overview of the various techniques and strategies for residual stress relief annealing in AM. The chapter begins with a brief history of AM and a discussion of the general principle behind it. It then delves into the different types of AM techniques, with a specific emphasis on metal SLM, which is one of the most commonly used powder-based systems.

The chapter then highlights the limitations of AM and discusses residual stress in manufactured components. The various methods for measuring residual stress, such as contour method, X-ray diffraction, and neutron diffraction, are also discussed. The chapter concludes with a detailed examination of the methods for simulating residual stress and the crucial role that residual stress relief annealing plays in minimizing residual stress in AM.

2.2 Brief history of additive manufacturing

AM is the layer-by-layer addition of material to form a 3D object. A layering method for creating 3D topographical relief maps was suggested as early as 1890, shown in Figure 2.2-1 [9, 24]. The method required stacking layers of wax, cut according to the contour lines on the relief map. These layers were then smoothed and formed both the positive and negative for the 3D relief [24].

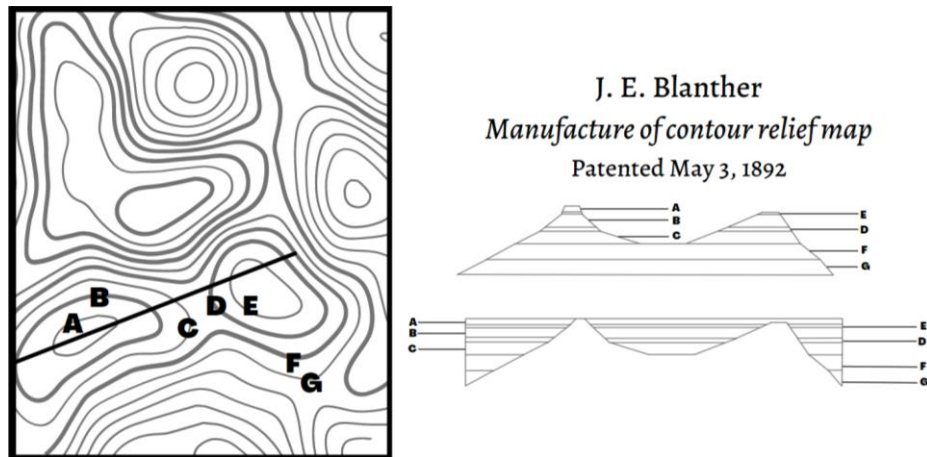


Figure 2.2-1: 3D relief map design. Adapted from Blanthier [24].

The first system that used stereolithographic techniques was proposed in 1951 and the technique involved exposing a transparent photo emulsion in a selective and layer-wise manner in order to fix the emulsion in a specific solid form [25]. In 1979 the first description of the selective laser sintering (SLS) method was patented by Dr. Carl Deckard and Dr. Joe Beaman. This technique required depositing planar layers of material and subsequently solidifying a selected portion of each layer by using heat. In 1992, this SLS system was made available to the public. Since the 1900s a plethora of AM techniques have been patented and many strides have been made in the field [21, 26–28].

2.3 The general principle of additive manufacturing

The general procedure for an AM process can be divided into eight steps. The process starts by generating a 3D computer aided design (CAD) model and exporting the design as a stereolithography file-type (STL). The file is then transferred to the AM machine and the machine setup is completed by levelling and heating the print surface, refilling the print medium and setting print parameters. The build stage is initiated after the desired setup has been completed. After the build stage is complete the printed part is removed from the platform and post-processing is performed [6, 8, 28].

All AM parts depend on a software model, created with the use of CAD software, that completely defines the geometry. Laser scanning software is also available to scan existing objects into a 3D CAD model. After the CAD model is fully defined the software can export the model as an STL file and transfer it to the machine. Each machine has its own software, which is used to position the CAD model, choose print parameters and view scanning strategies. All AM processes have unique requirements regarding the CAD model orientation, support structures and print

parameters. Once the machine has built the AM component it can be removed and sent for post-processing, which is a very important consideration for AM parts. Each AM process has specific post-process requirements such as support structure removal, surface finishing, or heat treatment [9, 27].

2.4 Types of additive manufacturing techniques

Since the advent of AM, various methods and processes have been designed to create 3D objects [25, 27]. AM systems consist of three main groups: liquid based systems, powder based systems, solid/extrusion based systems [26]. Classification of AM systems is summarized in Figure 2.4-1.

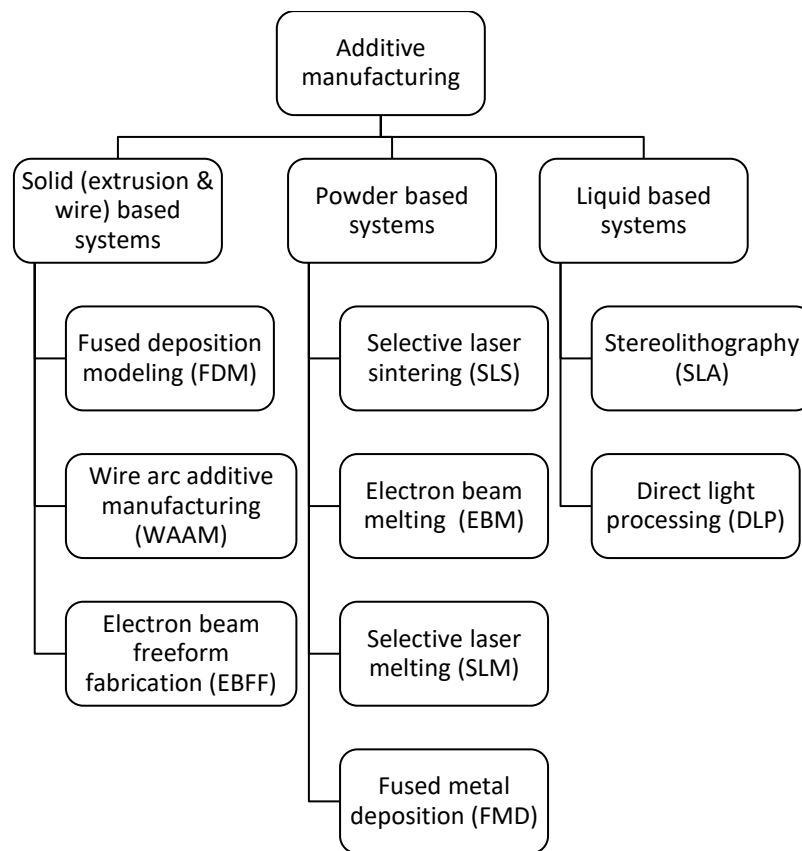


Figure 2.4-1: Diagram indicating summarized classifications of AM systems. Adapted from Joshi and Sheikh [29].

2.4.1 Liquid based systems

Liquid based systems use liquid photopolymers activated with ultraviolet radiation and then solidified by chemical reaction. Photopolymers were developed in the 1960s and the first AM commercial system, 3D Systems Stereolithography, made use of photopolymers to create 3D models. Three primary configurations are used for the photopolymerization process namely

vector scanning, mask projection and two-photon approaches. These configurations are illustrated in Figure 2.4-2 a, b and c respectively.

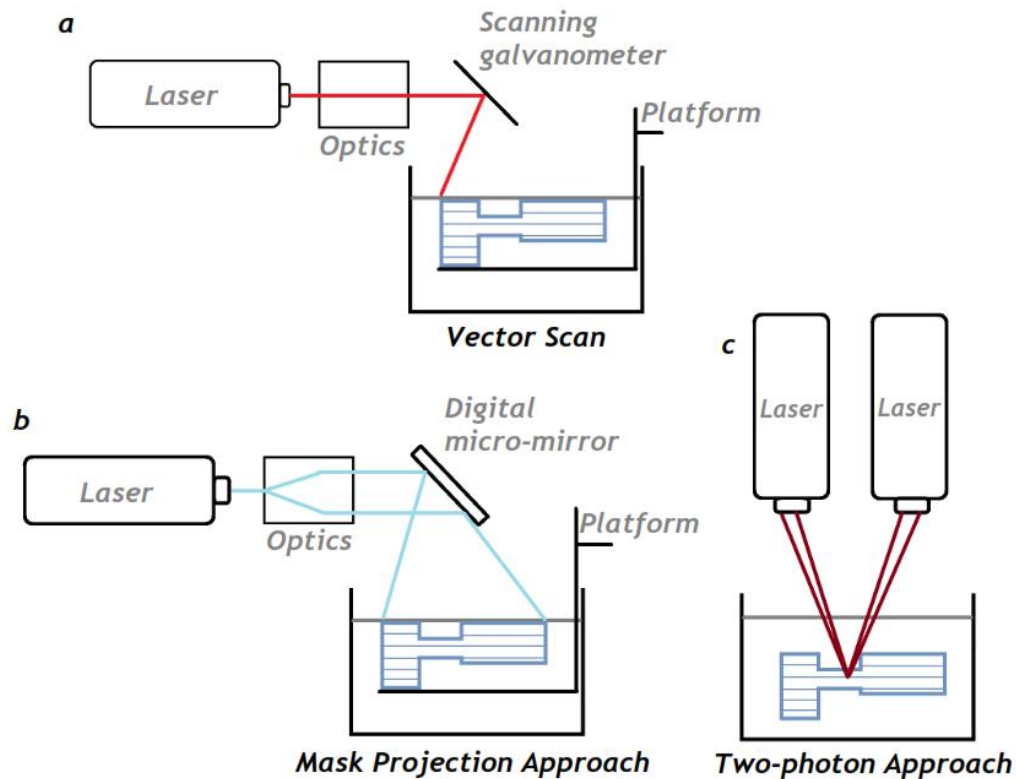


Figure 2.4-2: Three photo-polymerization configurations. Adapted from Gibson et al. [28].

2.4.2 Solid based systems

Molten material extrusion refers to systems where material is pre-heated to melting point and applied to a print platform through a delivery system. The most commonly used method is the fused deposition modelling (FDM) system, shown in Figure 2.4-3. FDM uses an extruder nozzle to deliver the material in a precise and controlled manner. This approach allows for two extrusion heads to be used allowing for the manufacturing of a component from two different materials simultaneously. The support structure can therefore be fabricated from easily dissolvable materials to simplify post-processing support removal [28].

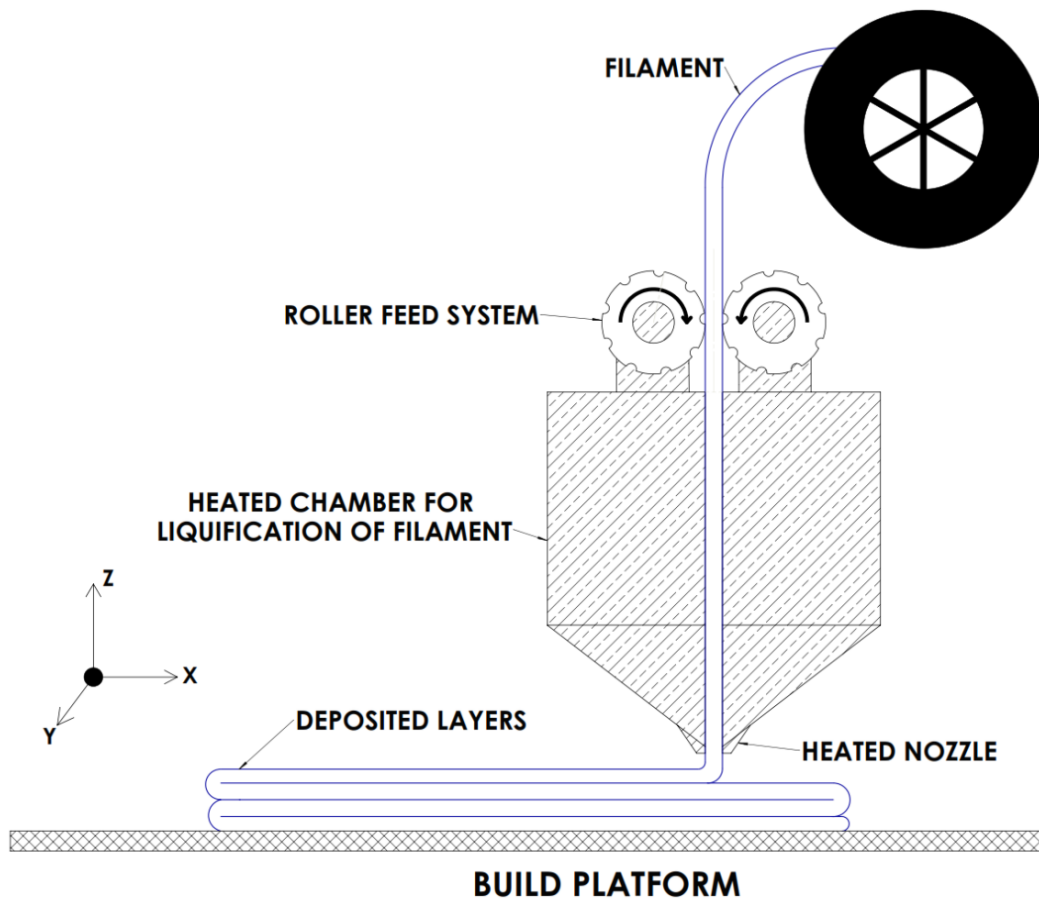


Figure 2.4-3: Solid extrusion-based system. Adapted from Gibson et al. [28].

2.4.3 Powder based systems

Discrete particle systems use powder material and thermal energy in a controlled manner to melt and re-melt the powder and bind consecutive layers to one another [27, 28]. Powder bed fusion (PBF) systems use a wide range of materials including polymers, metals, ceramics and composites. The PBF systems are extensively utilized in the production of end-use products globally due to their ability to create materials that possess properties that are akin to those of engineering-grade polymers, metals, and ceramics. This results in materials that are suitable for various high-performance applications [9, 27, 28]. The general setup for all PBF machines is presented in Figure 2.4-4.

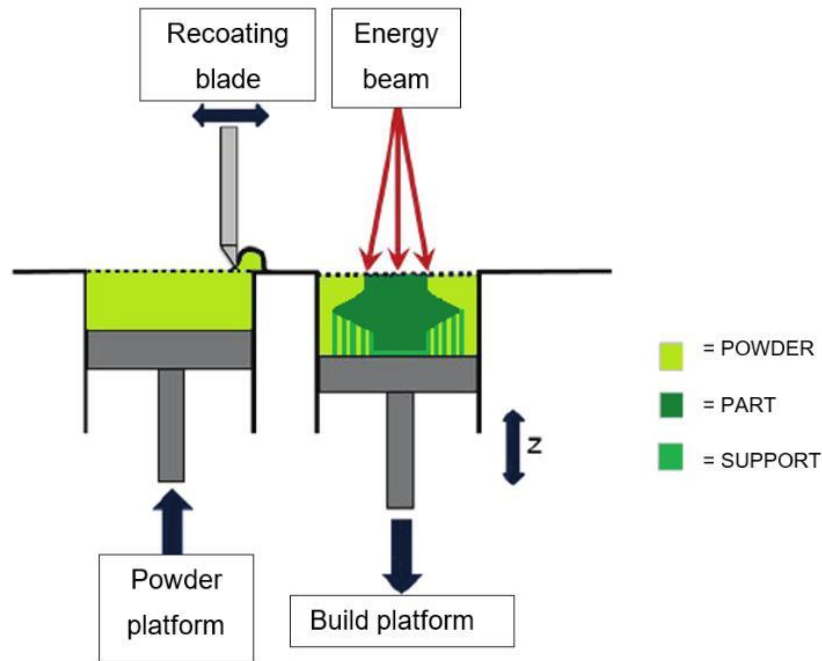


Figure 2.4-4: Powder-based system. Adapted from Moylan et al. [30].

2.4.3.1 Metal selective laser melting

SLM uses a laser beam to fuse thin layers of powder evenly spread across the build platform with a levelling roller. The build environment is enclosed and the chamber is filled with an inert gas to minimize oxidation of the powdered metal. The laser is carefully guided across the build platform to form the slice cross-section [27, 28, 31]. The build platform is lowered by one layer-thickness after each layer is successfully fused. The roller recoats and levels the powder after the build plate is lowered to ensure even layer thickness throughout the build process. A suitable cool-down period is initialized to ensure the part and the powder is not pre-maturely exposed to oxygen [27, 30].

The first powder laser sintering process was patented in 1979 [9, 25, 26]. The first commercial SLM machine was however only patented in 1992 by Carl R. Deckard [9, 26, 32]. In the early stage of SLM technology, only polymer powders could be processed until the first Metal SLM process was patented in 2004 by Suman Das and Joseph J. Beaman [33]. Metal powders are significantly harder to process since they tend to oxidize and exhibit low absorptivity. High energy lasers are required to sufficiently melt the metal powder, creating a molten pool that fuses multiple layers together. Insufficient energy transfer will result in porosity and reduce the strength of the final component. Further, oxidized metal powder will decrease the bonding strength between layers and is mitigated by purging the build chamber with an inert gas, such as Argon or Nitrogen [27].

2.5 Limitations of additive manufacturing

Despite the progress made in AM technology, many challenges persist such as the deformation of SLM manufactured components [9, 11]. During printing operations, the first layers start to cool down while the consecutive layers are being printed. The thermal inconsistencies create residual stresses in the component [34]. When the part is removed from the build plate the residual stress may exceed the tensile strength of the material which leads to deformation, cracking and delamination [4, 12, 13, 15].

2.6 Residual stress in manufactured components

Residual stress is stress induced in a material during processing. A small portion of the applied stress is stored in the form of residual stress within the structure as a network of dislocations, which subsequently increases the internal energy in the structure. The stress in metals can originate due to cold working or differential thermal expansion and contraction. The additional internal energy permits dislocations to move along the grain boundaries and create weak areas within the material resulting in reduced tensile strength in certain areas. [16]. As soon as the part is removed from the build plate severe deformation and cracks may occur due to the residual stress exceeding the tensile strength. The residual stress could also lead to warping and delamination, as presented in Figure 2.6-1 [13]. The build failure shown Figure 2.6-1 is common in practice and results in additional manufacturing time and material wastage [12].

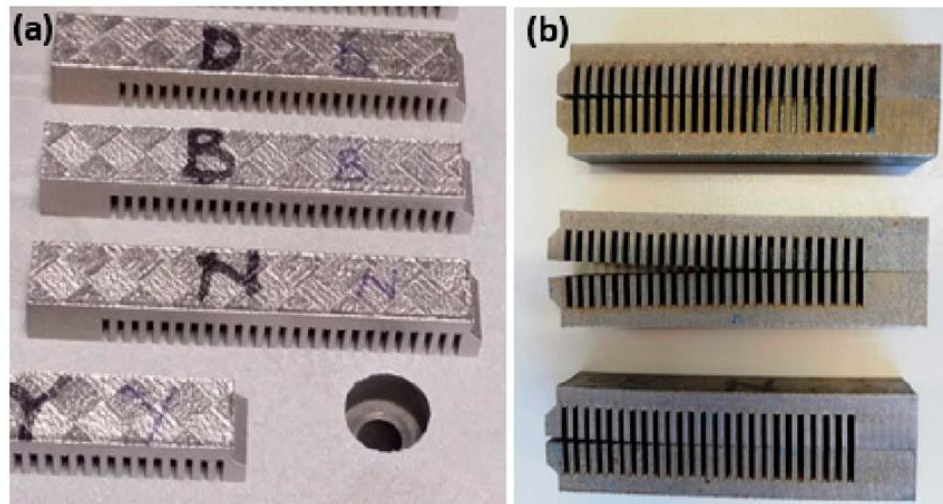


Figure 2.6-1: SLM manufactured part deformation. (a) Parts before removal from the base plate, (b) parts relative to each other after removal from the base plate. Reproduced with permission from Mugwagwa et al. [13].

2.7 Measuring residual stress

Several techniques can be used to measure the residual stress in samples and can be categorised as destructive or non-destructive. Destructive techniques include hole drilling and the contour method. Non-destructive techniques include ultrasonic, magnetic, X-ray diffraction and neutron diffraction [35]. Some of these techniques were reviewed to determine the technique that gives the most accurate residual stress measurements and to determine suitability for this study.

2.7.1 Contour method

The contour method is a destructive method used to determine the residual stress in samples. The sample is first cut with wire electro-discharge machining (EDM), after which the deformation profile of the newly cut faces is measured. The average deformation from both faces is determined and the experimental noise is filtered out to form a smoothed profile. Elastic strain analysis is then performed with finite element modelling (FEM). The calculated stress on the cut surface is representative of the true residual stress acting normal to the cut plane [36–38]. The principle of residual stress measurement with the contour method is illustrated in Figure 2.7-1.

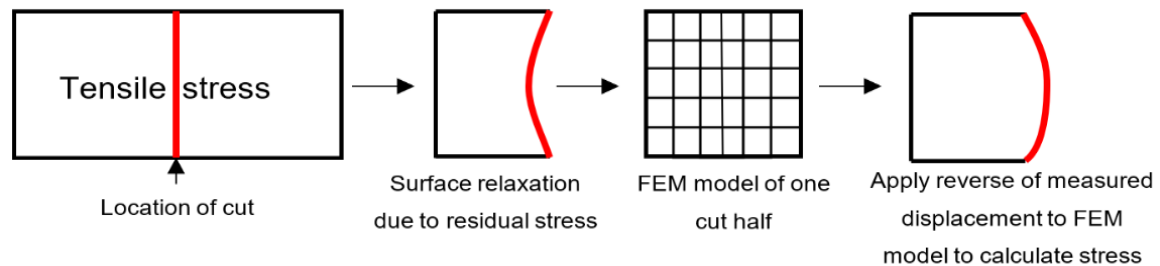


Figure 2.7-1: Principle of residual stress measurement with contour method. Adapted from Ahmad et al. [38].

There are several challenges to the cutting procedure to ensure no additional stress is added to the cutting surface and the surface finish is suitable for measuring deflection. EDM is the only method that is currently utilized for the cutting procedure as it contributes the least additional stresses to the cut surface, which can be corrected for during data processing [38–40]. This method was not considered for this study as the results are highly dependent on the cutting and surface measuring procedure. More accurate and reliable methods were available.

2.7.2 X-ray diffraction

X-ray diffraction (XRD) is a non-destructive method used to measure the residual stress in samples by using the distance between crystallographic planes as a strain gauge and calculating the residual stress from the measured strain. Several studies have determined that the accuracy of determination of the distance between crystallographic planes is dependent on the accuracy of the peak position measurement method [41–43]. Bragg's law is, as illustrated in Figure 2.7-2, is used to determine the distance between crystallographic planes.

