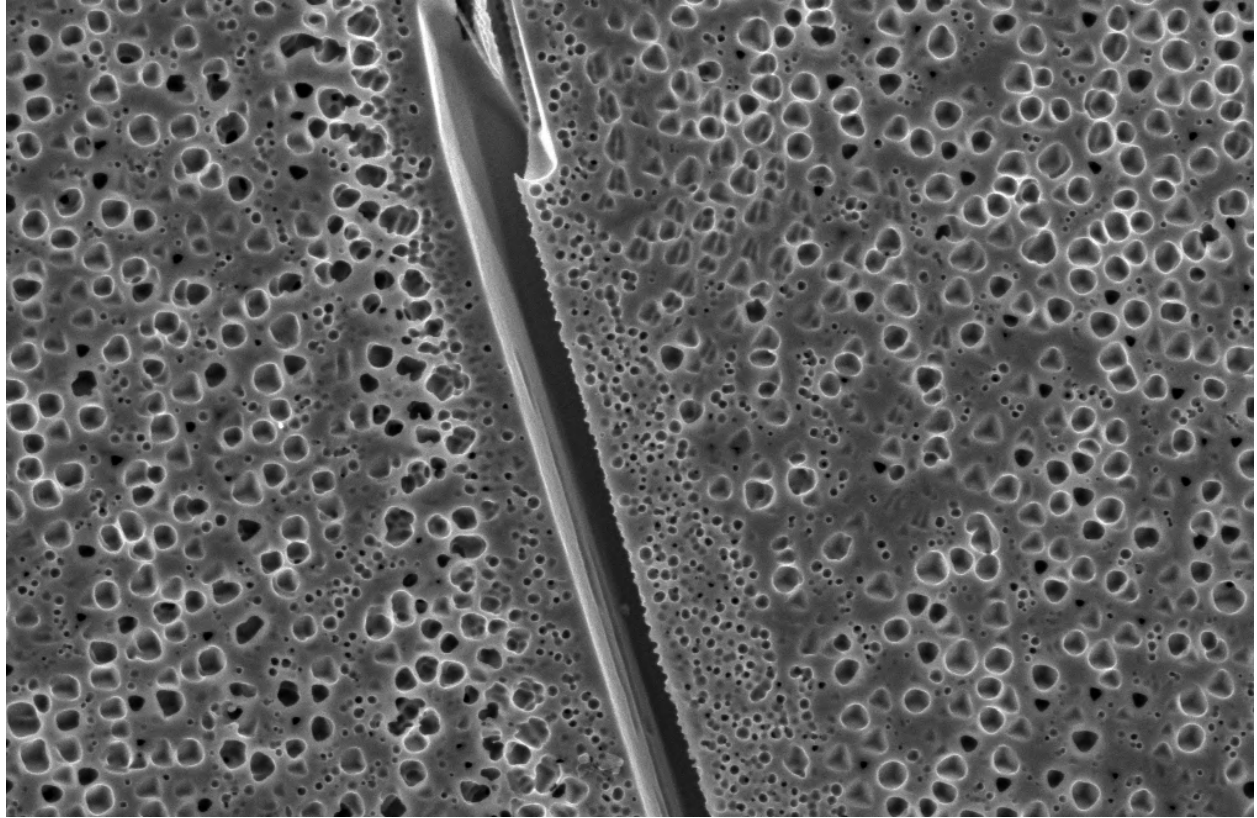




CHALMERS
UNIVERSITY OF TECHNOLOGY



Microstructural Evolution of VDM Alloy 780 under Isothermal Heat Treatment Conditions

In collaboration with GKN Aerospace

Master's thesis in Materials Engineering

ARDA BAYTAROGLU

DEPARTMENT OF INDUSTRIAL AND MATERIALS SCIENCE

CHALMERS UNIVERSITY OF TECHNOLOGY

Gothenburg, Sweden 2022

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Cover: Scanning electron microscopy image of VDM 780 sample after a standard heat treatment is performed, visualizing the bi-modal gamma prime (γ') precipitation and the high temperature phase.

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Abstract

VDM Alloy 780 is a novel, gamma prime (γ') strengthened, wrought Ni-base superalloy that is getting wide attention in the aerospace industry for applications requiring high-temperature stability. The specially tailored composition offers a combination of the properties of Waspaloy and Inconel 718, two well-established Ni-base superalloys, eliminating their drawbacks regarding high-temperature instability and difficult-to-forge properties. The microstructure of VDM 780, like all other alloys, is highly dependent on its thermal history. Previous studies on this alloy show that a heat treatment method that enables the formation of γ' phase and a high-temperature phase to tailor the microstructural properties has already been developed. However, the reasoning behind the heat treatment parameters was not elaborated upon. For this reason, an isothermal heat treatment study was performed under different conditions, and the findings were used to understand the standard heat treatment.

The first objective of this thesis work was to perform isothermal heat treatments and evaluate the microstructural evolution. The heat treatments were performed under 48 different conditions, consisting of 2 initial material states, six isothermal temperatures ranging from (a) °C to (f) °C and four heat treatment durations ranging from 1 to 16 hours. The results of the isothermal heat treatments were based on different studies found in the literature, simulations, and various material characterization methods performed at GKN Aerospace.