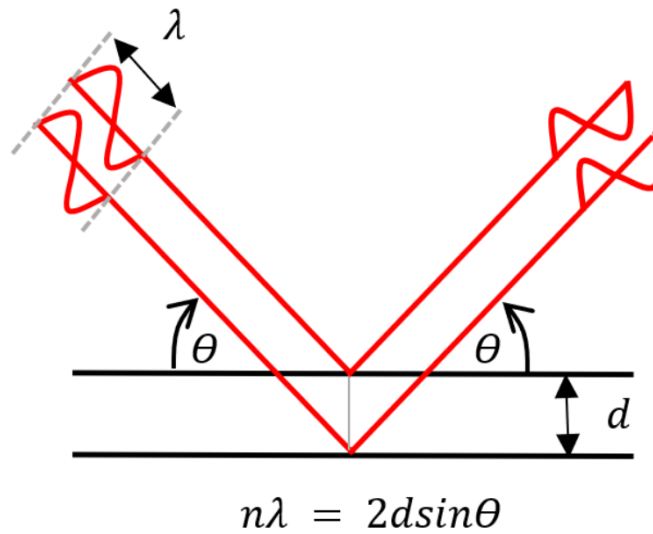


Figure 2.7-2: Illustration of Bragg's law. Adapted from Sedighi and Nazemnezhad [43].

The accuracy of the residual stress measured with the XRD method is highly dependent on the accuracy of the peak position. There are various methods for determining the peak position, each with its own limitations and level of difficulty [41–44]. Sedighi and Nazemnezhad [43] studied the effect of the peak positioning method on the accuracy of X-ray diffraction residual stress measurements. In this study five peak positioning methods were reviewed: maximum intensity method, middle position of the width at half maximum intensity method, Gaussian peak profile function method, parabola method and centre of gravity method. The diffraction peaks were measured for eight tilt angles and the diffraction peak position was measured for each tilt angle with the listed peak positioning methods above. The resulting average residual stress for all eight tilt angles is presented in Table 2.7-1.

Table 2.7-1: X-ray diffraction residual stress results for different peak positioning methods [43]

Peak positioning method	Residual stress [MPa]	Standard deviation
Maximum intensity method	-142	±17
Middle position of the width at half maximum intensity method	-86.8	±16
Gaussian peak profile function method	-126	±14.8
Parabola method	-125	±14.7
Centre of gravity method	-40.2	±2

The difference between the maximum intensity method and the centre of gravity method is -101.8 ± 15 MPa, which is 253%. The study concluded that X-ray residual stress measurements will always have a degree of uncertainty in results since the results are highly reliant on the peak position method. Small standard deviations can be achieved if the correct peak positioning method is chosen, but the best method can only be determined for specific cases [43]. An in-depth study must be conducted for each sample set to determine the best method for that specific study before accurate peak positioning can be obtained. The XRD method was not considered for this study as a more accurate and reliable method was available.

2.7.3 Neutron diffraction

One of the most accurate methods for determining residual stress and strain is neutron diffraction [35, 37, 45, 46]. This method is widely used due to the deep penetration of neutrons in metallic materials allowing for non-destructive measurement of spatial dependent internal stresses. Other methods such as XRD are limited to very thin surface layers and neutron diffraction is often used to validate the XRD residual stress values to ensure the peak position was accurate [42, 43, 47, 48]. Similar to the XRD technique, neutrons behave according to Bragg's Law and measure changes in lattice spaces due to residual stress [45, 48]. Neutron diffraction has been used since 1975 to study the structure of condensed matter. Most elements exhibit low neutron attenuation coefficients, which allow neutrons to penetrate the material several micrometres to several centimetres during diffraction. The depth of penetration of neutrons is dependent on the material properties, energy of the neutrons and the presence of other elements, such as hydrogen. The interaction of neutrons with matter provides additional information to complement other techniques. This information can be used to study the behaviour of magnetic materials, biological

systems, and the distribution of hydrogen atoms in the presence of heavy metals or elements [37, 45].

Neutron assisted experiments, including diffraction, radiography, and small angle neutron scattering, are commonly used at the High Flux Reactor in Petten, Netherlands for analyzing residual stress, micro-structural features, and defects in nuclear structures. These experiments, along with similar infrastructures throughout Europe, have led to the development of international standards via collaborative research efforts that are still ongoing. To improve industrial confidence in residual stress analysis, a collaborative project was launched at the Petten research facility using neutron diffraction techniques. The collective research showed that proper alignment procedures resulted in a general positional accuracy with a standard deviation of 0.1 mm. The strain measurements were recorded with a tolerance of $\pm 10^{-4}$, corresponding to a stress range of 7 to 20 MPa. However, it is important to note that measurements taken close to the surface of samples or within regions of variable microstructure resulted in greater errors [45].

A basic neutron diffraction setup consists of a fixed neutron beam source, an object to be measured and a detector. Neutron diffraction methods can be categorised into two groups: angular variance and wavelength dispersion. Angular variance is a method of neutron diffraction that requires a monochromatic beam source and measures the diffraction angle. The wavelength dispersion method, also known as time-of-flight neutron diffraction, requires measuring the time that elapses for a polychromatic beam to penetrate a sample, neutrons to diffract at a $\pm 90^\circ$ angle and neutrons to enter the detector banks, as illustrated in Figure 2.7-3 [46, 49].

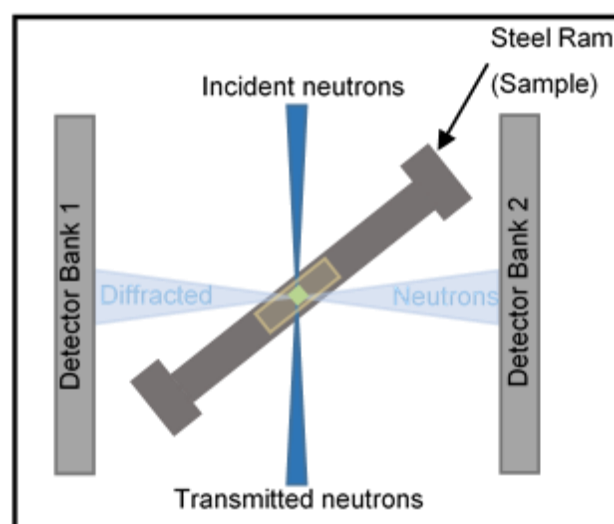


Figure 2.7-3: Neutron diffraction. Adapted from Rauch et al. [49].

In a study conducted by Whang et al. [47] the residual stress mapping in Inconel 625 fabricated using AM was done to determine a method for neutron diffraction measurements and to validate thermomechanical model predictions. The study manufactured three Inconel 625 samples with laser-based directed energy deposition (DED) and heat-treated one of the samples to 870°C for one hour. The study found good correlation between thermomechanical simulations and neutron diffraction results for samples that were not heat-treated. However, the neutron diffraction data of the heat-treated sample showed some discrepancy with the modelled residual stress. The difference was attributed to the anisotropic nature of AM materials, which cannot be fully captured by isotropic thermomechanical modelling. The study also concluded that heat treatment caused microstructural changes within the sample, altering the stress-free lattice spacing and contributing to the discrepancy in results. [47]. Adding to the observations made in this study, it has also been determined that AM components have anisotropic properties due to the layer-by-layer addition of materials [28, 50–52].

2.8 Simulating residual stress

Engineers and scientists have built on past research and have developed novel solutions for overcoming the challenges faced in modern manufacturing, including accurate design, manufacturing time and analysis. The technological breakthroughs made in computer science has further improved the design and analysis of AM processes. In the 1940s the development of the first general-purpose electronic computers set the stage for rapid growth in the field of simulation [26–29]. One of the first simulations ran on a general-purpose electronic computer was named the Monte Carlo approach and researchers made use of this method to solve problems that arose with the design of the hydrogen bomb [26].

Computer-aided engineering (CAE) software can be used to simulate manufacturing processes and verify structural and thermal properties of components before physical manufacturing begins. AM processes are challenging to simulate due to the complex thermal interaction between layers and the anisotropic material behaviour [53]. AM simulation approaches can be divided into three groups: micro-, meso- and macroscale. A microscale approach involves the complete manufacturing process including the moving heat source, non-linear thermal history, microstructural behaviour and mechanical behaviour. The mesoscale approach simplifies the process by dividing the component into elements and individually simulating each element and combining the results. This approach is less accurate than microscale but produces faster results. The macroscale approach only considers the mechanical behaviour of the material by using inherent strain [54].

MacNeal-Schwendler Corporation (MSC) Software offers a variety of simulation-based software for applications such as welding, joining and AM. These simulation packages allow for efficient design analysis, ensuring that the manufactured components meet all the requirements. Simufact Additive software focuses on AM processes and offers three calculation methods: mechanical stress; thermal stress; and thermo-mechanical stress analysis. These calculation methods can be used to predict deformation behaviour, detect hot spots, detect possible errors due to lack of fusion, examine the final microstructure, and digitally apply and test process parameters before manufacturing [55, 56].

2.9 Residual stress relieving

Residual stresses generated in structures, such as RPDs, may not always be desirable and can be relieved by a heat treatment known as stress-relief anneal [16, 17]. Stress-relief annealing is a widely used method to relieve residual stress in AM metal components. This process involves heating the component to a temperature that is specific to the material. The component is then cooled before removal from the build plate. During the heating process, the metal gains additional thermal energy which causes dislocations to move within the material, as described in Section 2.6. This process can occur without warping or distorting the component due to the presence of support structures that hold the component in place [18, 57].

2.9.1 Residual stress minimization in additive manufacturing

Process parameters such as support structure design, scanning pattern, scanning speed, laser power, layer thickness and build orientation need to be considered carefully to ensure minimum residual stress build-up [9]. Numerous studies have analysed the effect process parameters have on the distortion of AM parts, with some studies focused on minimizing distortion [9, 11–13, 15, 22]. These solutions can be categorised into three main groups: support structure design, scanning pattern design and, post-process stress-relief heat treatment. Although heat treatment studies have been performed for many years the research on the heat treatment of AM materials is limited and must specifically relate to each individual AM material to be effectively utilised. Heat treatment temperatures are determined using phase diagrams. The ramp rate and required holding time for heat treatment are however experimentally determined and therefore requires comprehensive research.

2.9.2 Stress relief annealing

The annealing temperature and holding time have a significant influence on the degree of residual stress relief and need to be considered carefully [22, 23, 58]. Several studies have investigated the heat treatment temperature and indicate improved fracture elongation at break as well as

minimized residual stress [11, 18, 22, 58, 59]. Improved fracture elongation at break is highly beneficial for thin-walled dental components, such as RPDs, due to the complex forces applied during mastication [57].

Ko et al. [59] compared the mechanical properties, microstructure and residual stress of lost-wax cast and SLM Co-Cr-Mo manufactured samples subjected to three heat treatment strategies. The samples were heat treated at 750°C, 950°C and 1150°C and the heat treatment was performed in an argon atmosphere at a heating rate of 10°C/min and holding time of 1 hour. The residual stress was measured with XRD at 0° and 90° using the Sin2Ψ technique. The results indicated that the heat treatment reduced the residual stress of each specimen however the compressive residual stress for samples heat treated to 750°C and 1150°C increased. The differential thermal expansion of different phases within the material, could have led to the development of compressive residual stresses. The microstructural changes that occur during heat treatment, such as grain growth or phase transformations, could also have altered the residual stress state of the material and may have attributed to the increased compressive residual stress. The samples heat treated to 950°C had low residual stress values [59]. The residual stress results are shown in Table 2.9-1.

Table 2.9-1: Residual stress of Co-Cr-Mo alloy manufactured with SLM (Mean ± SD). Adapted from Ko et al. [59]

Heat treatment temperature [°C]	Ψ=0 [MPa]	Ψ=90 [MPa]
As Built	280.3 ± 31.3	669.0 ± 15.9
750	19.9 ± 18.7	-99.0 ± 10.7
950	-8.7 ± 13.8	1.9 ± 15.9
1150	-32.2 ± 11.4	-35.0 ± 13

The study concluded that SLM manufactured Co-Cr-Mo dental components offer improved mechanical properties over the traditional lost-wax casting method if appropriate heat treatment was applied. The samples heat treated at 950°C and 1150°C showed a reduction in residual stress, homogenization of microstructure and mechanical properties. It was therefore concluded that the SLM manufactured Co-Cr-Mo samples must be heat treated at temperatures higher than 950°C.

Yan et al. [22] investigated the effect of two heat treatments on the mechanical properties of SLM manufactured Co-Cr RPD alloys for application in thin RPDs. Dumbbell shaped tensile bars were manufactured from Co-Cr RPD alloy and heat treated as shown in Table 2.9-2 [22].

Table 2.9-2: Heating processes for heat treatment of Co-Cr. Adapted from Yan et al. [22]

Heating process	Temperature [°C]	Ramp rate [°C/min]	Holding time at maximum temperature [min]	Total time [min]
1	500	8.33	45	105
	880	6.33	60	120
2	1100	8	30	167.5

The cooling process for both samples involved opening the furnace door when the furnace temperature was cooled to 600°C, followed by removing the specimen once the furnace cooled to 300°C. A 3-point bend test, tensile test and fracture surface analysis that was performed to obtain the mechanical properties of the samples [22]. The RPD samples exceeded the 500 MPa 0.2% yield strength requirement and the minimum 2% elongation requirement for the ISO 22674 standard for metallic materials used in removable restorations and appliances in dentistry [22, 60]. The fracture toughness of RPDs is one of the most important mechanical properties to ensure the retention clasps do not fracture. The retention clasps are very thin and are designed to fit over and around the back molars. These are subjected to high forces when removing or fitting the RPD. The sample heat-treated at 1100°C had higher strength and higher ductility than the control sample. The second heating process was, therefore, better suited for thin RPD applications. The study concluded that heat treatment at 1100°C was more efficient at releasing residual stress and also recommended that the residual stress should be measured in future studies to further investigate the effectiveness of the heat treatment strategy [22].

Wei et al. [18] investigated the microstructure and mechanical properties of SLM fabricated dental Co-Cr-Mo-W alloys at varying heat treatment temperatures. The Concept M1 Cusing SLM printer was used with a nitrogen-filled print chamber to manufacture the tensile specimens, as presented in Figure 2.9-1 [18].

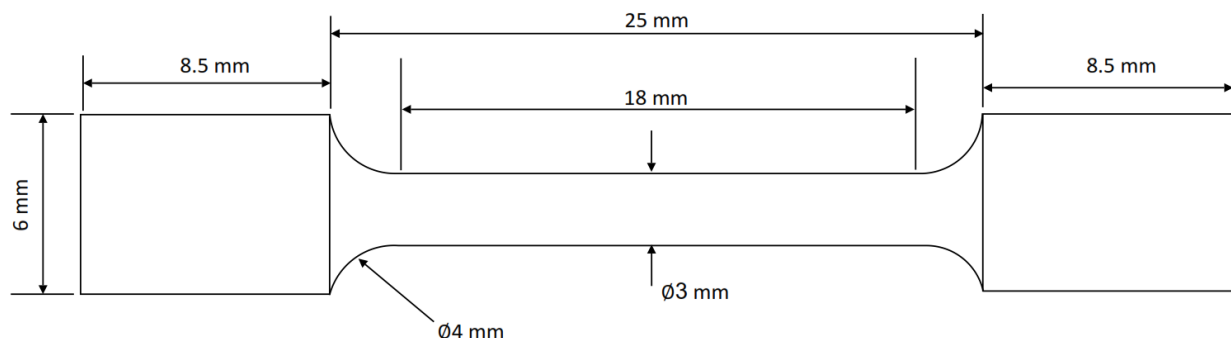


Figure 2.9-1: Tensile specimen geometry. Adapted from Wei et al. [18].

The samples were heat-treated at 750°C, 850°C, 950°C, 1050°C and 1150°C for one hour at each temperature. The samples were cooled in air, ground with emery paper, polished with diamond paste and ultrasonically cleaned in ethanol. To observe the microstructure the samples were etched in hydrochloric acid for 30 seconds. The results showed that the heat treatment allowed for molten pool boundaries to solidify and grain boundaries to form. Increased heat treatment temperature indicated grain growth. Optical metallography (OM) was used to observe the microstructure, as shown in Figure 2.9-2 [18].

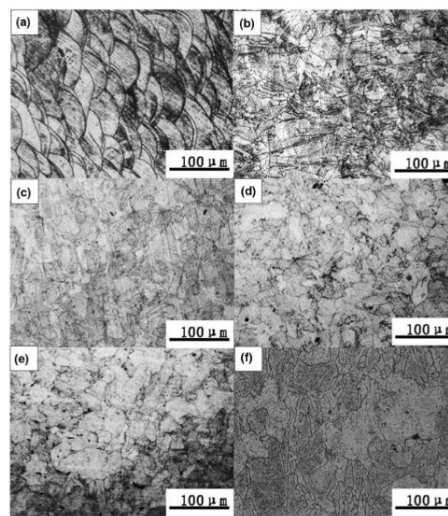


Figure 2.9-2: OM images at varying heat treatment temperatures (a) As-fabricated (b) 750°C (c) 850°C (d) 950°C (e) 1050°C (f) 1150°C. Reproduced with permission from Wei et al. [18].

The tensile testing results are presented in Figure 2.9-3. The mechanical properties that were observed were the ultimate tensile strength (UTS), offset yield strength (0.2% YS) and elongation. In Figure 2.9-3, it is shown that the samples heat treated at 1150°C showed the lowest strength and highest elongation. In contrast the samples heat treated to 850°C showed the highest strength and lowest elongation. The data obtained from the heat treatment reveals that there is a correlation between the temperature used for the heat treatment and the resulting tensile strength and elongation of the samples. Generally, as the heat treatment temperature decreases, the tensile strength of the samples increases and their elongation decreases. Conversely, as the heat treatment temperature increases, the tensile strength of the samples decreases and their elongation increases. This relationship between heat treatment temperature and the tensile properties of the samples highlights the need for finding an optimal balance between strength and

elongation for dental components. The heat treatment performed at 1050°C was found to result in the best overall balance of strength and ductility, with the tensile strength of the as-built sample remaining nearly unchanged, but its elongation being improved. The fracture surfaces were also observed and indicated that heat treatment at 750°C, 850°C and 950°C exhibited brittle fractures whereas the heat treatment at 1050°C and 1150°C exhibited ductile fracturing [18].

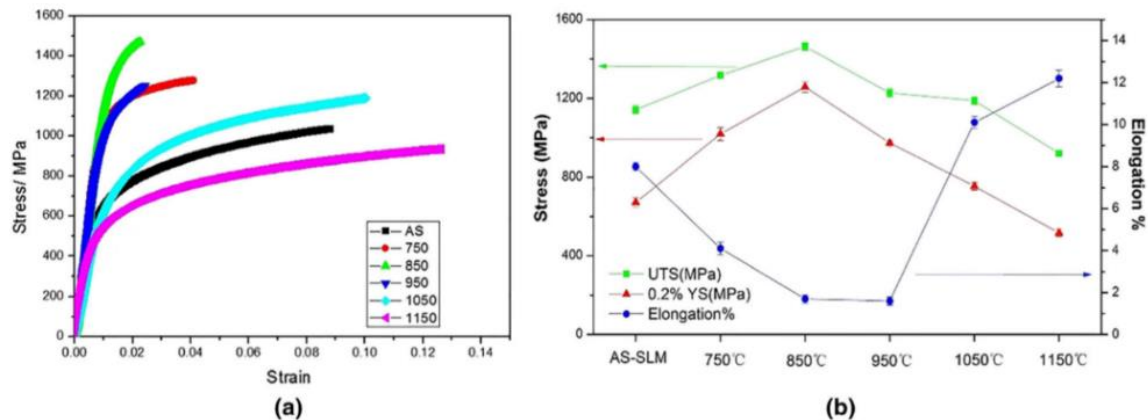


Figure 2.9-3: Mechanical properties of the specimen (a) Stress-strain curves of tensile test (b) Line graph indicating the relationship between UTS, 0.2% YS and elongation. Reproduced with permission from Wei et al. [18].

The study concluded that heat treatment must be performed at higher temperatures to ensure higher ductility of the material. The research indicated that heat treatment could lead to reinforcement and plasticization of SLM fabricated Co-Cr-Mo-W alloys [18]. The study does not provide any indication of residual stress behaviour at the varying heat treatment temperatures but suggests that careful consideration needs to be made with regards to mechanical properties when choosing heat treatment temperatures.

A similar investigation was done into the effect that heat treatment temperature has on microstructure and mechanical properties of Co-Cr-Mo alloys fabricated with SLM [58]. The samples were heat-treated at 750°C, 900°C, 1050°C and 1150°C for six hours at a ramp rate of 60°C/min and in an argon atmosphere. The samples were cooled to room temperature within the furnace, polished with emery paper, further polished with 9 µm diamond and finally polished with 0.04 µm colloidal silica suspension. Disc specimens were cut with a diamond cutter and Confocal laser scanning microscopy (CLSM) was used to obtain images of the microstructure, as presented in Figure 2.9-4.

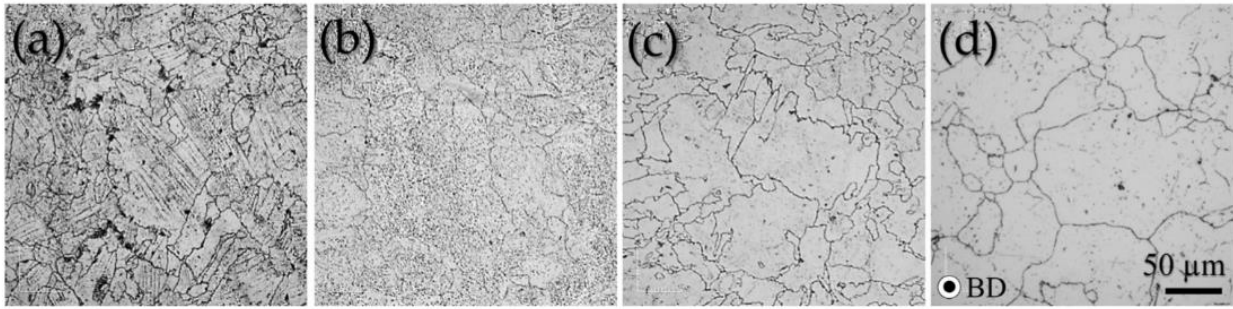


Figure 2.9-4: CLSM images of horizontal cross-section of Co-Cr-Mo alloy samples heated to (a) 750°C, (b) 900°C, (c) 1050°C, (d) 1150°C. Reproduced with permission from Kajima et al. [58].

The Vickers hardness tests (Table 2.9-3) indicated that the hardness increased for samples heat-treated at 750°C and 900°C but decreased for samples heat-treated at 1050°C and 1150°C [58]. This observation is consistent with the results reported by Wei et al in their previous study, which found that ductility decreases with heat treatment at lower temperatures and increases with heat treatment at higher temperatures. At lower temperatures, the heat treatment may not be sufficient to fully relieve stress and promote recrystallization, leading to a decrease in ductility. Conversely, at higher temperatures, the heat treatment can cause the formation of a more uniform microstructure, which can increase the material's ability to deform plastically and thus lead to an increase in ductility.

The fracture surfaces were analysed with scanning electron microscopy (SEM). The fracture mechanisms observed for samples heat-treated at 750°C, 900°C, 1050°C, were brittle fractures showing limited signs of elongation. The specimen heated to 1150°C showed signs indicative of a highly ductile fracture. The samples heat-treated to 1150°C however had much lower UTS and 0.2% YS than the as-built samples, as presented in Figure 2.9-5.

Table 2.9-3: Vickers Hardness test results. Adapted from Kajima et al. [58].

Specimen	Vickers Hardness [HV]
As-built	477
Heated to 750°C	498
Heated to 900°C	495
Heated to 1050°C	428
Heated to 1150°C	365

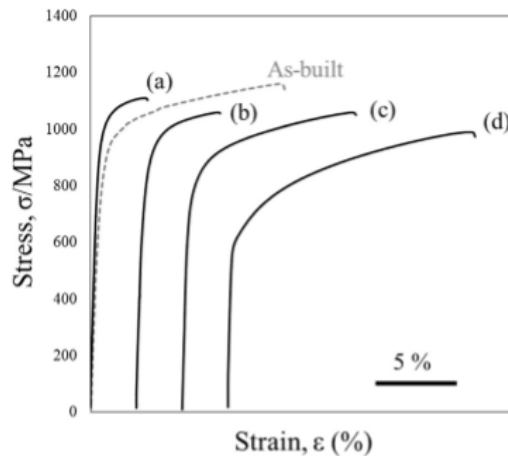


Figure 2.9-5: Stress-strain curves for specimen heat-treated at (a) 750°C, (b) 900°C, (c) 1050°C, (d) 1150°C. Reproduced with permission from Kajima et al. [58].

Longer heat treatment holding time caused increased grain growth and therefore further investigation into the effect that longer heat treatment holding time would have on the mechanical properties should be considered. The study further supports the observation that higher heat treatment temperatures are beneficial for improving the ductility of SLM manufactured parts and highlights the fact that heat treatment duration also needs to be considered.

One study conducted by Szala et al., [23] investigated the effect of annealing hold time and temperature parameters on the microstructure, hardness and strain-hardening coefficients of 42CrMo4 steel. Several heat treatment strategies were tested, as presented in Figure 2.9-6 [23]. Cylindrical samples with 15 mm diameter and 18 mm length were heat-treated according to the heat treatment strategies. The objective of the study was to determine the most time-effective annealing process [23].

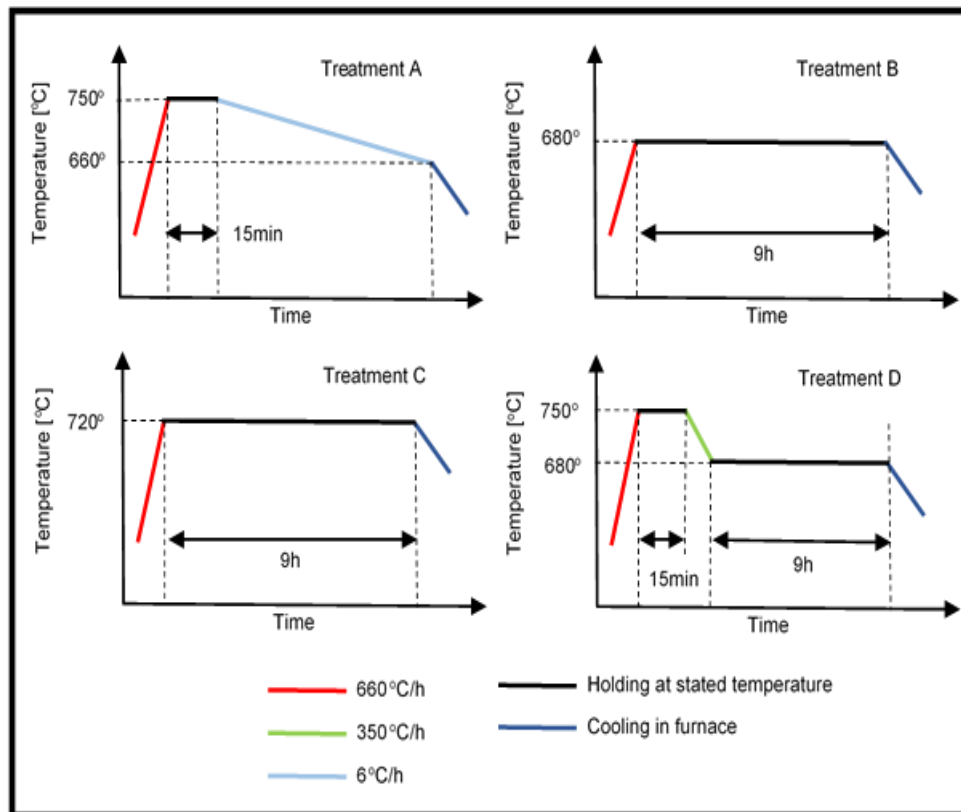


Figure 2.9-6: Time and temperature strategies for the annealing process. Adapted from Szala et al. [23].

The study concluded that the optimal annealing treatment strategy is Treatment A, presented in Figure 2.9-6 [23]. Treatment A involved a steady increase in temperature to 750°C, holding the temperature constant for 15 min and then slowly cooling the sample at a rate of 6°C per hour. This annealing strategy resulted in decreased hardness, increased strain hardening coefficients and increased cold-hardening ability [23]. The results emphasize the importance of slow cooling and suggest that shorter annealing hold times can be implemented.

CHAPTER 3 THEORY

3.1 Crystal structures

The crystal structure of a material is the arrangement of atoms in the material into a repeatable lattice. The lattice is made up of a collection of points that divide space into smaller, equally sized segments [16, 17]. There are 14 Bravais lattices that all engineering materials are made up of, as presented in Figure 3.1-1.

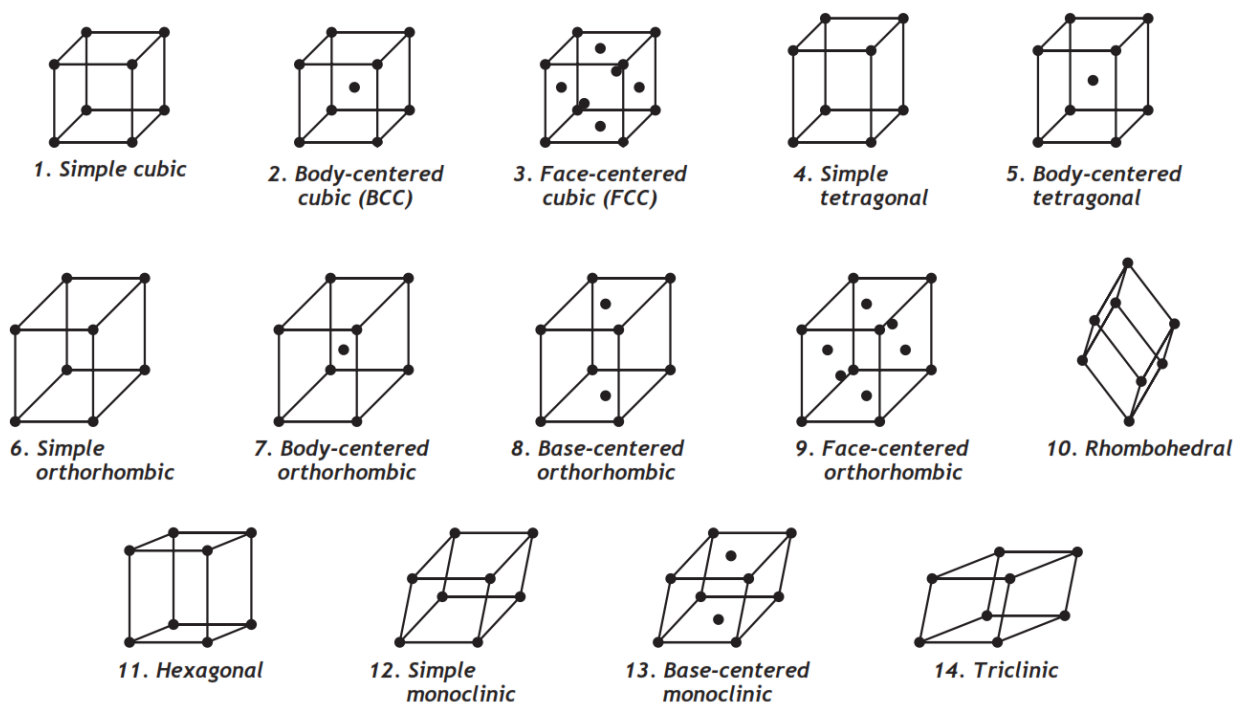


Figure 3.1-1: Fourteen Bravais lattices grouped in seven crystal systems. Adapted from Shackelford [17].

The three most common crystal structures for metals are the body-centred cubic (BCC), face-centred cubic (FCC) and hexagonal close-packed (HCP). The Co-Cr-Mo alloys exist in two crystallographic structures, HCP and FCC. The HCP (ϵ) crystallographic structure contributes to the corrosion resistance of the Co-Cr-Mo alloys through its highly ordered atomic arrangement and limited grain boundaries. This structure also enhances the strength-to-weight ratio of the alloy and has the potential to improve its biocompatibility [5, 61, 62]. The FCC (γ) phase is the austenitic phase containing many slip planes along which dislocations tend to glide. Dislocation movement is the mechanism by which plastic deformation takes place [16, 17].

3.2 Stress relief annealing

Residual stresses generated in structures may not always be desirable and can be relieved by stress-relief annealing, as described in Section 2.9. The low-temperature treatment causes recrystallization by promoting dislocation movement and thereby removing residual stress. To determine the transformation temperature at which heat treatment must be performed, phase diagrams are used [16, 17, 49].

3.3 Phase diagrams

The Co-Cr-Mo alloy powder used in the dental industry must conform to the chemical composition specified by the ASTM 75 standard, as presented in Table 3.3-1 [63, 64].

Table 3.3-1: Chemical composition specified for Co-Cr-Mo alloy by ASTM 75. Adapted from Li et al. [63].

	Co	Cr	Mo	Ni	C	Fe	Si	Mn
Co-Cr-Mo Alloy	Balance	27-30	5-7	<0.25	<0.35	<0.75	<1	<1

The principal chemical components that need to be considered when determining heat treatment temperatures is the cobalt, chromium and molybdenum since these components represent a considerable weight percentage within the material. Chromium improves the corrosion resistance of the alloy and the addition of molybdenum improves crystal grain refinement, which increases the tensile strength of the alloy [62, 65–67]. The presence of a maximum of 0.35% carbon, as allowed by ASTM F75 standards, acts as a strengthening mechanism by forming metal carbides that enhance both strength and hardness of the material. Toosi [62], calculated the phase diagram for Co-Cr-6Mo-0.25C through simulations, presented in Figure 3.3-1 [62].

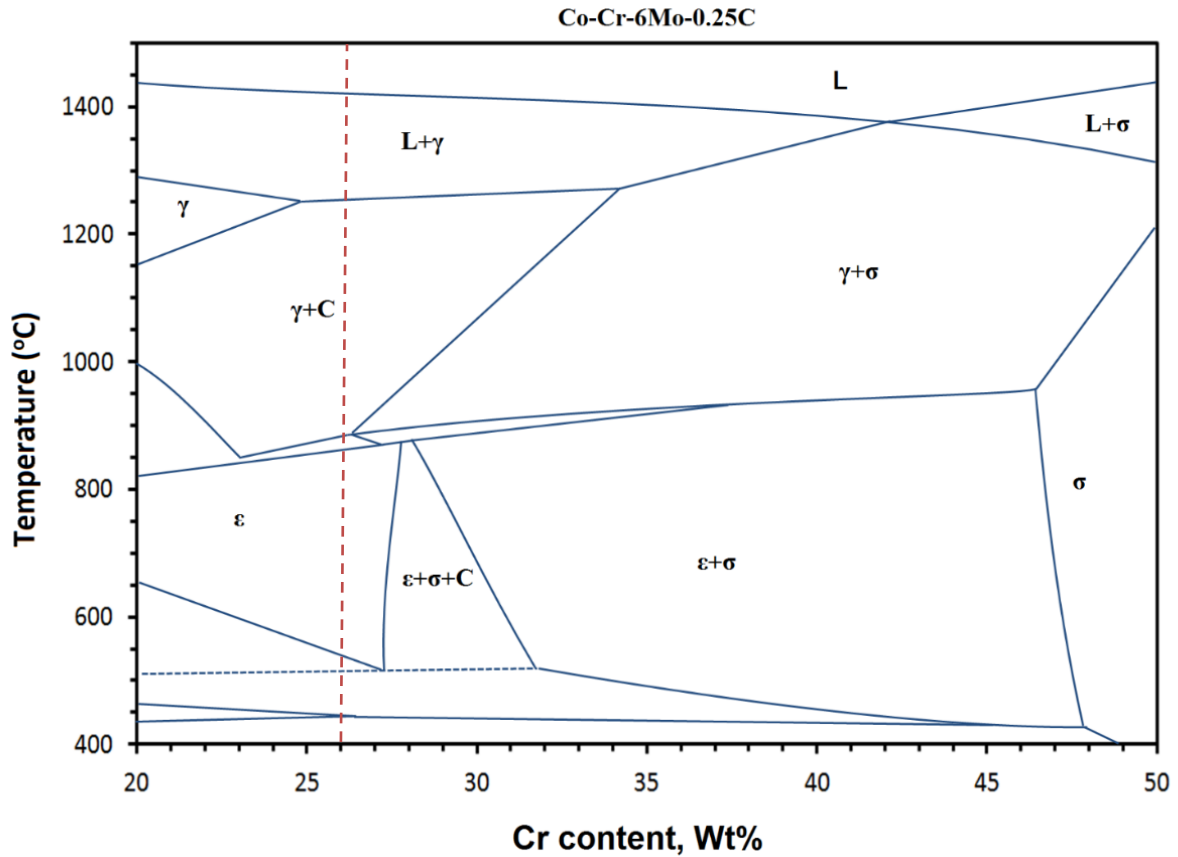


Figure 3.3-1: Phase diagram for Co-Cr-6Mo-0.25C alloy. Red dotted line indicating Cr content of 27 Wt%. With permission from Toosi [62].

Analysis of the phase diagram indicates that in a Cr range of 27-30 weight percentage (wt%) the transformation temperature from FCC to HCP is around 870°C [5, 61, 62, 65, 66, 68]. The FCC (γ) phase has highly ductile properties due to the concentration of slip planes [12]. Various studies have shown that higher heat treatment temperatures increase the volume fraction of the FCC (γ) phase and therefore increase the ductility of the material [5, 61, 62, 69]. The transformation to HCP can be observed in the phase diagram as the material cools down. At temperatures above 870°C, the transformation from γ to ϵ occurs through a martensite reaction, enabling dislocation movement on the octahedral planes of the lattice. [62, 65–67]. Under air cooling conditions, the transformation from FCC (γ) to HCP (ϵ) is slow and therefore some of the FCC structure is retained at room temperature which improves the ductile properties of the material.

3.3.1 Critical temperatures

The critical temperatures are at the binary phase boundaries. These phase boundaries indicate the vapour-liquid or liquid-solid transition temperatures. The liquid-solid critical temperature for

Co-Cr-Mo alloy powder conforming to ASTM F75 is therefore 1250°C, as seen in Figure 3.3-1. To avoid liquid transition during heat treatment the temperature should be lower than 1250°C.

3.4 Neutron diffraction

Neutron diffraction is a method used to examine the internal structure of matter [70]. Excess neutrons from the nuclear fission process are used to penetrate a material. The angular variance method uses a monochromatic beam to penetrate the material. The neutrons then diffract from the crystal structure and are captured in a detector bank to determine the diffraction angle. The diffraction angle corresponds to the lattice spacing for a specific lattice plane of the material. A diffractometer can be equipped with an XYZ orthogonal stage to move the sample into the desired positions. The monochromator is oriented such that a monochromatic narrow band wavelength is extracted from a polychromatic wide band incident beam at a specific angle [46, 71, 72]. A basic diffractometer with a Si-multi-wafer monochromator is presented in Figure 3.4-1.

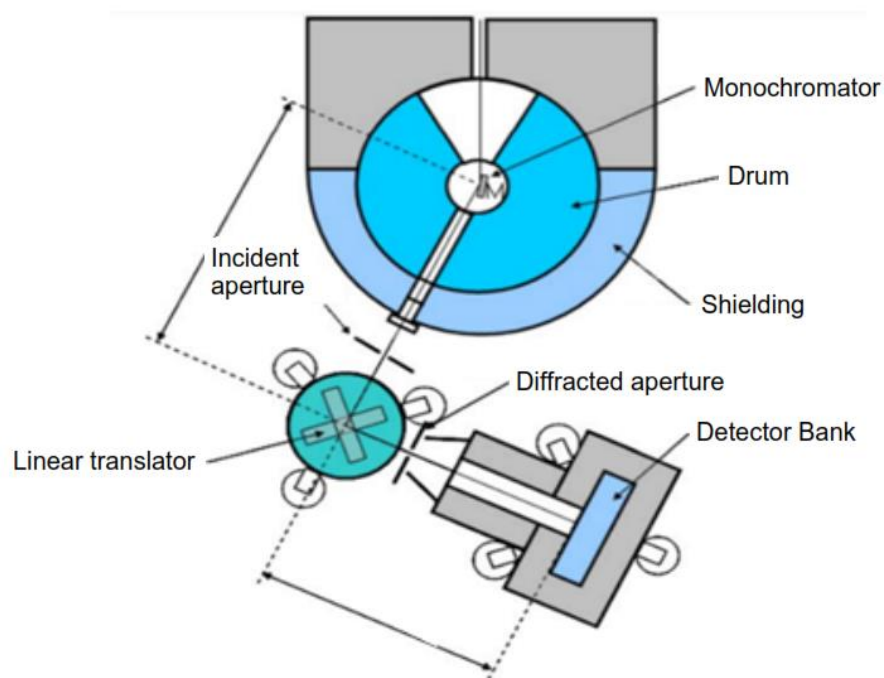


Figure 3.4-1: Basic layout of instruments for the measurement of residual stress with the use of Neutron Diffraction. Adapted from Lee et al. [73].

The lattice spacing of the material acts as an intrinsic strain gauge [45, 74]. The lattice spacing is increased or decreased depending on the tensile or compressive stress within the material. A reference value for a completely stress-free sample of material is required to use as a comparison. An average strain is measured over a small volume within the sample relating to the incident

beam size. The measured volume is called the gauge volume by analogy with the gauge length sampled by a conventional wire strain gauge [73]. The gauge volume is set by adjusting the size of the incident and diffracted apertures through which the incident and diffracted beam travels, as indicated in Figure 3.4-2.

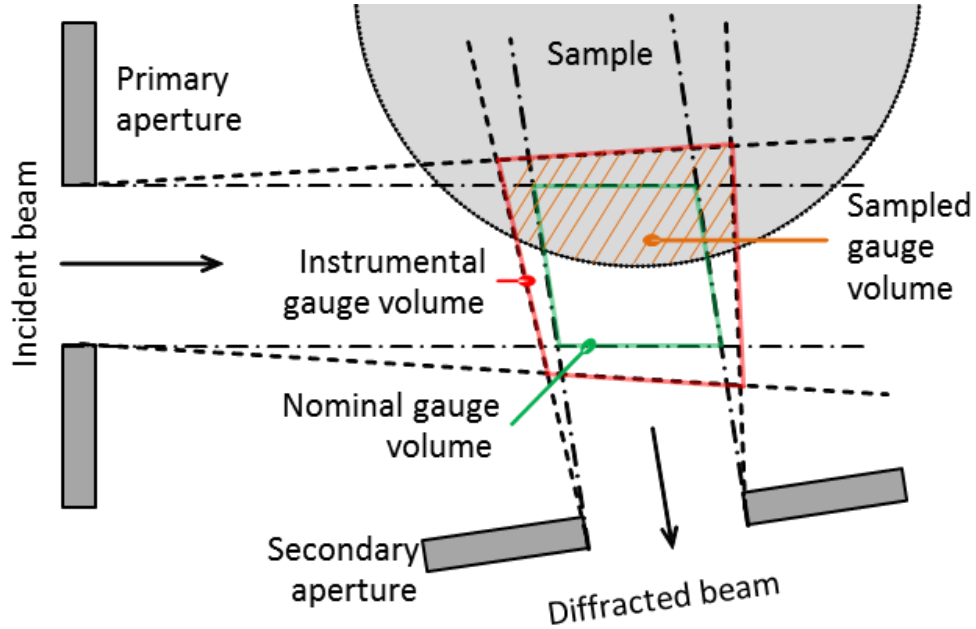


Figure 3.4-2: Illustration of neutron gauge volume.

The neutron diffraction technique for measuring strain utilises Bragg's law. The relationship between the lattice spacing (d^{hkl}) of the atomic planes described by Miller indices (hkl), the angle of the corresponding diffraction peak ($2\theta^{hkl}$), the wavelength of the radiation (λ) and order of diffraction (n) is given by Bragg's Law, as presented in Equation (1) [46, 74]:

$$\lambda = 2 d^{hkl} \sin \theta^{hkl} \quad (1)$$

The elastic lattice strain at a specific position in the material with a reflection (hkl) and a reference stress-free lattice spacing of d_0^{hkl} can be calculated with Equation (2) [74, 75].

$$\varepsilon_{ii}^{hkl} = \frac{d_{ii}^{hkl} - d_0^{hkl}}{d_0^{hkl}} \quad (2)$$

Where i is either x, y or z . The mechanical stress state (σ_{ij}) at a position in a material is represented by a second-order tensor given by Equation (3).

$$\sigma_{ij} = \frac{\partial F_i}{\partial S_j} \quad (3)$$

Where F_i represents the component of force that acts in direction x_i on the element of volume characterized by surface area ∂S_j for which the normal direction is x_i [76]. Since the stress tensor is symmetrical and the normal stress acts along a defined axes only three unknowns need to be determined. The residual stress is calculated from measured d -spacing data and estimated strains (ϵ), using Hooke's law: $\sigma_{ij} = C_{ijkl} \times \epsilon_{kl}$, where C_{ijkl} is the elasticity coefficient. The diffraction elastic constants (s_1 and $\frac{1}{2}s_2$) are related to Young's modulus and the Poisson ratio through Equations (4) and (5).

$$s_1 = -\frac{\nu}{E} \quad (4)$$

$$\frac{1}{2}s_2 = \frac{(1 + \nu)}{E} \quad (5)$$

The reference value is determined by measuring the lattice spacing of a sample that is completely stress relieved or through calculation by applying appropriate theory. The first method requires various destructive processes to ensure that the reference sample is completely stress-free. For samples created with the SLM process, this is a very time consuming and expensive method. The second method allows for the determination of the reference lattice parameters by using a thin-walled sample. This technique has been implemented in multiple studies and has been accepted to be a reliable method [75, 77–79]. When a thin-walled sample is used a plane stress state $\sigma_{xx} = 0$ is assumed and d_0^{hkl} can be calculated for each measuring position with Equation (6).

$$d_0^{hkl} = \frac{d_{xx}^{hkl} + \nu(-d_{xx}^{hkl} + d_{yy}^{hkl} + d_{zz}^{hkl})}{1 + \nu} \quad (6)$$

Where ν is Poisson's ratio and the interplanar spacing in X, Y and Z directions is presented by d_{xx}^{hkl} , d_{yy}^{hkl} and d_{zz}^{hkl} , respectively. The internal stress can then be calculated with Equation (7).

$$\begin{pmatrix} \sigma_{xx} \\ \sigma_{yy} \\ \sigma_{zz} \end{pmatrix} = \frac{E}{(1 + \nu)(1 - 2\nu)} \begin{pmatrix} 1 - \nu & \nu & \nu \\ \nu & 1 - \nu & \nu \\ \nu & \nu & 1 - \nu \end{pmatrix} \begin{pmatrix} \epsilon_{xx} \\ \epsilon_{yy} \\ \epsilon_{zz} \end{pmatrix} \quad (7)$$

Assuming plane stress state enables simplification of the neutron diffraction measurement and calculation process for AM samples.

3.5 Simufact Additive

AM processes are highly complex thermo-mechanical processes typically requiring hours of simulation. The mechanical and thermal calculation methods have been developed to decrease simulation time by focusing on specific result requirements. The mechanical calculation method uses the inherent strain method to give insight into distortion behaviour and internal stresses. The inherent strain is a non-elastic strain produced in the component by the AM process. The inherent strain method assumes that the laser path and subsequent size is very small in comparison to the manufactured component and that each laser pass has a similar thermo-mechanical history [69]. The need for thermal calculations is therefore neglected and the calculations become purely mechanical. The initial inherent strain values can be determined through calibration and can then be used to calculate distortion and residual stress [56].

The thermal calculation method gives insight into thermal processes such as peak temperatures, temperature gradients and energy dissipation rates. The thermo-mechanical calculation method combines mechanical and thermal calculations to give full insight into the additive process. This calculation method takes all possible thermal and mechanical aspects into consideration and therefore delivers the most accurate results but has the largest computational time and effort [67].

3.5.1 Inherent strain

The inherent strain is dependent on the material properties and the process parameters. During the simulation process various strain increments are calculated consisting of elastic strain ($d\varepsilon^e$); plastic strain ($d\varepsilon^p$); thermal strain ($d\varepsilon^{therm}$); and phase transformation induced strain ($d\varepsilon^{phase}$) [80]. The summation of all the increments is equal to the total residual strain (ε_T) as represented by Equation (8) [56, 81]:

$$\varepsilon_T = \varepsilon^e + \varepsilon^p + \varepsilon^{therm} + \varepsilon^{phase} \quad (8)$$

The plastic strain ($d\varepsilon^p$) is calculated using flow curves at different temperatures. The phase transformation induced strain ($d\varepsilon^{phase}$) has been proven to have a negligible influence on the total inherent strain [82]. The thermal strain ($d\varepsilon^{therm}$) is calculated using Equation (9).

$$\varepsilon^{therm} = \alpha(T)\Delta T \quad (9)$$

The symbol α represents the volumetric expansion factor, which is determined through the thermo-mechanical calibration process. The inherent strain (ε^*) is defined as the difference between the total residual strain and elastic strain, as presented in Equation (10).

$$\varepsilon^* = \varepsilon_T - \varepsilon^e \quad (10)$$

By substituting Equation (8) into (10) the inherent strain is defined as the sum of the thermal strain and the plastic strain. Elastic FEM is used to calculate residual stress from inherent strain values by using Equations (11) and (12).

$$[K][u] = [f^*] \quad (11)$$

$$f^* = \int [B][D][\varepsilon^*]dV \quad (12)$$

Where $[K]$ represents the elastic stiffness matrix; $[u]$ represents the nodal displacement vector; f^* represents the nodal force vector; $[B]$ represents the nodal deformation matrix; and $[D]$ the material elastic matrix. The displacement vector $[u]$ is calculated by using Equation (11) and can then be used to calculate the total strain (ε) and residual stress (σ) with Equations (13) and (14) [56, 81]:

$$\varepsilon = [B][u] \quad (13)$$

$$\sigma = [D](\varepsilon_T - \varepsilon^*) \quad (14)$$

The computational flow for inherent strain values through FEM analysis can be summarized as presented in Figure 3.5-1.

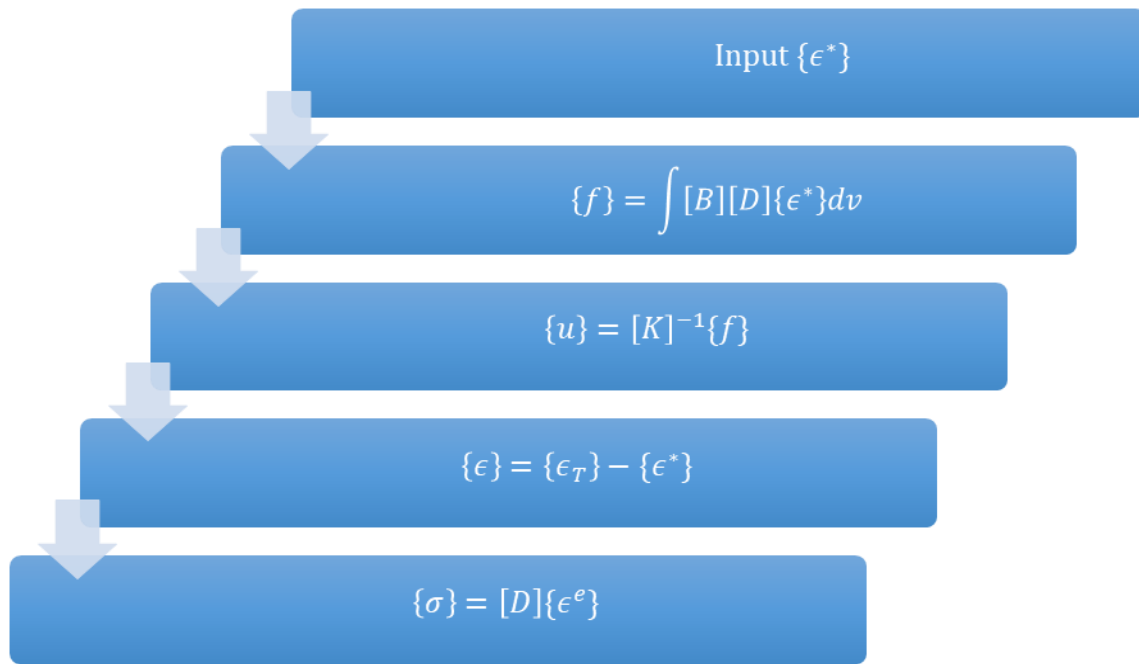


Figure 3.5-1: Diagram depicting the computational flow for inherent strain calculations through FEM analysis [56, 81]

3.5.2 Simulation calibration

Thermo-mechanical simulations are highly accurate, but they require thorough calibration procedures to ensure the most precise results. The calibration process is divided into three subsequent parts: mechanical, thermal and thermo-mechanical [80, 83].

3.5.2.1 Mechanical calibration

Mechanical calibration requires cantilever beams to be manufactured with the specific machine, material and print parameters for which the inherent strains are required [80, 83]. The cantilever beams, as presented in Figure 3.5-2, after being partially cut, experience deformation due to residual stress. The distortion in the z-axes is then measured. The sample deformation is presented in Figure 3.5-3.

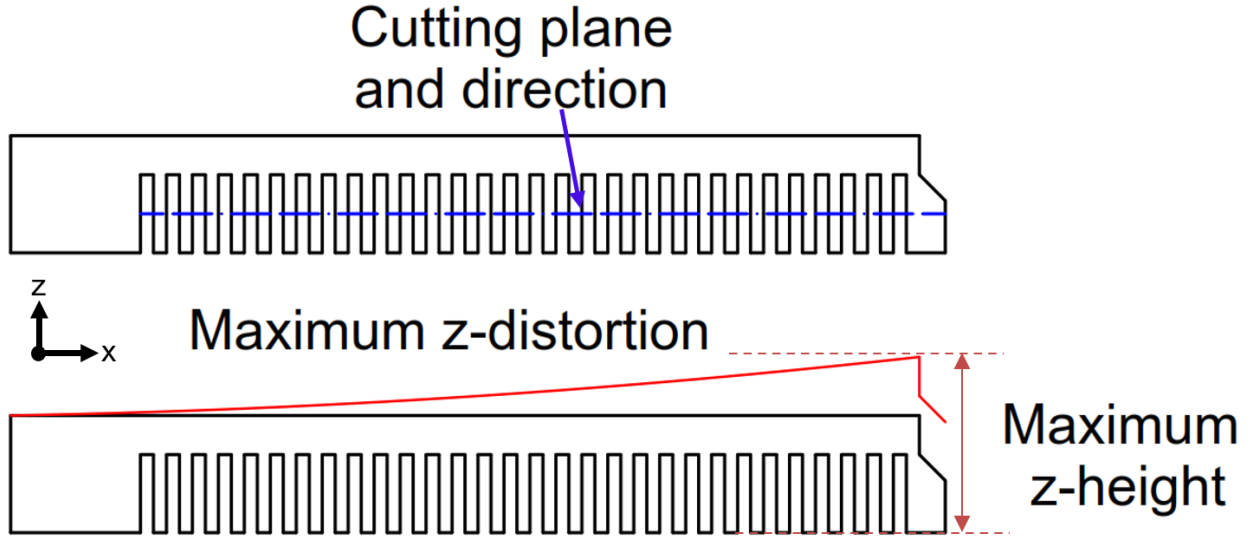


Figure 3.5-2: Mechanical calibration cutting and measuring method. Adapted from Simufact Engineering [83].

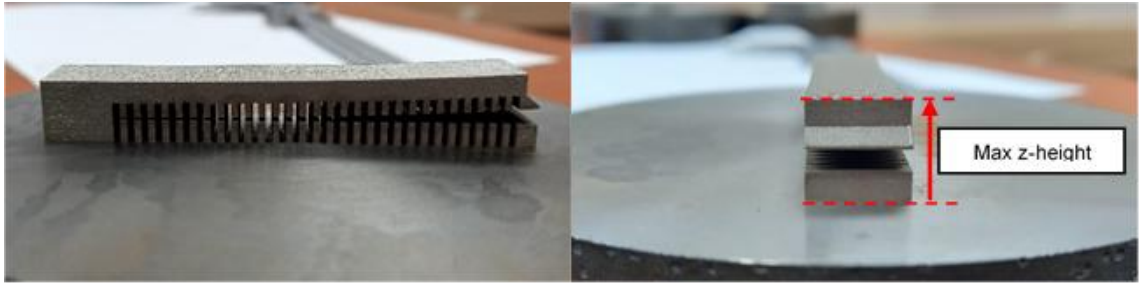


Figure 3.5-3: Example of cantilever beam distortion

The manufacturing and cutting process is then simulated within the simulation software and the measured maximum z-height values are added as input values [80, 83]. The software estimates the value of inherent strain values by using the least squares method, as presented with the Equation (15) [55, 81].

$$\varepsilon = \sum_{i=1}^n r_i^2 \quad (15)$$

Where S is the inherent strain; r_i is the difference between maximum z-height at point i of the simulation ($z_{i,sim}$) and the maximum z-height at point i of the experiment ($z_{i,exp}$); i is the number of running index; and n is the number of samples. The inherent strain values calculated by the

software are then presented as ε_{xx} , ε_{yy} , and ε_{zz} . These values can be used to run simulations through the mechanical calculation method [55, 80, 83]

3.5.2.2 Thermal calibration

Thermal calibration is performed to determine the exposure energy fraction (EEF) responsible for the peak temperature that the sample experiences during manufacturing. EEF is described as the ratio between the fraction of energy that melts the powder and the fraction of energy that dissipates into other layers [80]. The cantilever beam in the mechanical calibration process is used as the sample geometry and the thermal calibration simulation is performed with the same process parameters used during the mechanical calibration [80].

3.5.2.3 Thermo-mechanical calibration

Thermo-mechanical calibration is performed to calibrate the volumetric expansion factor (VEF). The VEF is required to determine the thermal strain. The thermal calibration simulation is adapted for the thermo-mechanical calibration by incorporating a cutting stage after the completion of the build stage. The EEF value determined in the thermal calibration is used as an input that would otherwise have been estimated by the programming. A target Z-distortion value, as obtained from the mechanical calibration, is then entered into the simulation and the software varies the VEF until the required Z-distortion is reached. The calibrated values for EEF and VEF are now used to perform all simulations with the same simulation parameters [80].

CHAPTER 4 METHODOLOGY

4.1 Introduction

A scientific approach was utilised to realise and understand the research fully. The process is illustrated in Figure 4.1-1. This chapter provides the experimental design, measuring and analysis techniques and data processing methods. The experimental design is discussed in detail and information on the test specimen material, manufacturing and design is provided. Post-processing is fully outlined and described. Lastly, the neutron diffraction measurements and data analysis methods are discussed.

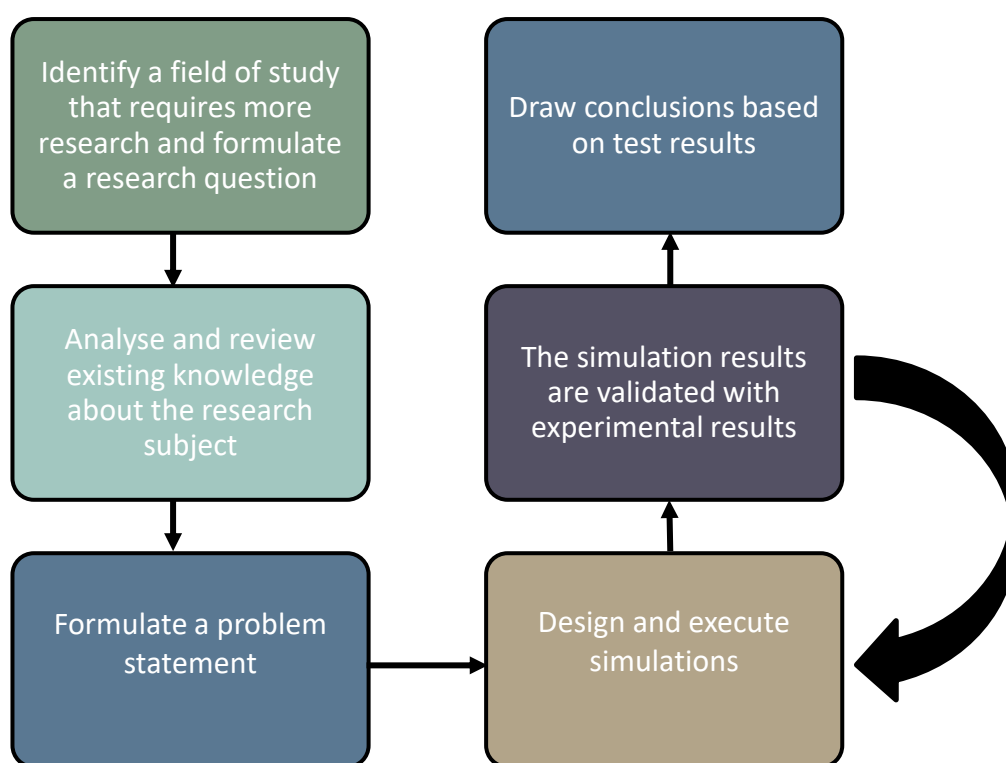


Figure 4.1-1: Scientific approach of research methodology.

4.2 Test specimen

4.2.1 Material

The material used throughout the study is medical grade Co-Cr-Mo alloy conforming to the ASTM F75 standard [64]. The powder is manufactured by Praxair and the specific batch used has the chemical composition and mechanical properties as shown in and .

Table 4.2-1: Chemical composition of Praxair Co-Cr-Mo Powder

Co	Cr	Mo	Ni	C	Fe	Si	Mn
Balance	28.31	6.15	0.17	0.08	0.37	0.25	0.14

Table 4.2-2: Mechanical properties of Praxair Co-Cr-Mo Powder compared to literature

	0.2% Yield Strength [MPa]	Ultimate Tensile Strength [MPa]
Wei et al. [18]	1019 ± 33	1142 ± 21
Lee et al. [57]	834.24 ± 55.24	1025.03 ± 62.74
Ko et al. [59]	849 ± 1.2	1024 ± 3.9
Praxair	691.36	927.4184

4.2.2 Manufacturing

The samples were manufactured with the SLM process on an ORLAS Creator SLM machine located at the North-West University, Potchefstroom. The SLM parameters used were provided by O.R Lasertechnologie GmHB (OR LASER) and are indicated in Table 4.2-3. These printing parameters were kept constants for all the samples.

Table 4.2-3: Print parameters

Parameter	Value
Laser power	145 [W]
Laser speed	600 [mm/s]
Layer height	0.025 [mm]
Inert gas	Nitrogen

A total of 12 samples were fabricated by implementing a dual orientation approach, in which both horizontal and vertical orientations were manufactured for each heat treatment strategy. The horizontal and vertical orientated samples are presented in Figure 4.2-1. Neutron diffraction was performed on both samples to determine the measurability of the samples based on the interference of the build plate and the measuring time.

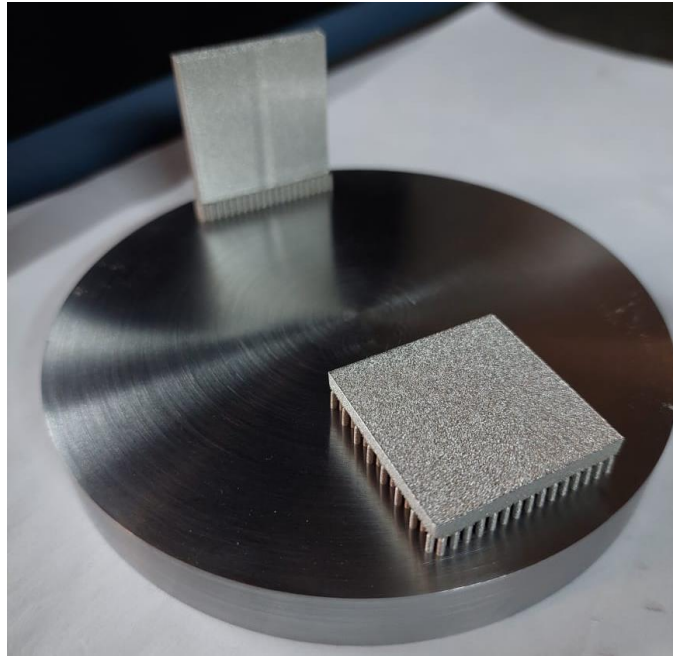


Figure 4.2-1: Co-Cr-Mo sample pair printed on a build plate

The purpose of this study was to test residual stress and therefore it was important to note that the samples were not removed from the build plate before testing as removal from the build plate would result in residual stress relief due to warping. The residual stress in these samples can also be affected by external deformation and vibrations due to handling. It was therefore important to carefully handle the samples to ensure the highest accuracy results.

4.2.3 Dimensions

The test specimen dimensions shown in Figure 4.2-2 were selected to simplify neutron diffraction measurements. The theory on neutron diffraction (Section 3.4), indicated that thin-walled samples simplify the measurement protocol required to determine the residual strains. The thin-walled models also gave insight into the residual stress build-up for thin-walled components, such as RPDs, manufactured with SLM.

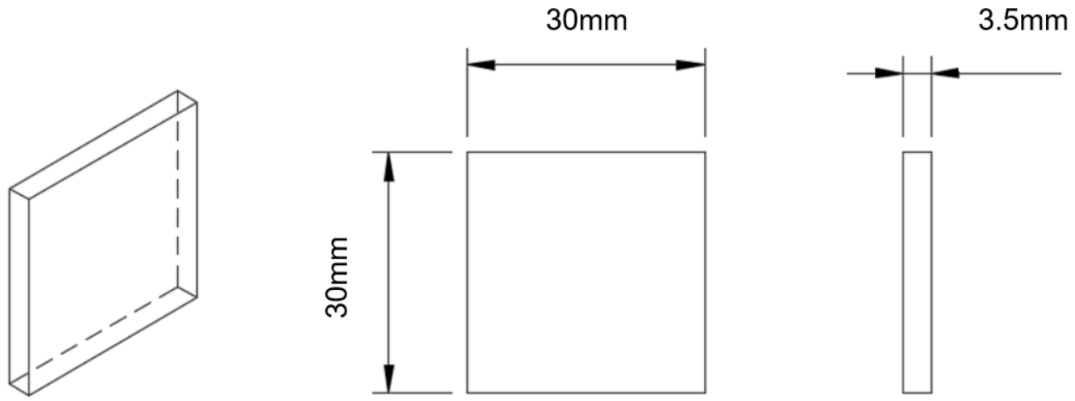


Figure 4.2-2: Test specimen dimensions

As a neutron beam passes through a material, neutron intensity is reduced as neutrons are attenuated through absorption and scattering mechanisms. An important consideration was to minimise the thickness of the build plate since the neutron beam would travel through the build plate and the sample before entering the detector bank. The percentage intensity lost during the neutron diffraction process was calculated using Lambert's law to measure the intensity of the transmitted neutron beam. The incident neutron beam intensity is a measure of the number of neutrons in the incident beam that enters the sample and the transmitted neutron beam intensity is a measure of the number of neutrons that are recorded by the detector bank. Equation (16) was used to calculate the transmitted beam intensity (I) where; I_0 is the incident beam intensity; u is the attenuation coefficient which is a measure of how easily a material can be penetrated by an incident neutron beam; and t is the path length [72, 84, 85]

$$I = I_0 \times e^{-u \times t} \quad (16)$$

The South African Nuclear Energy Corporation SOC Limited (Necsa) facility expected the Bragg's peaks of Co-Cr-Mo alloy to have been measured at an angle close to 99° and the path length was expected to be constant since the sample was measured at a constant depth from the surface. The path length was therefore calculated using Equation (17), where d represented the thickness of the build plate:

$$t = d / \cos\left(\frac{99.52}{2}\right) \quad (17)$$

Assuming the initial intensity was 1 and using attenuation coefficient of the build plate material (Inconel 718) as 1.19 cm^{-1} , the transmitted beam intensity was calculated for varying build plate thicknesses. The graph presented in Figure 4.2-3 illustrates the decrease in transmitted neutron beam intensity due to an increase in build plate thickness.

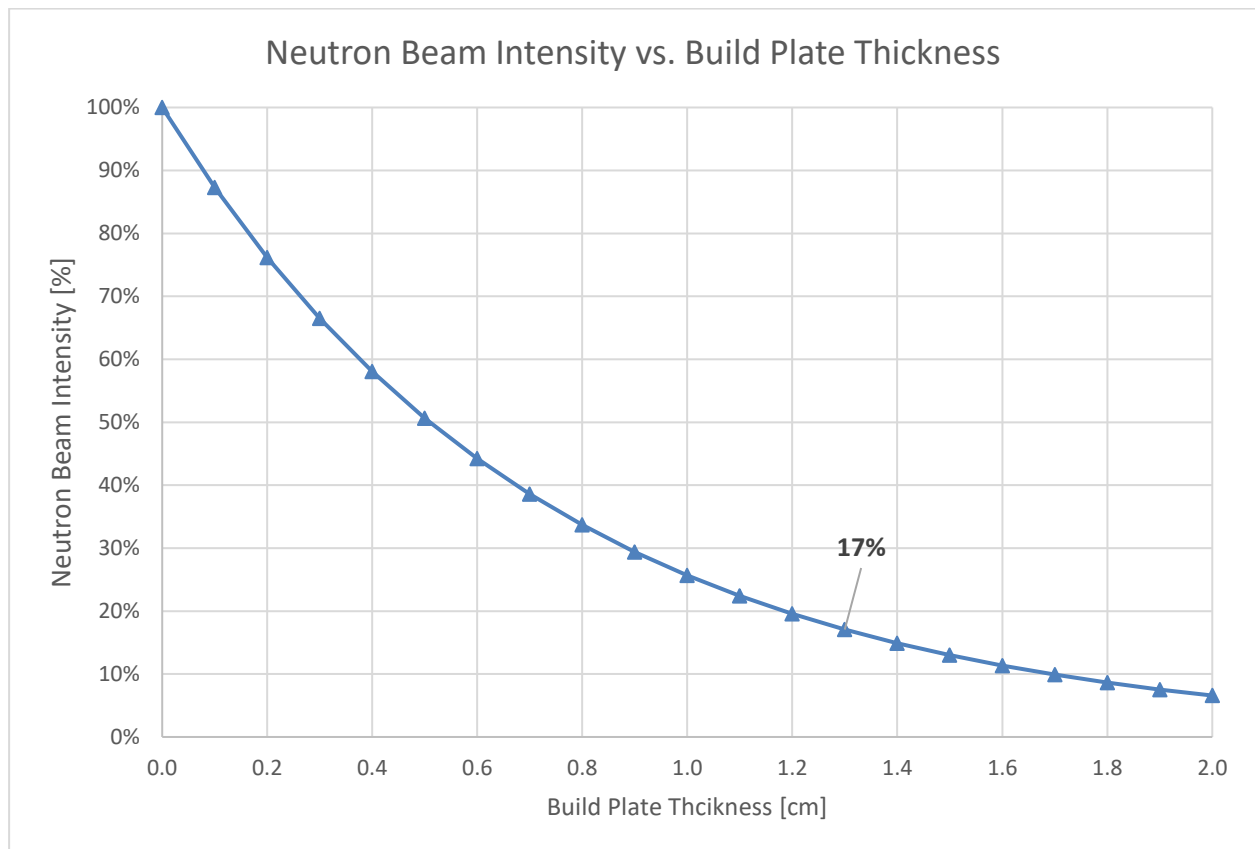


Figure 4.2-3: Graph illustrating neutron beam intensity versus material thickness.

Analysis of Figure 4.2-3 indicated that the transmitted neutron beam intensity exponentially decreased with increase in build-plate thickness. The time required to achieve accurate results was directly linked to the transmitted neutron beam intensity and negatively influenced by the thickness of the build plate. The minimum thickness for a build-plate in the ORLAS Creator was determined to be 1.3 cm due to the clamping mechanisms used within the build chamber which results in low neutron beam intensity and extended measuring durations.

4.3 Heat treatment

4.3.1 Furnace

The furnace used in this study was a Nabertherm LH 120/12 model with a maximum temperature of 1200°C , and located at the North-West University, Potchefstroom. This furnace used a

thermocouple to measure the temperature and a control panel to register the temperature and apply adjustments accordingly. The programming required two inputs: the temperature and the time in which to reach the temperature. The heating rate required to reach the set temperature within the set time was determined by the program using Equation (18); where R is the heat rate, T is the temperature; and t is the time:

$$R = T/t \text{ [}^{\circ}\text{C/min]} \quad (18)$$

The power applied by the system to the heating elements was adjusted to achieve the heating rate and the control panel continuously regulated the power according to the current temperature measurement. The continuous power regulation resulted in thermal gradients within the heating chamber for which the inert gas chamber acted as a buffer zone to counteract the temperature spikes and gradually heat the sample.

4.3.2 Heating strategy

The heating strategy was determined by fitting the highest possible constant heat rate to the heat rate curve of the furnace with three subsequent time and temperature set points. The maximum heat-rate of the furnace was established by manually noting the temperature at varying time intervals whilst the furnace is heating up at maximum power to 1065°C. The graph presented in Figure 4.3-1 indicates the heat rate of the furnace and best possible heating strategy fitted to the curve in blue.

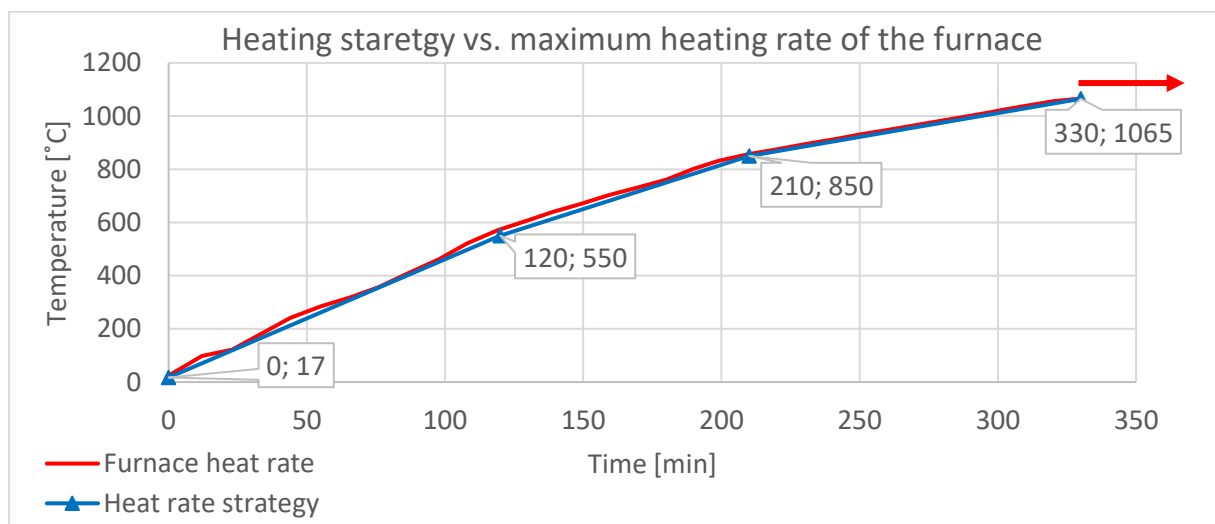


Figure 4.3-1: Line graph illustrating the heating strategy determined by fitting three set temperature and time points with the maximum heating rate of the furnace.

The literature on heat treatment temperatures (Section 2.9), indicated that the heat treatment temperature must be between 900°C and 1150°C to ensure sufficient residual stress relief whilst maintaining above standard mechanical properties such as ductility and UTS [3, 4, 22, 23, 52, 58, 86, 87]. The furnace capabilities were considered and a maximum temperature of 1065°C was achievable. Five of the test samples were heat treated at a maximum temperature of 1065°C. Electric Optical Systems (EOS), a well-known Co-Cr-Mo powder manufacturer, suggested heat treatment at 750°C for one hour. One sample was therefore heat treated according to the EOS material specifications and the residual stress was compared to the other samples [88]. The holding time variations were also derived from literature. The maximum holding time previously studied was 6 hours and the minimum was 15 minutes [3, 4, 22, 23, 42, 52, 86, 87]. For this study, the samples were heat treated at 15 minutes, one hour, two hours and three hours which ensured a broad range of heat treatment durations were comparable.

The ramp rate was chosen due to the heating capabilities of the furnace and is shown in Figure 4.3-1. The steady ramp rate strategy and nitrogen gas chamber within the furnace facilitated homogenous heating and prevented thermal gradients. The holding time variation took place after the samples reached the set temperature of 1065°C. The furnace automatically opened two air inlet ports when the hold time was reached. Cooling took place at an average rate of 2°C/min.

A summary of the heat treatment strategies that were simulated is presented in Table 4.3-1.

Table 4.3-1: Summary of heat treatment strategies.

Symbol	Set temperature [°C]	Holding time at set temperature [min]
CT	NA	NA
HT1	1065	15
HT2	1065	60
HT3	1065	120
HT4	1065	180
HT5	750	60

In Table 4.3-1 CT refers to the control test with no heat treatment and HT# to each heat treatment strategy,

4.4 Neutron Diffraction

Since neutron diffraction is a reliable standard for residual stress measurement, it was used to measure the inherent strain in each sample. The experimental neutron work was conducted at

the Neutron Diffraction Facility (NDIFF) of Necsa. NDIFF is located at Beam Port 5 of the South African Fundamental Atomic Research Installation 1 (SAFARI-1) nuclear research reactor, Pelindaba, South Africa. The internal strain was measured with the Materials Probe for Internal Strain Investigation (MPISI) angular dispersive neutron diffractometer [71, 72]. A nominal gauge volume of $2.0 \times 2.0 \times 2.0 \text{ mm}^3$ was established, as described in Section 3.4, by the width and height of the incident beam aperture, as well as the width of the diffraction-side aperture. This nominal gauge volume results in an instrumental gauge volume shown in Figure 4.4-1. The instrumental gauge volume with a side length of 2.25 mm allows for full submersion of the gauge volume within the sample area.

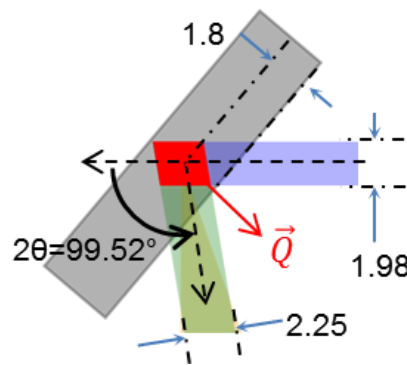


Figure 4.4-1: Instrumental gauge volume.

The measuring points were located at $x = 1.85 \text{ mm}$ and are shown in Figure 4.4-2. The side length of 2.25 mm implies that the closest measurement to the surface ensuring a fully submerged gauge volume is 1.71 mm. Measurements closer to the surface of the sample experienced partial gauge volume illumination and pseudo strains, which influenced the result. Correcting the near-surface measurements with the build plate attached to the sample was not possible due to positional constraints and therefore these measurements were not considered for comparison in this study, indicated as red dots in Figure 4.4-2.

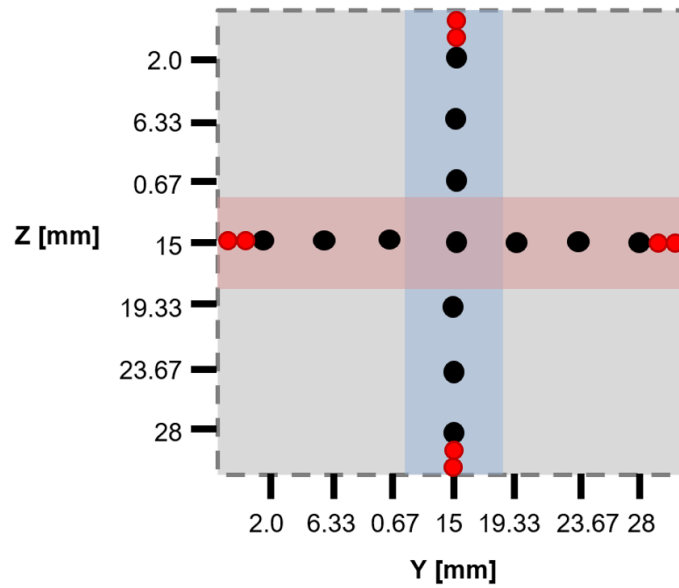


Figure 4.4-2: Coordinates of measurement positions.

The calculation procedure, as outlined in Section 3.4, requires the use of Bragg's Peak data to calculate the lattice spacing at each measuring position and in each axial direction. The variation in the strained lattice plane spacing (d_{hkl}^i) with reference to the strain-free lattice plane spacing (d_{hkl}^0) was used to calculate the directional elastic lattice strain (ϵ_{hkl}^i) in the material using Equation (2). From this calculation, it was evident that accurate representative " d^0 " values are required for the analysis procedure. The mechanical stress was calculated using Equation (3) and material properties shown in Table 4.4-1. Relevant material properties and constants used for this study are given in Table 4.4-1 and were determined from CoCr₂₀Mo₁₀ alloy, which has a similar composition to the ASTM F75 specification, as ASTM F75 elastic constants have not been identified from literature [89, 90].

Table 4.4-1: Material properties and constants used for neutron diffraction.

Material	Lattice plane (<i>hkl</i>)	s_1 [MPa ⁻¹ ×10 ⁻⁶]	$\frac{1}{2} s_2$ [MPa ⁻¹ ×10 ⁻⁶]	E [GPa]	ν [-]
CoCr ₂₀ Mo ₁₀	(311)	-1.70984	6.891192	193	0.33

The internal residual stress was calculated for each reference axes using the generalized form of Hooke's law given in Section 3.4 by Equation (10).

4.5 Simufact Additive simulations

4.5.1 Simulation calibration

Simufact Additive simulations were performed by firstly calibrating the simulation process, as described in Section 3.5. The first calibration process performed was the mechanical calibration where three cantilever beams were manufactured parallel to the x-axes (0°) and three parallel to the y-axes (90°) with dimensions presented in Figure 4.5-1. The print parameters shown in Table 4.2-3 were used as inputs with a Co-Cr-Mo powder conforming to the ASTM F75 standard. The printed cantilever beams were cut at a height of 2.5 mm and the maximum distortion from the build plate was measured at the fixed end (position A), in the middle (position B) and at the cut end of the sample (position C), shown in Figure 4.5-1. The values at each measuring position were averaged and used as inputs in the calibration simulation.

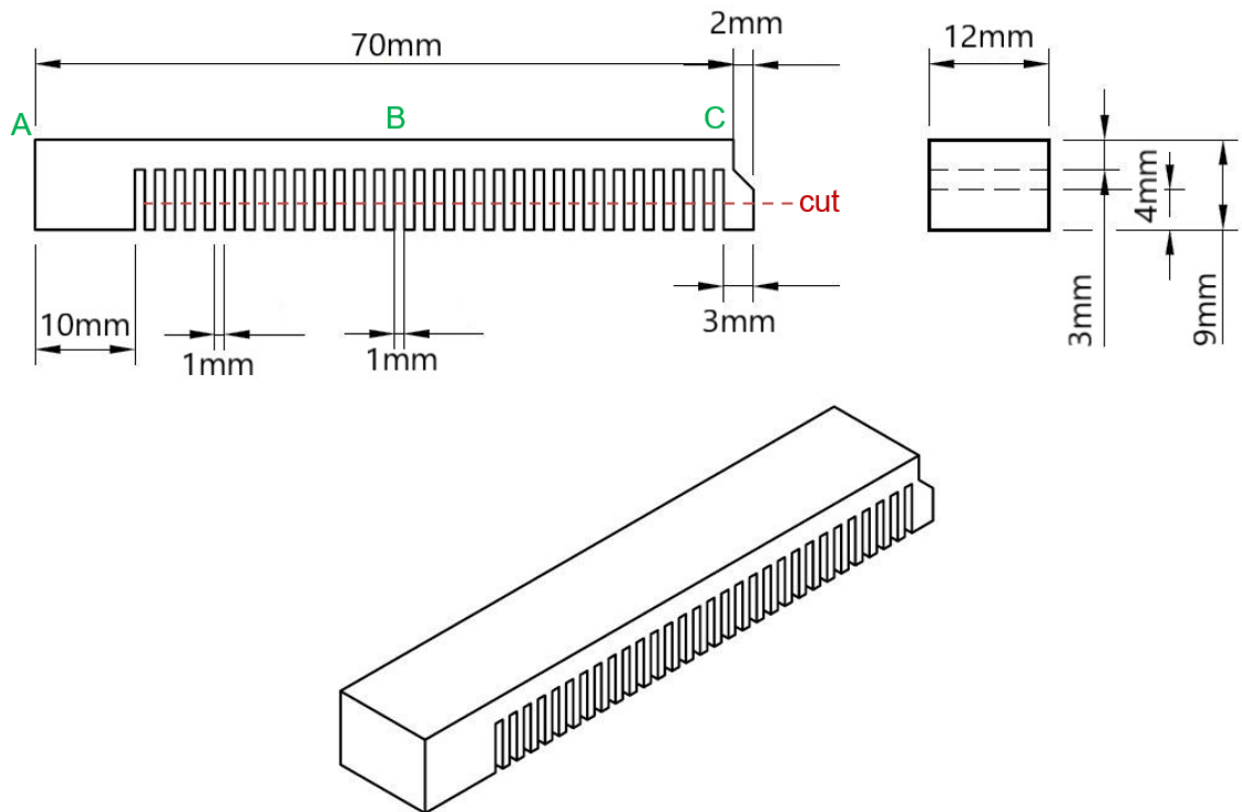


Figure 4.5-1: Calibration cantilever beam dimensions, cutting position and measuring positions A, B and C.

Simufact Additive calculates the inherent strain values required to reach the input maximum z-height. The calculated inherent strain values were then validated by simulating the distortion of a cantilever beam manufactured at an angle of 45° relative to the x-axes. The simulated distortion

was compared to three more samples manufactured at a 45° angle relative to the x-axes and cut at 2.5 mm. After verification of the mechanical calibration, the mechanical calibration was modified to change the simulation configuration to thermal calibration. In the Build Properties window, the peak temperature target was set at 1250°C, as determined in Section 3.3. The material and mesh generation were identical to the mechanical calibration and the resulting EEF value was noted for use in the thermo-mechanical calibration. The thermal calibration process was then modified and the simulation configuration changed to thermo-mechanical calibration. The process parameters were setup as shown in Table 4.5-1 and the EEF value was manually set in the build parameters as determined from the thermal calibration. The calibration results calculated the VEF value to be used in the main simulation.

Table 4.5-1: Mechanical calibration simulation setup procedure.

Step number#	Process	Description
1	Setup simulation main process properties	Simulation configuration: mechanical Type of simulation: calibration Manufacturing process stages: build and cutting
2	Define machine properties	Build space: 250x250x200 mm Base plate: circular Machine parameters: maximum laser power set at 145 W
3	Specify base plate design parameters	Thickness: 13 mm Material: Inconel 718
4	Choose powder material	Material selection: Co-Cr-Mo powder based on EOS Cobalt Chrome MP1
5	Import sample geometry	Sample geometry: stl. file type import Positioning: centre
6	Input cantilever parameters	Add measuring positions: one at the fixed end of the specimen, one in the middle and one at the cut end Set target for maximum distortion by entering the experimental average maximum z-height
7	Input build properties	Layer thickness: 0.025 mm Un-tick rotation parameter tick box Strain type: orthotropic Inherent strains: estimation
8	Define cutting properties	Height: 2.5 mm Width: 12 mm Set starting point at the cut end of the sample Define cutting direction Cut length: 62 mm
9	Generate the mesh	Mesh type: Uniform Levels of coarsening: 0 Voxel size: 0.5x0.5x0.5 mm
10	Run the calibration	Start calibration
11	Save inherent strains	Save the inherent strain results as a template file for use in the thermo-mechanical calibration

4.5.2 Simulations

The simulation process is summarised and outlined in Table 4.5-2. The process parameters were constant and consistent with the calibration process parameters. Each heat treatment strategy was repeated, only varying the annealing holding time at a fixed temperature.

Table 4.5-2: Simulation setup procedure

Step number#	Process	Description
1	Setup simulation main process properties	Simulation configuration: thermo-mechanical Type of simulation: manufacturing Manufacturing process stages: build and heat treat
2	Define machine properties	Build space: 250x250x200 mm Base plate: circular Machine parameters: maximum laser power set at 145 W
3	Specify base plate design parameters	Thickness: 13 mm Material: Inconel 718
4	Choose powder material	Material selection: Co-Cr-Mo powder based on EOS Cobalt Chrome MP1
5	Import sample geometry	Sample geometry: stl File type import Positioning: centre and 4 mm z-offset from base plate
6	Generate support structure	Method: Simufact Support radius: 0.35 mm Support distance: 0.185 mm
7	Input build properties	Heat flux: input EEF and VEF values as determined by calibration Chamber initial temperature: 25°C Base plate initial temperature: 25°C
8	Define heat treatment parameters	Approach: transient heat treatment Time-temperature table: input specific heat treatment strategy
9	Generate the mesh	Mesh type: non-uniform Levels of coarsening: 0 Voxel size: 0.4375 x 0.5 x 0.5 mm
10	Run the analysis	Start analysis Voxel results: export arc. file type
11	Extract results from exported file	Simufact Arc Tool: extract data to txt. file format Excel: copy relevant output values to Microsoft Excel for further processing

After the results were exported and converted to a Microsoft Excel document the values were processed for comparison with the neutron diffraction data. The gauge volume area on the y-z plane is illustrated in Figure 4.5-2 with the horizontal measuring positions indicated on the blue y-line and the vertical measuring positions indicated on the green z-line. The average transverse stress (TY), longitudinal stress (LZ) and normal stress (NX) acting on the gauge volume area, was compared to the Neutron Diffraction data for each measuring position on the y-line and z-line.

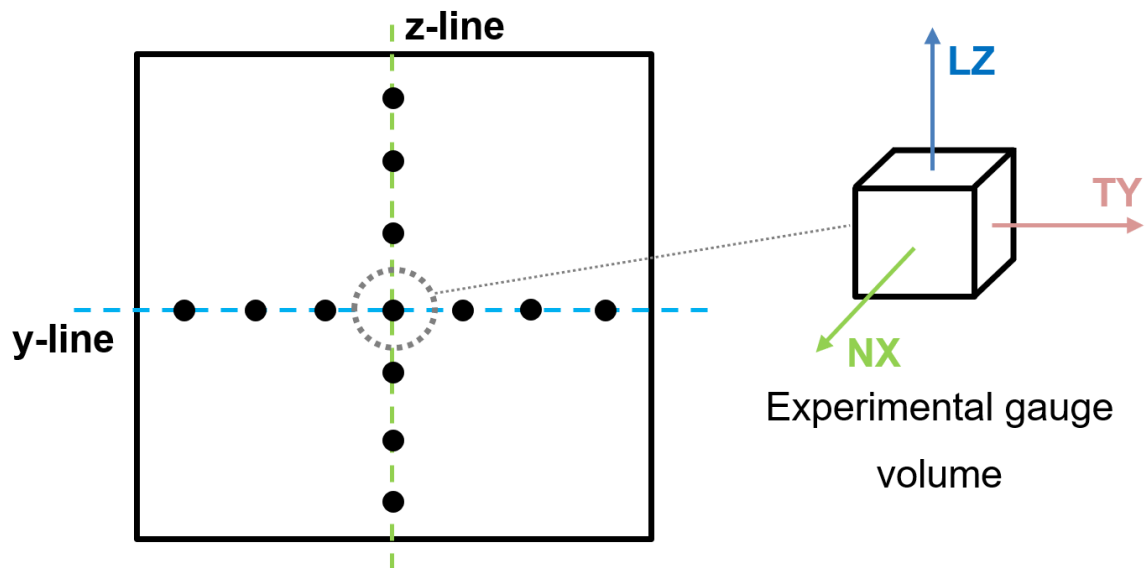


Figure 4.5-2: Simufact Additive data point positioning

4.6 Data analysis

The data analysis was divided into two sections: organising the Simufact Additive simulation results into readable data points and comparing the simulation data with the results obtained from neutron diffraction testing. The simulation data was retrieved from Simufact Additive by extracting the results within an arc-type file. Simufact Additive provides a software tool to extract relevant data within the arc-type file to a text-type file. The relevant nodal positions and residual stress results were then transferred to an Excel spreadsheet, processed and compared to the neutron diffraction data.

The neutron diffraction data was measured within the gauge volume on y-line and z-line, as described in Section 4.4. The simulation data therefore had to be extracted and averaged within the same gauge volume through the y-z plane to be comparable to the neutron diffraction data. The standard deviation for each neutron diffraction measurement was calculated and the data

was then compared to the averaged simulation data at each measuring position for each heat treatment strategy.

CHAPTER 5 RESULTS AND DISCUSSION

5.1 Introduction

This chapter aims to provide insight into the results obtained from the experimental procedures used in this study. The Simufact Additive calibration results will be presented, after which, the residual stress results obtained from the Simufact Additive analysis will be discussed in four sections: normal stress, transverse stress, longitudinal stress, and a performance-based comparison. Within each section the processed residual stress results will be analysed and the residual stress relief after heat treatment will be discussed. The next section will present the residual stress results obtained through neutron diffraction measurements and will be organized in a manner similar to the four previously described sections. The final section of the chapter will provide a comprehensive comparison and discussion of the simulation and neutron diffraction results, encompassing normal, transverse, and longitudinal stress.

5.2 Simufact Additive analysis

5.2.1 Simulation calibration result

The simulation calibration was performed as outlined in Section 4.5.1. The manufactured cantilever beam deflections were measured and the average z-height deflections are presented in Table 5.2-1.

Table 5.2-1: Mechanical calibration deflection results

Measuring Position	Average z-height [mm]	
	0°	90°
A	10.30	11.26
B	10.32	11.23
C	10.38	11.30

The cantilever beam manufactured at an angle of 45° relative to the x-axis was simulated and the deflected results compared to the experimentally measured data in Table 5.2-2.

Table 5.2-2: Mechanical calibration validation

Measuring Position	Average z-height [mm]		
	Experimental	Simulated	% Error
A	10.66	10.65	0.1
B	11.07	10.74	3.1
C	11.50	11.04	4.0

The percentage error between the experimental and simulated deflection for the cantilever manufactured at an angle of 45° relative to the x-axis indicated good correlation and it was therefore determined that the mechanical calibration was successful. The calculated inherent strain values (ε_{xx} , ε_{yy} , ε_{zz}) were used in the thermal calibration to determine the EEF and the thermo-mechanical calibration determined the VEF. The calibration results are presented in Table 5.2-3.

Table 5.2-3: Simufact Additive calibration results

Calibration constant	Value
ε_{xx}	-0.00391917
ε_{yy}	-0.0013574
ε_{zz}	-0.03
EEF	15.4738%
VEF	0.352055

5.2.2 Simufact Additive analysis results

The Simufact Additive analysis resulted in 33493 data points for each sample. The equivalent transverse, longitudinal and normal stresses at each measuring position were extracted from the arc-type file, as well as the nodal position for each stress value. These data points were sorted as described in Section 4.5. An instrument gauge volume of $2.25 \times 1.98 \times 2.00 \text{ mm}^3$ was considered and the data points corresponding with the nodal positions within the gauge volume area of each measuring position averaged.

5.2.2.1 Normal stress results

The processed results for normal stress on the y-line are shown in Table 5.2-4 and Figure 5.2-1.

Table 5.2-4: Processed Simufact Additive analysis results for normal stress on the y-line.

Data point position [mm]			Normal stress [MPa]					
x	y	z	CT	HT1	HT2	HT3	HT4	HT5
1.85	2.00	15.00	-19.81	0.26	1.43	1.59	-5.04	-45.36
1.85	6.33	15.00	3.81	0.25	-0.04	-0.09	-1.22	-30.54
1.85	10.67	15.00	3.26	-0.23	-0.08	-0.13	-1.22	-31.19
1.85	15.00	15.00	2.49	0.15	0.09	0.03	-3.19	-40.63
1.85	19.33	15.00	3.25	-0.12	-0.07	-0.12	-3.48	-33.48
1.85	23.67	15.00	3.75	0.42	-0.04	-0.09	-2.89	-25.89
1.85	28.00	15.00	-20.84	0.36	1.43	1.59	-3.54	-32.54

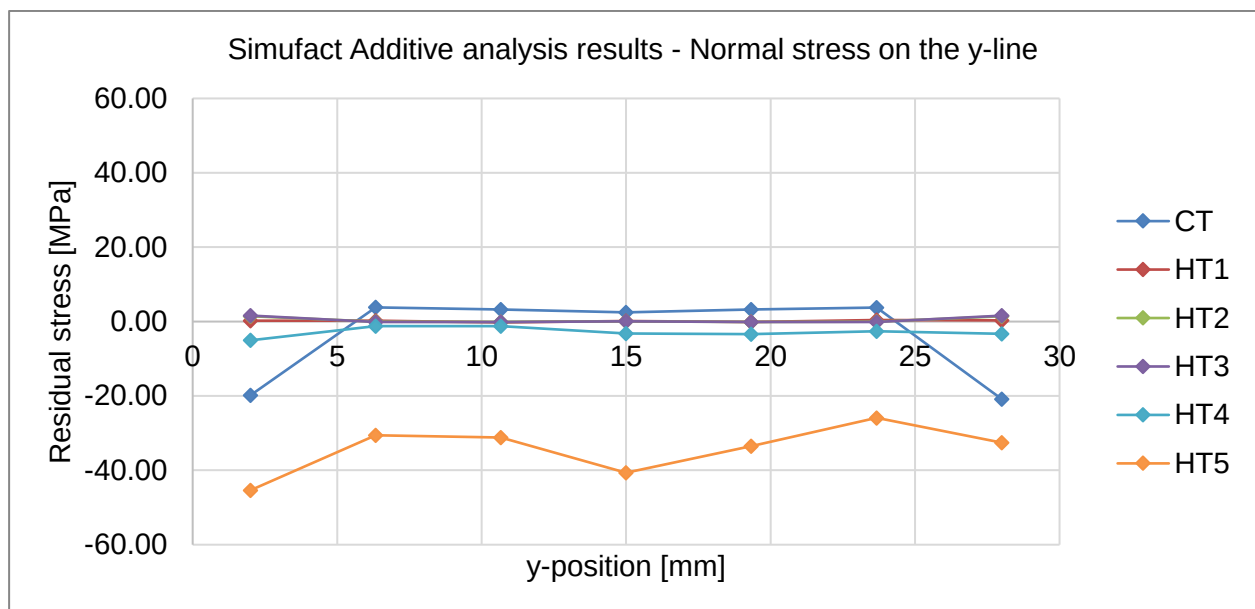


Figure 5.2-1: Graph of the processed Simufact Additive analysis results relating to normal stress on the y-line.

Analysis of the results in Table 5.2-4 indicate that HT1, HT2 and HT3 exhibited similar tensile and compressive normal stress results of no more than 1.59 MPa and 0.23 MPa respectively. HT4 showed slight deviation from the first three heat-treated samples as only compressive normal stress was present with a maximum value of 5.04 MPa. The results for HT5 exhibited larger deviation from the other heat-treated samples and indicated compressive normal stress at all measuring positions. The compressive normal stress was the highest value recorded across all samples with a maximum of 45.36 MPa. CT resulted in the second highest compressive normal

stress measurement of 20.84 MPa and exhibited tensile normal stress at all the measuring positions except for the first and last position with a maximum of 3.81 MPa. The higher residual stress at the data points nearing the edge of the sample was expected as uneven cooling and contraction of the material at the surface induced residual stress.

Using CT results as a baseline for the normal stress before heat treatment at each measuring position the reduction in normal stress at each measuring position was analysed to gauge the effect that each heat treatment strategy had on the normal stress. The analysis indicated that HT1, HT2 and HT3 showed the highest reduction in normal stress over all the heat-treated samples with a maximum reduction in compressive normal stress of 22.43 MPa. The results for HT4 indicate a maximum compressive normal stress reduction of 17.30 MPa. Comparison of the results from CT and HT5 are irregular as all results for HT5 represent compressive normal stress higher than observed in CT. Analysis of the results indicate HT5 contributed to an increase in compressive normal stress by a maximum of 43.12 MPa.

5.2.2.2 Transverse stress results

The unprocessed results from the Simufact Additive analysis of the CT sample for transverse stress is shown in Figure 5.2-2 (a). The overall maximum tensile transverse stress was located at the top of the sample and was equal to 815.76 MPa. The maximum compressive transverse stress was located towards the centre of the sample and was equal to 456.49 MPa. The powder supplier provided a yield stress value of 691.36 MPa for the material, which suggested that the transverse stress in this sample surpassed the yield strength of the material. This could result in permanent deformation or even failure.

The cross-sectional view on the y-z plane is obtained at $x = 1.85 \text{ mm}$ to indicate the plane on which the transverse stress was analysed and to assist in visualizing the stress distribution, as presented in Figure 5.2-2 (b). Analysis of the stress distribution with the use of the colour legend indicated that the overall maximum tensile and compressive residual stress was not located on the y-z plane and therefore no values exceeding the overall maximum stress components could be expected from the processed results. The analysis also indicated that the transverse stress on the y-line resulted in exclusively compressive stress ranging from approximately 29.28 MPa at the outer edges to 231.79 MPa in the centre of the sample.

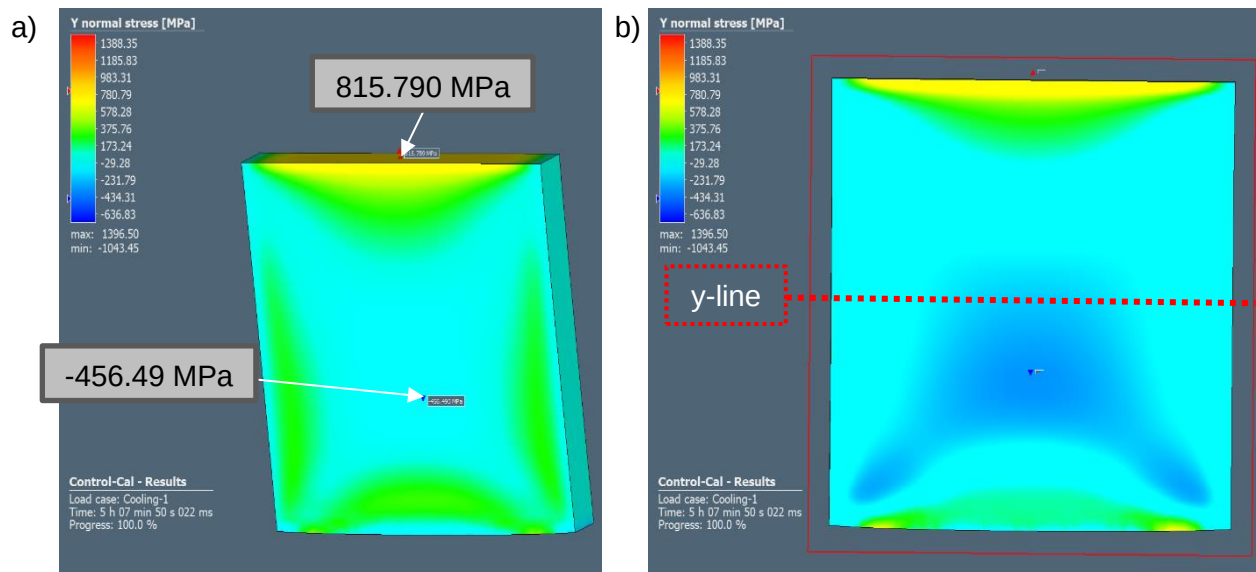


Figure 5.2-2: Unprocessed Simufact Additive analysis results of the CT sample for transverse stress. a) Maximum and minimum stress over the entire sample. b) Cross-sectional view on the y-z plane at $x = 1.85 \text{ mm}$ with the position of the y-line in red.

The processed results for the transverse stress on the y-line are presented in Table 5.2-5 and Figure 5.2-3. The transverse stress is positioned at the centre of the gauge volume area for each measuring position.

Table 5.2-5: Processed Simufact Additive analysis results for transverse stress on the y-line.

Data point position [mm]			Transverse stress [MPa]					
x	y	z	CT	HT1	HT2	HT3	HT4	HT5
1.85	2.00	15.00	-32.84	4.88	5.00	5.08	0.27	-21.56
1.85	6.33	15.00	-111.42	4.29	5.00	5.34	6.44	-64.54
1.85	10.67	15.00	-193.40	1.16	2.09	2.71	4.75	-111.52
1.85	15.00	15.00	-224.00	-0.11	0.94	1.66	3.68	-131.88
1.85	19.33	15.00	-193.36	1.17	2.10	2.74	4.77	-111.20
1.85	23.67	15.00	-107.46	4.31	4.93	5.36	6.46	-63.66
1.85	28.00	15.00	-35.78	4.89	5.01	5.09	0.13	-26.48

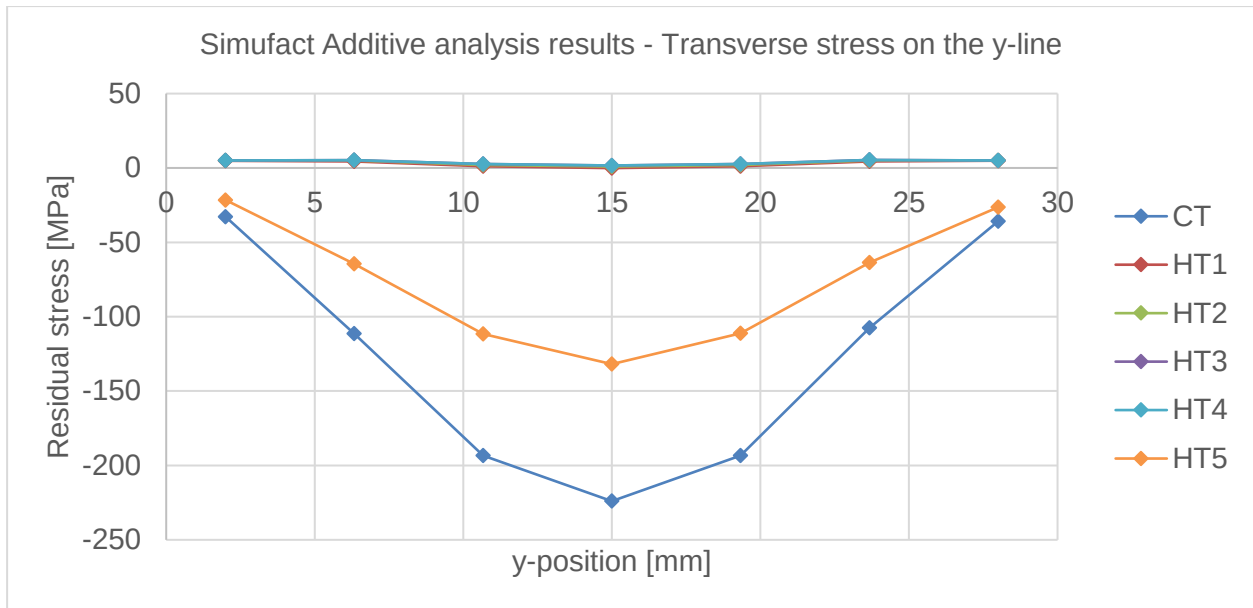


Figure 5.2-3: Graph of the processed Simufact Additive analysis results relating to transverse stress on the y-line.

Analysis of the results in Table 5.2-5 indicate that HT1, HT2, HT3 and HT4 exhibited similar tensile transverse stress results of no more than 6.46 MPa with HT1 presenting with the only compressive transverse stress value of 0.11 MPa. The results for HT5 indicated compressive transverse stress at all measuring positions with a maximum of 131.88 MPa. The processed results for CT exhibited stress distribution as expected from Figure 5.2-2 with the lowest compressive transverse stress presented as 32.84 MPa and highest as 224 MPa.

Using CT results as a baseline the transverse residual stress relief was calculated at each measuring position to gauge the effect that each heat treatment strategy had on the transverse stress, presented in Table 5.2-6.

Table 5.2-6: Simufact Additive analysis results for the transverse stress relief on the y-line.

Data point position [mm]	x	1.85	1.85	1.85	1.85	1.85	1.85	1.85	Average stress relief [MPa]
	y	2.00	6.33	10.67	15.00	19.33	23.67	28.00	
	z	15.00	15.00	15.00	15.00	15.00	15.00	15.00	
Stress Relief [MPa]	HT1	37.72	115.71	194.56	223.89	194.53	111.77	40.67	131.26
	HT2	37.84	116.42	195.49	224.94	195.46	112.39	40.79	131.90
	HT3	37.92	116.76	196.11	225.66	196.1	112.82	40.87	132.32
	HT4	33.11	117.86	198.15	227.68	198.13	113.92	35.91	132.11
	HT5	11.28	46.88	81.88	92.12	82.16	43.8	9.3	52.49

Review of Table 5.2-6 indicates that HT3 and HT4 showed the highest reduction in transverse stress over all the heat-treated samples with a maximum reduction in compressive transverse stress of 227.68 MPa. All samples heat treated to 1065°C (HT1 to HT4) showed similar results with a maximum difference in average transverse stress relief of 1.06 MPa. Comparison of the results from CT and HT5 indicated a maximum decrease in compressive transverse stress of 92.12 MPa. The results indicate that HT5 was less effective at reducing the transverse stress than the other heat treatment strategies and the difference in heat treatment holding time did not have a significant influence on the average reduction in transverse residual stress.

5.2.2.3 Longitudinal stress results

The unprocessed results from the Simufact Additive analysis of the CT sample for longitudinal stress is shown in Figure 5.2-4 (a). The overall maximum tensile longitudinal stress was located towards the centre of the sample and was equal to 1171.14 MPa. The maximum compressive longitudinal stress was located on the lower right side of the sample and was equal to 913.57 MPa. The longitudinal stress values were higher than the tangential stress values and also exceeded the yield stress provided by the powder manufacturer. The cross-sectional view on the y-z plane was obtained at $x = 1.85 \text{ mm}$ to indicate the plane on which the longitudinal stress was analysed and to assist in visualizing the stress distribution, as presented in Figure 5.2-4 (b). Analysis of the stress distribution with the use of the colour legend indicated that the maximum tensile and compressive residual stress was not located on the y-z plane and therefore no values exceeding the overall maximum stress components could be expected from the processed results. Due to large overall stresses throughout the sample the colour legend was averaged over a large range and exact stress distribution on the y-z plane was difficult to visualize. The analysis

however indicated that the longitudinal stress on the z-line resulted in compressive stress throughout the sample ranging from approximately 50 MPa at the top edge to 350 MPa at the bottom edge.

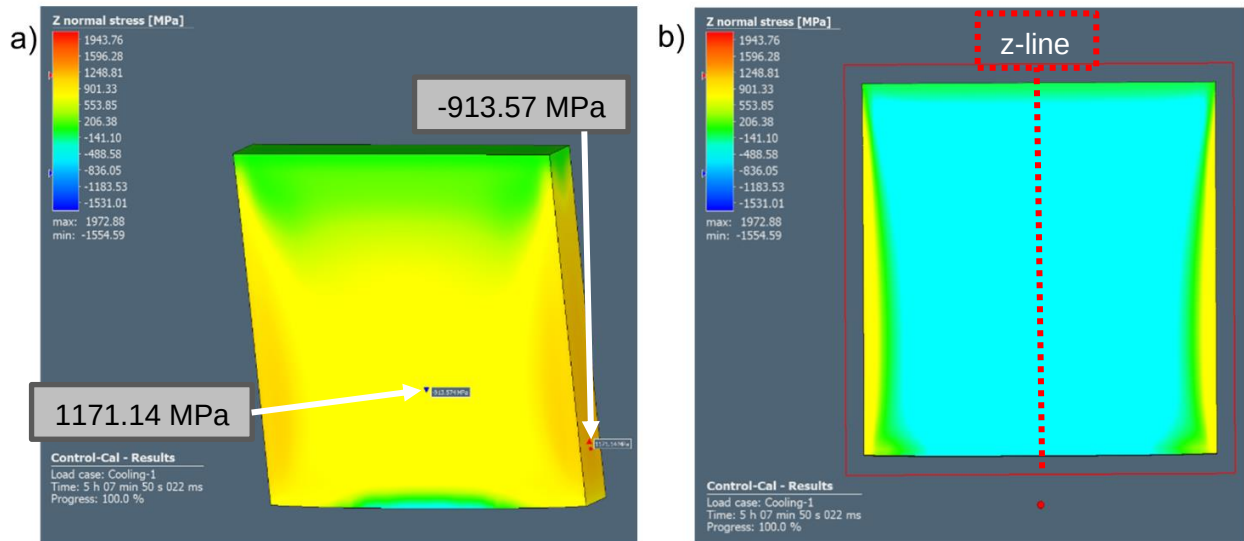


Figure 5.2-4: Unprocessed Simufact Additive analysis results of the CT sample for longitudinal stress. A) Maximum and minimum stress over the entire sample. B) Cross-sectional view on the y-z plane at $x = 1.75 \text{ mm}$ with the position of the z-line in red.

The processed results for the longitudinal stress on the z-line are presented in Table 5.2-7 and Figure 5.2-5. The longitudinal stress is positioned at the centre of the gauge volume area for each measuring position.

Table 5.2-7: Processed Simufact Additive analysis results for longitudinal stress on the z-line

Data point position [mm]			Longitudinal stress [MPa]					
x	y	z	CT	HT1	HT2	HT3	HT4	HT5
1.85	15.00	2.00	-118.32	0.70	5.00	5.08	0.27	-94.66
1.85	15.00	6.33	-227.78	6.73	5.00	5.34	6.44	-188.27
1.85	15.00	10.67	-279.34	4.85	2.09	2.71	4.75	-242.39
1.85	15.00	15.00	-255.18	3.55	0.94	1.66	3.68	-226.35
1.85	15.00	19.33	-177.00	4.87	2.10	2.74	4.77	-159.26
1.85	15.00	23.67	-100.43	6.76	4.93	5.36	6.46	-92.29
1.85	15.00	28.00	-40.59	0.71	5.01	5.09	0.13	-39.52

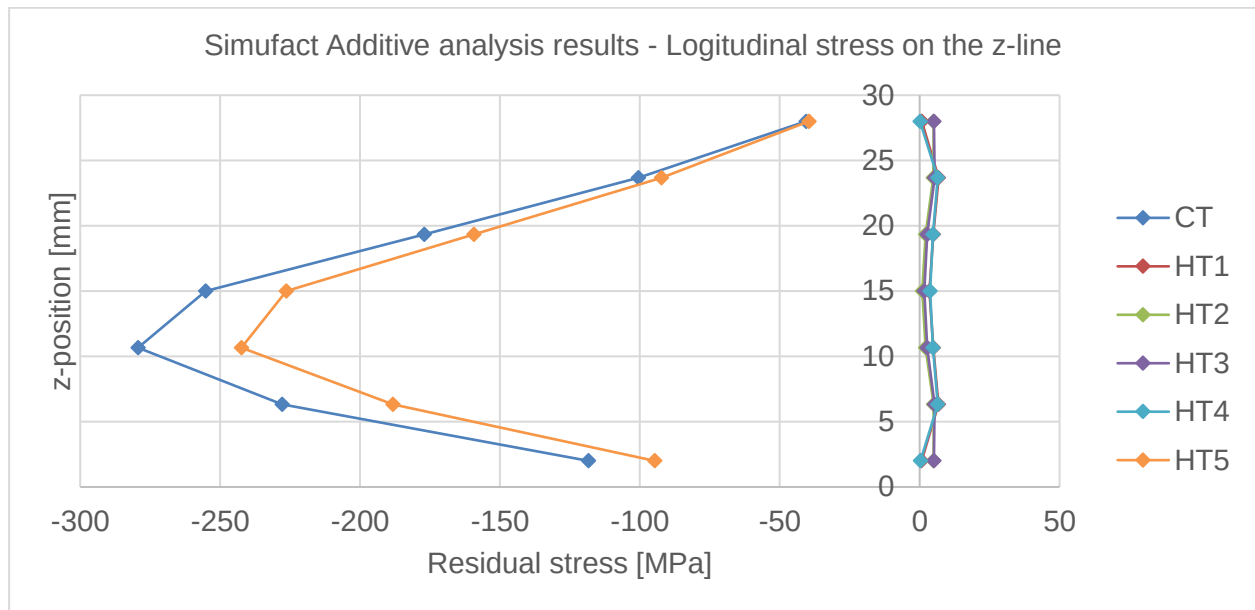


Figure 5.2-5: Graph of the processed Simufact Additive analysis results relating to the longitudinal stress on the z-line.

Analysis of the results in Table 5.2-7 indicate that HT1, HT2, HT3 and HT4 exhibited exclusively tensile longitudinal stresses with similar results of no more than 6.76 MPa. The results for HT5 indicated compressive longitudinal stress at all measuring positions with a maximum of 242.39 MPa. The processed results for CT exhibited stress distribution as expected from Figure 5.2-4 with the lowest compressive longitudinal stress presented as 40.59 MPa and highest as 279.34 MPa.

Using CT results as a baseline the longitudinal residual stress relief was calculated at each measuring position to gauge the effect that each heat treatment strategy had on the longitudinal stress, presented in Table 5.2-8.

Table 5.2-8: Simufact Additive analysis results for the longitudinal stress relief on the z-line.

Data point position [mm]	x	1.85	1.85	1.85	1.85	1.85	1.85	1.85	Average stress relief [MPa]
	y	15.00	15.00	15.00	15.00	15.00	15.00	15.00	
	z	2.00	6.33	10.67	15.00	19.33	23.67	28.00	
Stress Relief [MPa]	HT1	119.02	234.51	284.19	258.73	181.87	107.19	41.3	175.26
	HT2	123.32	232.78	281.43	256.12	179.1	105.36	45.6	174.82
	HT3	123.4	233.12	282.05	256.84	179.74	105.79	45.68	175.23
	HT4	118.59	234.22	284.09	258.86	181.77	106.89	40.72	175.02
	HT5	23.66	39.51	36.95	28.83	17.74	8.14	1.07	22.27

Review of Table 5.2-8 indicated that HT1 and HT4 showed the highest reduction in longitudinal stress over all the heat-treated samples with a maximum reduction in compressive longitudinal stress of 284.19 MPa. All samples heat treated to 1065°C showed similar results with a maximum difference in average transverse stress relief of 0.44 MPa. Comparison of the results from CT and HT5 indicated maximum decrease in compressive longitudinal stress of 39.51 MPa. The results indicated that HT5 was less effective at reducing the longitudinal stress than the other heat treatment strategies and the difference in heat treatment holding time did not have a significant influence on the average reduction in longitudinal residual stress.

5.2.2.4 Simufact Additive analysis results discussion

To easily visualise and compare the transverse and longitudinal residual stress relief results at each measuring position the heat treatment strategies were given a rating of 1 to 5, for which 5 relates to the heat treatment strategy resulting in the largest stress reduction and the results were tabulated in Table 5.2-9. Each column indicates the seven measuring positions from 2 mm to 28 mm.

Table 5.2-9: Performance based rating for the Simufact Additive analysis residual stress relief results.

	Transverse stress							Longitudinal stress							Total
HT1	3	2	2	2	2	2	3	3	5	5	4	5	5	3	46
HT2	4	3	3	3	3	3	4	4	2	2	2	2	2	4	41
HT3	5	4	4	4	4	4	5	5	3	3	3	3	3	5	55
HT4	2	5	5	5	5	5	2	2	4	4	5	4	4	2	54
HT5	1	1	1	1	1	1	1	1	1	1	1	1	1	1	14

The results indicate that all the heat-treated samples have less residual stress compared to the as-built condition. The sample heat treated according to the material specification at 750°C for 1 hour (HT5) resulted in less residual stress relief compared to the other heat treatment strategies. The samples heat treated at 1065°C resulted in the most noticeable residual stress relief. The observed outcome was anticipated since higher heat treatment temperatures added more energy to the system, enabling atoms to move more freely and resulting in greater relaxation of residual stress. Additionally, higher temperatures promoted greater diffusion of material within the sample, which may contribute to the redistribution and relaxation of localized strains.

The heat treatment holding time had very little varying influence when comparing these samples. This indicated that most of the residual stress relief occurred within the first 15 min of heat treatment. The performance-based rating from Table 5.2-9 indicated that HT3 resulted in the most residual stress relief with an overall rating of 55, closely followed by HT4 with an overall rating of 54. It is also clear that HT5 was the worst performing heat treatment strategy as it was outperformed at every measuring position which resulted in the lowest possible rating of 14.

Analysis of the simulation data showed that the increase in heat treatment temperature resulted in the most noticeable residual stress relief and was therefore the deciding factor when the heat treatment was only aimed at residual stress relief. Considering residual stress as the only deciding variable, none of the samples that were heat-treated at 1065°C benefitted from the longer heat treatment holding time.

5.3 Neutron diffraction analysis

The neutron diffraction measurements made use of averaged Bragg's peak values and therefore the standard deviation was calculated and is included in the results.

5.3.1 Neutron diffraction results

5.3.1.1 Normal stress results

The normal stress was assumed to be 0 as part of the plane stress state assumption (Section 3.4). The standard deviation for normal stress is shown in Table 5.3-1.

Table 5.3-1: Standard deviation for the normal stress on the y-line

Data point position [mm]			Standard deviation [MPa]					
x	y	z	CT	HT1	HT2	HT3	HT4	HT5
1.85	2.00	15.00	40.69	41.01	61.72	115.87	48.55	45.81
1.85	6.33	15.00	43.27	41.04	45.60	68.14	48.48	49.02
1.85	10.67	15.00	40.33	40.57	44.24	54.62	48.50	47.55
1.85	15.00	15.00	40.65	44.34	44.69	70.69	48.66	46.86
1.85	19.33	15.00	41.68	42.66	45.65	47.51	48.63	45.64
1.85	23.67	15.00	39.43	38.81	48.56	48.16	48.43	47.50
1.85	28.00	15.00	40.16	39.50	48.43	48.19	48.38	45.99

The standard deviation data only served as a measure of error that could be expected for future measurements and could not be discussed separate from any comparative data sets.

5.3.1.2 Transverse stress results

The neutron diffraction results relating to transverse stress on the y-line are presented in Table 5.3-2 and Figure 5.3-1. The transverse stress is shown at the centre of the gauge volume area for each measuring position and the error bars indicate the standard deviation calculated for each measurement.

Table 5.3-2: Neutron diffraction results for the transverse stress on the y-line.

Data point position [mm]	x	1.85	1.85	1.85	1.85	1.85	1.85	1.85
	y	2.00	6.33	10.67	15.00	19.33	23.67	28.00
	z	15.00	15.00	15.00	15.00	15.00	15.00	15.00
Transverse stress [MPa]	CT	-41.33	-152.64	-174.94	-217.26	-174.29	-125.33	-55.70
	SD - CT	54.64	43.77	42.23	45.72	42.62	41.64	44.18
	HT1	-13.71	-49.10	-48.14	-34.67	-24.30	-38.00	-34.94
	SD - HT1	41.00	41.08	40.80	43.87	42.75	39.41	39.67
	HT2	4.33	-37.13	-20.56	-59.89	-46.63	-17.88	-0.51
	SD - HT2	61.32	45.37	44.14	45.27	45.23	49.10	46.73
	HT3	-14.15	-96.41	-52.69	-74.37	-33.30	-32.18	-33.13
	SD - HT3	134.64	66.92	54.23	69.57	47.82	48.13	47.17
	HT4	-8.92	-33.88	-42.45	-94.39	-63.25	-44.46	-53.56
	SD - HT4	48.53	48.51	48.48	48.68	48.65	48.45	48.45
	HT5	-25.63	-109.32	-128.04	-126.12	-89.16	-104.21	-65.74
	SD - HT5	45.77	48.96	47.53	46.94	45.66	47.94	46.00

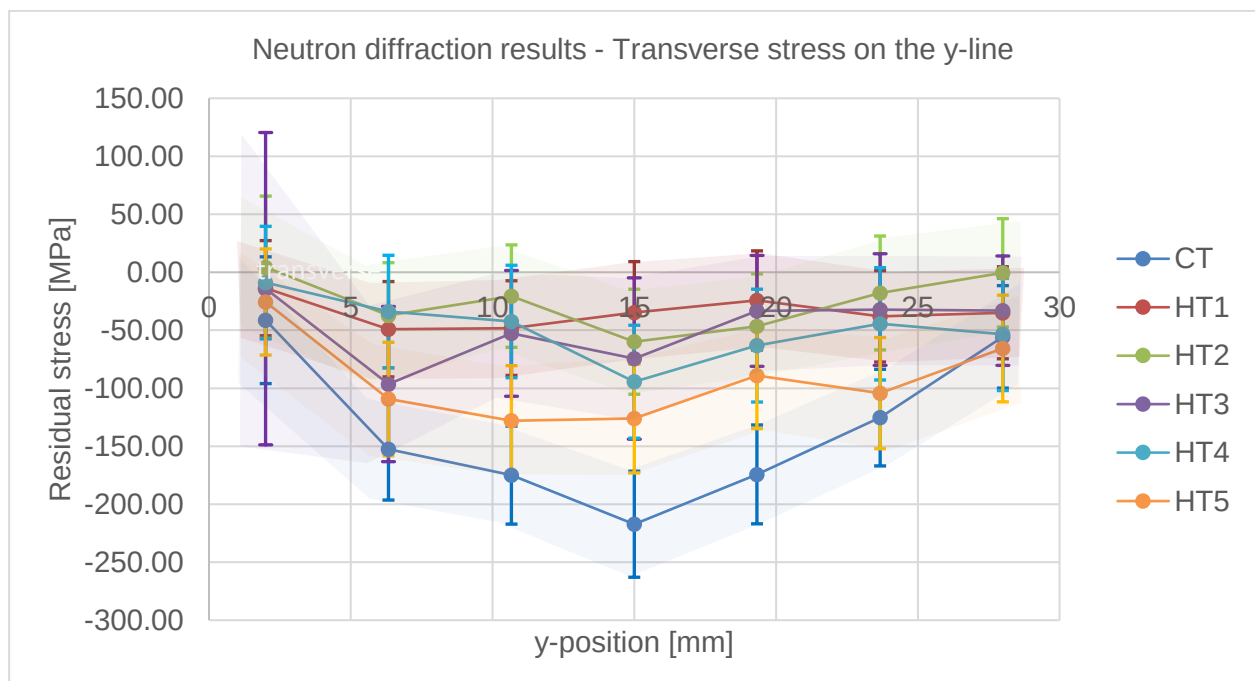


Figure 5.3-1: Graph of the neutron diffraction results for transverse stress on the y-line.

Analysis of the results in Table 5.3-2 and Figure 5.3-1 indicate that HT1 and HT2 exhibited similar tensile and compressive transverse stress results of no more than 4.33 ± 62.32 MPa and 59.89 ± 45.27 MPa respectively. HT3 and HT4 showed mostly similar results to HT1 and HT2 except for one outlier compressive transverse stress value of 96.41 ± 66.92 MPa at $y=6.33$ mm and 94.39 ± 48.68 MPa at $y=15.00$ mm, respectively. If the standard deviation of HT1, HT2, HT3 and HT4 is considered the results are considered as similar as all of the results fall within the same standard deviation range. The results for HT5 indicated compressive transverse stress at all measuring positions with a maximum of 128.04 ± 47.53 MPa. The processed results for CT also indicated compressive transverse stress at all measuring positions with a maximum of 217.26 ± 45.72 MPa.

Using CT results as a baseline the transverse residual stress relief was calculated at each measuring position to gauge the effect that each heat treatment strategy had on the transverse stress, presented in Table 5.3-3.

Table 5.3-3: Neutron diffraction results for the transverse stress relief on the y-line.

Data point position [mm]	x	1.85	1.85	1.85	1.85	1.85	1.85	1.85	Average stress relief [MPa]
	y	2.00	6.33	10.67	15.00	19.33	23.67	28.00	
	z	15.00	15.00	15.00	15.00	15.00	15.00	15.00	
Stress relief [MPa]	HT1	27.62	103.54	126.80	182.59	149.99	87.33	20.76	99.80
	SD1	54.64	43.77	42.23	45.72	42.62	41.64	44.18	44.97
	HT2	45.66	115.51	154.38	157.37	127.66	107.45	55.19	109.03
	SD2	41.00	41.08	40.8	43.87	42.75	39.41	39.67	41.23
	HT3	27.18	56.23	122.25	142.89	140.99	93.15	22.57	86.47
	SD3	61.32	45.37	44.14	45.27	45.23	49.1	46.73	48.17
	HT4	32.41	118.76	132.49	122.87	111.04	80.87	2.14	85.80
	SD4	134.64	66.92	54.23	69.57	47.82	48.13	47.17	66.93
	HT5	15.70	43.32	46.90	91.14	85.13	21.12	10.04	44.76
	SD5	48.53	48.51	48.48	48.68	48.65	48.45	48.45	48.54

Review of Table 5.3-3 indicated that HT1 and HT2 resulted in the highest reduction in transverse stress over all the heat-treated samples with a maximum reduction in compressive transverse stress of 182.59 MPa. HT3 and HT4 showed similar results with a difference of 23.23 MPa in average stress relief from HT2. Comparison of the results from CT and HT5 indicated a maximum decrease in compressive transverse stress of 91.14 MPa. The results indicated that HT2 was the

most effective heat treatment strategy for relieving residual stress, closely followed by HT1. HT5 was the least effective at reducing the transverse stress. Despite variations in the heat treatment time, all samples that were heat treated to 1065°C exhibited a close correlation with the lowest average standard deviation of 41.23 MPa. Furthermore, the analysis revealed that the heat treatment time did not have a significant impact on the average transverse stress relief.

5.3.1.3 Longitudinal stress results

The neutron diffraction results for longitudinal stress on the z-line are presented in Table 5.3-4 and Figure 5.3-2. The longitudinal stress is shown at the centre of the gauge volume area for each measuring position and the error bars indicate the standard deviation calculated for each measurement.

Table 5.3-4: Neutron diffraction results for the longitudinal stress on the z-line.

Data point position [mm]	x	1.85	1.85	1.85	1.85	1.85	1.85	1.85
	y	15.00	15.00	15.00	15.00	15.00	15.00	15.00
	z	2.00	6.33	10.67	15.00	19.33	23.67	28.00
Longitudinal stress [MPa]	CT	228.99	-208.04	-286.47	-214.26	-191.56	-99.51	130.71
	SD - CT	41.14	44.26	41.75	42.56	43.60	40.50	40.70
	HT1	-72.72	-37.37	-28.22	-30.07	-58.51	-36.55	-37.58
	SD - HT1	65.83	41.45	41.31	40.56	40.73	39.98	42.54
	HT2	-11.21	-5.99	-44.85	-37.58	-48.92	-64.63	-69.03
	SD - HT2	47.14	45.06	47.05	45.16	46.19	46.51	55.13
	HT3	-52.03	-79.20	-80.74	-87.85	-67.58	-76.02	-35.99
	SD - HT3	47.40	47.54	53.00	66.36	49.14	46.00	46.49
	HT4	-55.88	-55.01	-75.44	-39.56	-39.32	-67.38	-133.76
	SD - HT4	48.40	48.56	48.40	48.71	48.48	50.97	62.68
	HT5	-43.62	-109.04	-82.48	-145.80	-138.00	-128.91	-35.38
	SD - HT5	46.52	48.67	47.35	47.24	46.35	46.55	55.25

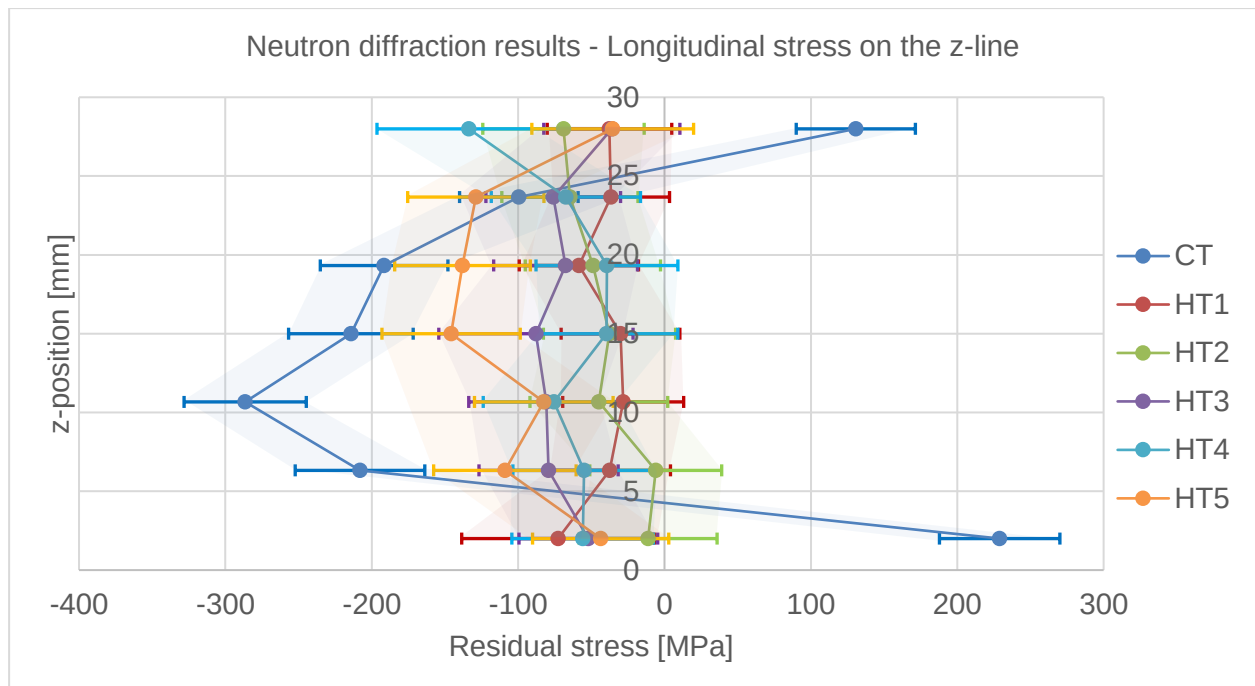


Figure 5.3-2: Graph of the neutron diffraction results for longitudinal stress on the z-line.

Analysis of the results in Table 5.3-4 and Figure 5.3-2 indicate that HT1, HT2 and HT3 exhibited only compressive longitudinal stress results of no more than 72.72 ± 65.83 MPa, 69.03 ± 55.13 MPa and 87.85 ± 66.36 MPa respectively. HT4 also showed similar results to HT1, HT2 and HT3 except for one outlier compressive longitudinal stress value of 133.76 ± 62.68 MPa at $z=28$ mm. If the standard deviation of HT1, HT2, HT3 and HT4 is considered the results are considered as similar as all of the results fall within the same standard deviation range. The results for HT5 indicated compressive longitudinal stress at all measuring positions with a maximum of 145.80 ± 47.24 MPa. Considering the standard deviation for HT5 all but one of the results lie within the standard deviation range of HT4 indicating correlation between these heat treatment strategies. The processed results for CT indicated tensile and compressive longitudinal stress with a maximum of 228.99 ± 41.14 MPa and 286.47 ± 41.75 MPa respectively.

Using CT results as a baseline the longitudinal residual stress relief at each measuring position was calculated to gauge the effect that each heat treatment strategy had on the longitudinal stress, as presented in Table 5.3-5.

Table 5.3-5: Neutron diffraction results for the longitudinal stress relief on the z-line.

Data point position [mm]	x	1.85	1.85	1.85	1.85	1.85	1.85	1.85	Average stress relief [MPa]
	y	15.00	15.00	15.00	15.00	15.00	15.00	15.00	
	z	2.00	6.33	10.67	15.00	19.33	23.67	28.00	
Stress relief [MPa]	HT1	301.71	170.67	258.25	184.19	133.05	62.96	168.29	182.73
	SD1	65.83	41.45	41.31	40.56	40.73	39.98	42.54	44.63
	HT2	240.20	202.05	241.62	176.68	142.64	34.88	199.74	176.83
	SD2	47.14	45.06	47.05	45.16	46.19	46.51	55.13	47.46
	HT3	281.02	128.84	205.73	126.41	123.98	23.49	166.70	150.88
	SD3	47.40	47.54	53.00	66.36	49.14	46.00	46.49	50.85
	HT4	284.87	153.03	211.03	174.70	152.24	32.13	264.47	181.78
	SD4	48.40	48.56	48.40	48.71	48.48	50.97	62.68	50.89
	HT5	272.61	99.00	203.99	68.46	53.56	29.40	166.09	127.59
	SD5	46.52	48.67	47.35	47.24	46.35	46.55	55.25	48.28

Review of Table 5.3-5 indicated that HT1 and HT4 resulted in the highest reduction in longitudinal stress over all the heat-treated samples with a maximum reduction of 301.71 MPa. HT2 and HT3 showed similar results with a maximum difference in average residual stress relief of 31.85 MPa from HT1. Comparison of the results from CT and HT5 indicate the lowest average stress relief of 127.59 MPa. The results indicate that HT1 and HT4 were the most effective heat treatment strategies for relieving longitudinal residual stress and HT5 was the least effective. Despite variations in the heat treatment time, all samples that were heat treated to 1065°C exhibited a close correlation with the lowest average standard deviation of 44.63 MPa. Furthermore, the analysis revealed that the heat treatment time did not have a significant impact on the average transverse stress relief.

5.3.1.4 Neutron diffraction results discussion

To easily visualise and compare the transverse and longitudinal residual stress relief results for each measuring position the heat treatment strategies were given a rating of 1 to 5, for which 5

relates to the heat treatment strategy resulting in the largest stress reduction and the results were tabulated in Table 5.3-6. Each column indicates the seven measuring positions from 2 mm to 28 mm.

Table 5.3-6: Performance based rating for the neutron diffraction residual stress relief results.

	Transverse stress							Longitudinal stress							Total
HT1	3	3	3	5	5	3	3	5	4	5	5	3	5	3	55
HT2	5	4	5	4	3	5	5	1	5	4	4	4	4	4	57
HT3	2	2	2	3	4	4	4	3	2	2	2	2	1	2	35
HT4	4	5	4	2	2	2	1	4	3	3	3	5	3	5	46
HT5	1	1	1	1	1	1	2	2	1	1	1	1	2	1	17

The performance-based rating from Table 5.3-6 indicated that HT2 resulted in the most residual stress relief with an overall rating of 57, closely followed by HT1 with an overall rating of 55. It is also clear that HT5 was the worst performing heat treatment strategy as it was outperformed at most measuring positions which resulted in the lowest overall rating of 17. The results indicate that all the heat-treated samples have less residual stress compared to the as-built condition. The samples heat-treated at 1065°C show the most residual stress relief and the heat treatment holding time had very little varying influence when comparing these samples.

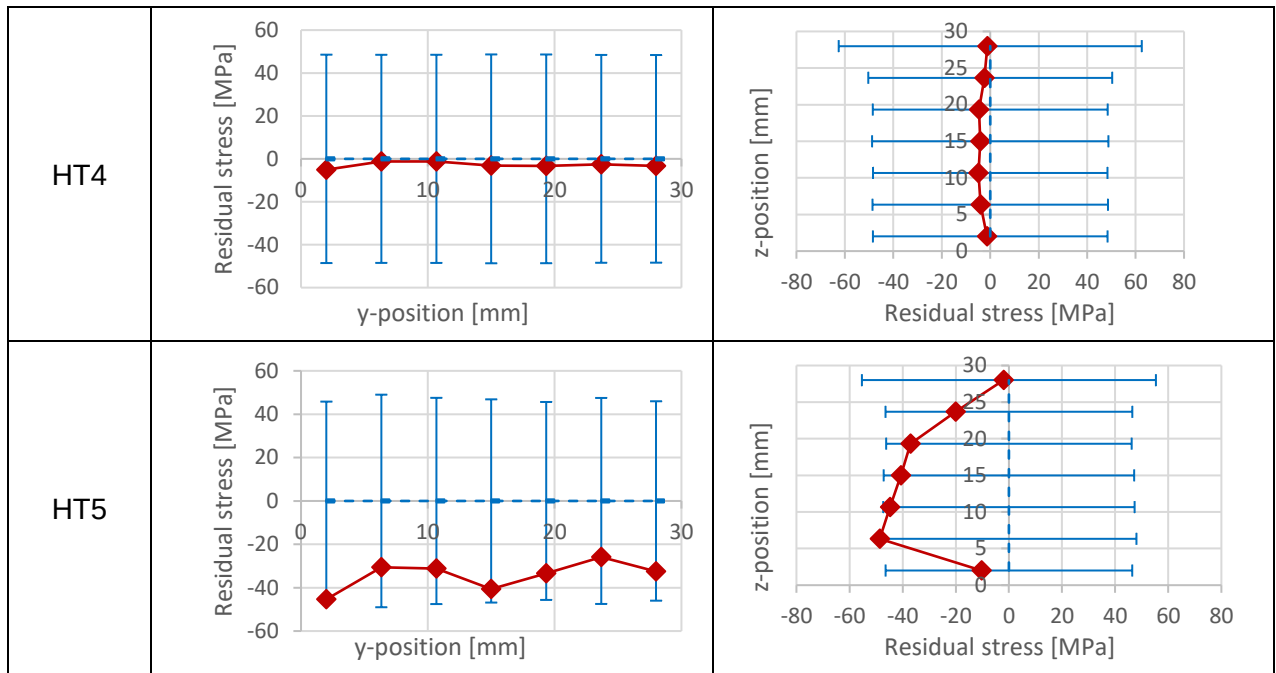
The data from Figure 5.3-1 and Figure 5.3-2 indicated uneven residual stress relief throughout the length and height of the samples. The residual stress calculations from neutron diffraction data relied on Youngs' modulus, which is not constant due to the anisotropic behaviour of AM components (Chapter 2.7). The uneven residual stress relief may therefore have been due to the variations prevalent in a combination of constants that are used during the calculation process and to the anisotropic properties of the material.

5.4 Simulation vs. neutron diffraction results

The simulation and neutron diffraction results for normal stress were first analysed and compared to validate the plane stress state ($\sigma_{xx} = 0$) assumption that was made in Section 3.4. The comparison between the simulation and neutron diffraction data is shown in Table 5.4-1.

Table 5.4-1: Comparison between simulation and neutron diffraction results for normal stress on the y-line and z-line

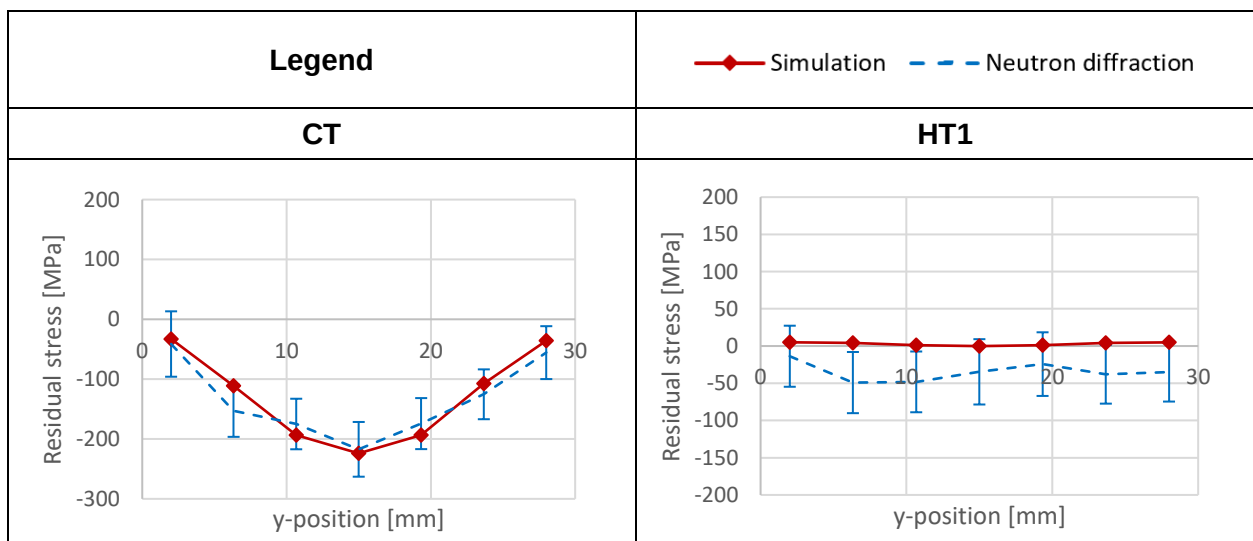
	y-line	z-line
Legend	—◆— Simulation --- Neutron diffraction	
CT		
HT1		
HT2		
HT3		

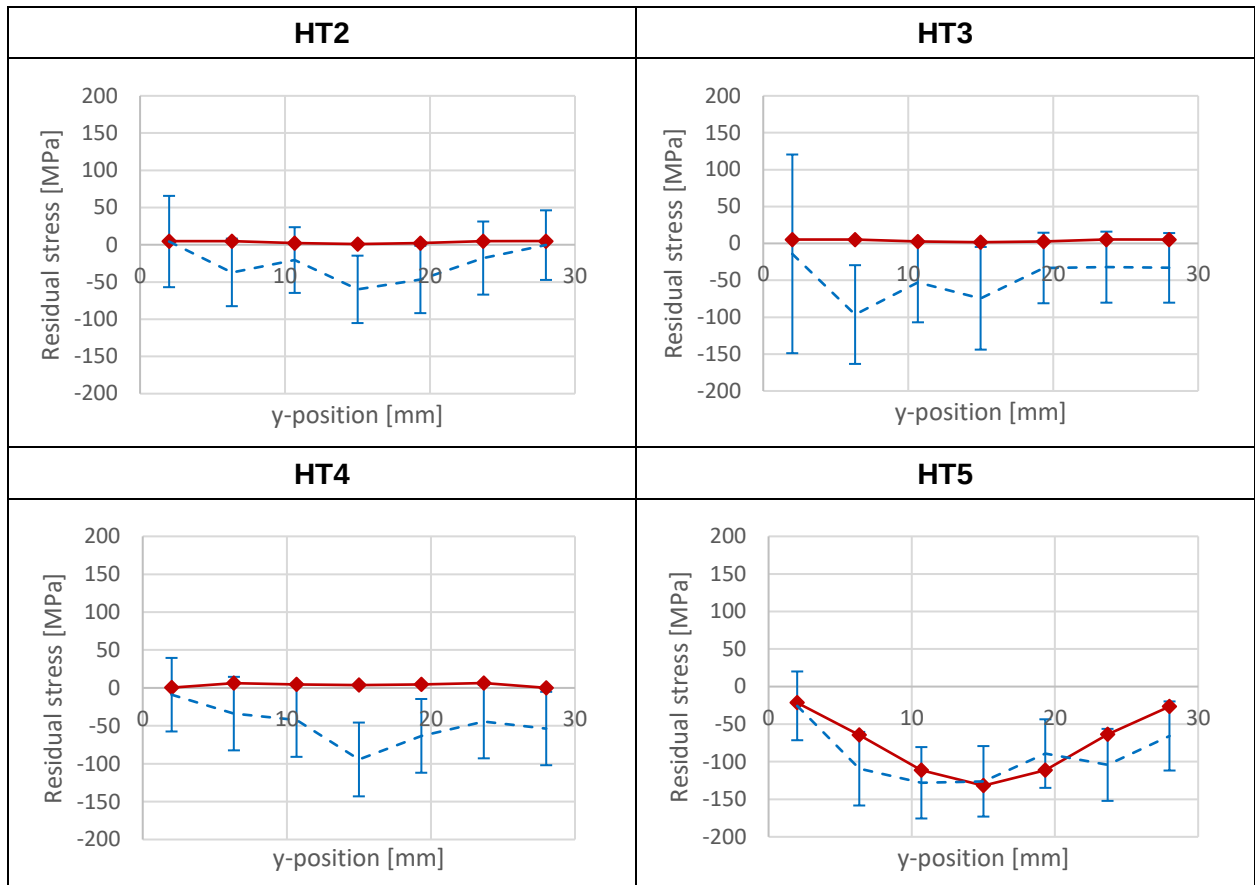


Analysis of Table 5.4-1 indicated that the simulation data points were all within the neutron diffraction data standard deviation range. The residual stress was considered to be negligible and the thin-walled assumption made in Section 3.4 was therefore upheld. A plane stress state ($\sigma_{xx} = 0$) was assumed and d_{hkl}^0 calculated for each measuring position.

The simulation and neutron diffraction data for the transverse stress on the y-line for each heat treatment strategy was individually compared in Table 5.4-2. The transverse stress is shown at the centre of the gauge volume area for each measuring position.

Table 5.4-2: Comparison between simulation and neutron diffraction results for the transverse stress on the y-line.





The difference between the simulation and neutron diffraction results was calculated and the standard deviation for neutron diffraction data was considered, shown in Table 5.4-3.

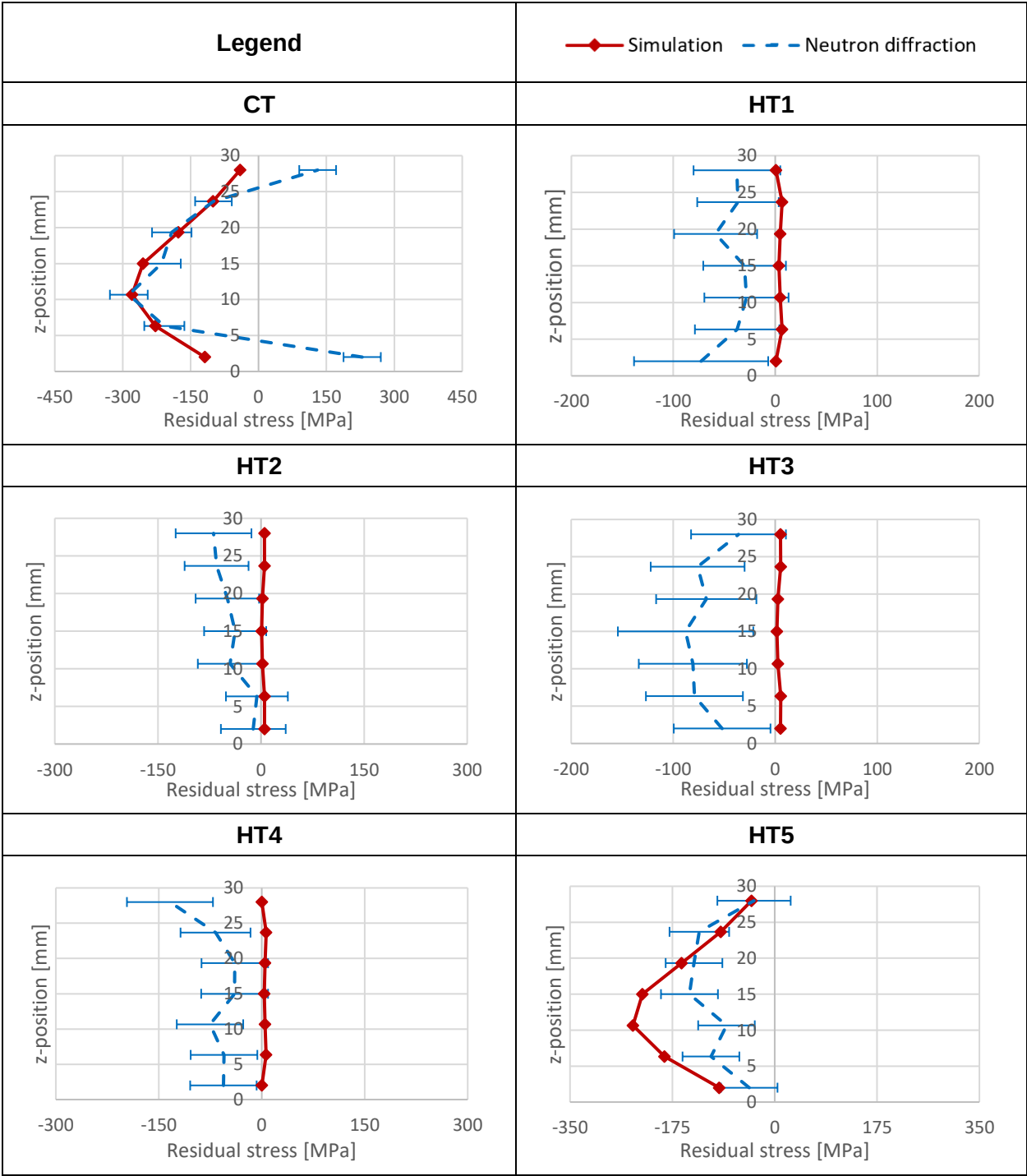
Table 5.4-3: Difference between simulation and neutron diffraction results for the transverse stress on the y-line.

Data point position [mm]	x	1.85	1.85	1.85	1.85	1.85	1.85	1.85	Average Difference [MPa]
	y	2.00	6.33	10.67	15.00	19.33	23.67	28.00	
	z	15.00	15.00	15.00	15.00	15.00	15.00	15.00	
Difference in residual stress [MPa]	CT	8.49	41.22	18.46	6.74	19.07	17.87	19.92	18.82
	SD CT	54.64	43.77	42.23	45.72	42.62	41.64	44.18	44.97
	HT1	18.59	53.39	49.3	34.56	25.47	42.31	39.83	37.64
	SD1	41	41.08	40.8	43.87	42.75	39.41	39.67	41.23
	HT2	0.67	42.13	22.65	60.83	48.73	22.81	5.52	29.05
	SD2	61.32	45.37	44.14	45.27	45.23	49.1	46.73	48.17
	HT3	19.23	101.75	55.4	76.03	36.04	37.54	38.22	52.03
	SD3	134.64	66.92	54.23	69.57	47.82	48.13	47.17	66.93
	HT4	9.19	40.32	47.2	98.07	68.02	50.92	53.69	52.49
	SD4	48.53	48.51	48.48	48.68	48.65	48.45	48.45	48.54
	HT5	4.07	44.78	16.52	5.76	22.04	40.55	39.26	24.71
	SD5	45.77	48.96	47.53	46.94	45.66	47.94	46	46.97

Comparing individual results at each measuring position the CT and HT5 showed the best correlation between the simulation and neutron diffraction results with no values lying outside of the standard deviation range. Comparing the average difference in Table 5.4-3, HT4 showed the worst correlation between the simulation and neutron diffraction results with an average difference of 52.49 MPa which falls 3.95 MPa outside of the standard deviation.

The simulation and neutron diffraction data for the longitudinal stress on the z-line was also individually compared for each heat treatment strategy, as shown in Table 5.4-4. The longitudinal stress is shown at the centre of the gauge volume area for each measuring position.

Table 5.4-4: Comparison between simulation and neutron diffraction results for longitudinal stress on the z-line.



The difference between the simulation and neutron diffraction results was calculated and the standard deviation for neutron diffraction was considered, shown in Table 5.4-5.

Table 5.4-5: Difference between simulation and neutron diffraction results for the longitudinal stress on the z-line.

Data point position [mm]	x	1.85	1.85	1.85	1.85	1.85	1.85	1.85	Average Difference [MPa]
	y	15.00	15.00	15.00	15.00	15.00	15.00	15.00	
	z	2.00	6.33	10.67	15.00	19.33	23.67	28.00	
Difference in residual stress [MPa]	CT	347.31	19.74	7.13	40.92	14.56	0.92	171.3	85.98
	SD CT	41.14	44.26	41.75	42.56	43.6	40.5	40.7	42.07
	HT1	73.42	44.1	33.07	33.62	63.38	43.31	38.29	47.03
	SD1	65.83	41.45	41.31	40.56	40.73	39.98	42.54	44.63
	HT2	16.21	10.99	46.94	38.52	51.02	69.56	74.04	43.90
	SD2	47.14	45.06	47.05	45.16	46.19	46.51	55.13	47.46
	HT3	57.11	84.54	83.45	89.51	70.32	81.38	41.08	72.48
	SD3	47.4	47.54	53	66.36	49.14	46	46.49	50.85
	HT4	56.15	61.45	80.19	43.24	44.09	73.84	133.89	70.41
	SD4	48.4	48.56	48.4	48.71	48.48	50.97	62.68	50.89
	HT5	51.04	79.23	159.91	80.55	21.26	36.62	4.14	61.82
	SD5	46.52	48.67	47.35	47.24	46.35	46.55	55.25	48.28

Comparing individual residual stress difference results in Table 5.4-5 at each measuring position, HT2 showed the best correlation between the simulation and neutron diffraction results with three values lying outside of the standard deviation range. Comparing the average difference in Table 5.4-5, HT2 was the only heat treatment strategy lying within the standard deviation range. Across all data sets more residual stress was present in the neutron diffraction data than was anticipated by Simufact Additive software. The simulation data showed smooth curves and even residual stress relief whereas, from the neutron diffraction data, the anisotropy of real samples was evident (Chapter 2.7). Comparison of the residual stress at the vertical measuring positions showed slightly higher error values which may be due to the layer-wise addition of material increasing the thermal gradient in the vertical build direction.

The Simufact Additive analysis showed results for residual stress relief under ideal conditions with some isotropic material properties such as Young's modulus. The simulation did not account for variation in the printing procedure such as powder particle size, powder distribution on the build plate, heat flux variation during heat treatment, daily temperature affecting the heat treatment cooling rate, variation in furnace heating rate and microstructural defects within the material. The heat treatment could also have changed the microstructure at certain positions within the sample which influenced the strain-free lattice space and created a discrepancy in results (Chapter 2.7).

CHAPTER 6 CONCLUSION

6.1 Conclusion

The aim of this study was to determine the effect of the heat treatment hold time at maximum temperature on the stress relief of AM parts. The simulation results (Section 5.2) showed that heat treatment relieved residual stress in AM manufactured components. The heat treatment hold time however had very little effect on the average stress relief. Comparing the average residual stress relief results for sample heat treated to 1065°C, the maximum difference between heat treatments was 1.06 MPa for the simulation results and 25.95 MPa for the neutron diffraction results. This fell within the uncertainty associated with the neutron diffraction data and was determined to be negligible. Considering residual stress as the only deciding variable, none of the samples that were heat-treated at 1065°C benefitted from the longer heat treatment holding time. Comparison of the simulation and neutron diffraction results indicated close correlation between data sets when the standard deviation was considered. It was therefore concluded that the heat treatment holding time had very little influence on the residual stress relief. The findings indicated that residual stress relief occurred in a shorter time frame than anticipated, and that heat treatments lasting longer than 15 minutes did not yield improved results. It may therefore be beneficial to decrease the traditional stress-relief annealing hold times of an hour or multiple hours when only considering residual stress relief.

The heat treatment temperature was a critical factor to consider when residual stress relief was the goal. The samples that were heat treated at 1065°C showed a higher degree of residual stress relief compared to the sample heat treated at 750°C. Of the samples heat treated to 1065°C, the most effective heat treatment strategy was HT3 which resulted in an average transverse stress relief of 132.32 MPa and an average longitudinal stress relief of 175.23 MPa. The least effective heat treatment strategy was HT2 which resulted in an average transverse stress relief of 131.9 MPa and an average longitudinal stress relief of 174.82 MPa. The largest difference in average stress relief between the most and least effective heat treatment strategies was only 0.42 MPa.

The neutron diffraction results (Chapter 5.3) also indicated that the samples heat treated at 1065°C showed a higher degree of residual stress relief compared to the sample heat treated at 750°C. The uneven stress relief seen in the neutron diffraction data was attributed to the anisotropic behaviour of AM materials and the unpredictable nature of the AM process parameters. Of the samples heat treated to 1065°C, the most effective heat treatment strategy was HT2 which resulted in an average transverse stress relief of 109.03 MPa and an average longitudinal stress relief of 176.83 MPa. The least effective heat treatment strategy was HT3

which resulted in an average transverse stress relief of 86.47 MPa and an average longitudinal stress relief of 150.88 MPa.

6.2 Recommendations for further study

Many other variables must be considered when heat treating AM components. Some recommendations for future studies to build on the current research is as below.

- The influence of the heat treatment holding time variations on microstructural changes and mechanical properties of AM components should be studied further. Further research in this field could lead to a critical change in the heat treatment process.
- The influence of shorter heat treatment durations on the residual stress of AM components should be tested to determine the shortest possible heat treatment holding time at 100% residual stress relief.
- The mechanical properties of AM Co-Cr-Mo alloy heat treated to 1065°C for 15 min should be studied to determine the validity of this heat treatment strategy for use in the AM process of RPDs.

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