The second objective was to understand the necessity of the long duration of the standard heat treatment. Therefore, each step in the standard heat treatment is studied individually in the isothermal heat treatment study. Therefore, the findings of that study are used to understand the standard heat treatment steps. It has been concluded that the isothermal heat treatment conducted at (c) °C for 8 hours is the most efficient one to maximize the hardness. This is related to the size and volume fraction of the "gamma" phase precipitation in the same condition.

Furthermore, each step of the standard heat treatment was confirmed to have a critical role in the precipitation of the high-temperature phase and γ' phase.

Keywords: VDM 780, isothermal heat treatments, gamma prime phase, high temperature phase, microstructural evolution.

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1

Introduction

This master thesis work was conducted at the GKN Aerospace Sweden AB facilities in Trollhättan. Hence, this chapter will briefly introduce the company, give a background to this thesis work and propose the problem statement, as well as the study's scope and constraints and lastly give an outline of the structure of the thesis.

1.1 GKN Aerospace

GKN is a multinational engineering firm established in the south of Wales in 1759, and its foundations can be linked all the way back to the onset of industrialization. GKN's success has been recognized by many countries and companies over the course of 260 years, and they have become a world-class technology company which is actively working in 30 countries with approximately 58,000 employees. Although GKN was initially an ironworks and steel manufacturing company at the birth of the industrial revolution, with the advancements in their understanding of different technologies, they have started to focus on many different areas of engineering. Today, there are three main divisions that GKN has: Aerospace, Powder Metallurgy, and Automotive. [1]

GKN Aerospace is a prominent tier-1 provider of airframe and engine structures, landing gear, electrical interconnection systems, transparency, and aftermarket services for both commercial and military aircraft throughout the world. GKN Aerospace has become one of the world's largest suppliers to the aviation industry, with over 15,000 employees on 38 different manufacturing sites in 12 countries. Aerostructures, engine components, and unique items requested from the customers are among the commodities. The production site at Trollhättan, which is also the main headquarters for the subdivision of Engine Systems, has approximately 2000 employees. Apart from the site in Sweden, GKN Aerospace Engine Systems has production plants in the USA, Norway, and Mexico. Figure 1.1 represents the different product positions that GKN Aerospace has a significant role in. [1]

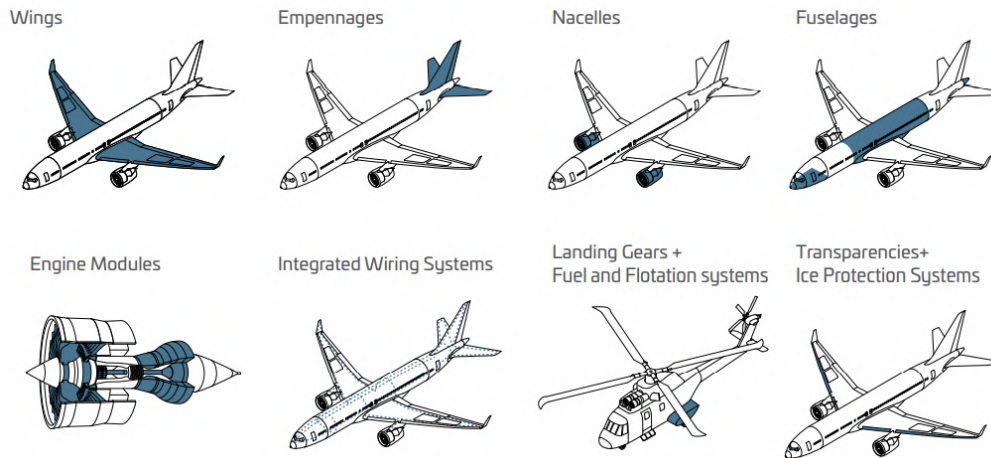


Figure 1.1: GKN Aerospace products. [1]

1.2 Background

The success of aerospace industry has always been dependent on novel developments and technologies that will allow for increased fuel efficiencies. Aerodynamically improved wings, increased use of composite materials and new alloys, and novel fabrication methods all contributed to weight reduction in new aircraft models, which is a direct factor in fuel efficiency. Aside from these crucial structural components, the gas turbine engine (GTE) holds great value to maximize efficiency. Engineers seek to maximize this efficiency by trying to increase the maximum service temperature that GTEs can operate under before reaching the crucial temperatures. Developing new materials becomes a key factor at this point.

One of the most active phases of research is for high-temperature performing materials that can be placed into service in critical sections of engines, which have improved high temperature applications and contribute to increased engine efficiency. In the first few decades of the 20th century, stainless steels were frequently employed in contexts that required them to withstand high temperatures. However, this class of materials was unable to meet the temperature and corrosion requirements imposed on them; as a result, designers were forced to create novel alloy compositions with greater high-temperature capabilities. Because of the widespread use of "superior alloy compositions," a new category of materials known as "super-alloys" for use in environments with high temperatures has been developed.

A superalloy is defined as "material developed for high temperature conditions and put into areas of application where a tendency for severe mechanical loading has been observed and there is a need for high surface integrity" [2]. This class of materials was created to be used at temperatures above 540 °C. Many superalloys have been researched over the years in the lengthy history of superalloy development and application, but only a few have been used in the aerospace sector [3, 5].

Although it has been decades since nickel superalloys have been developed and

adopted by the industry, there is still room for improvements to optimize the high temperature stability of these alloys while maintaining their manufacturability.

1.3 Problem Statement

GKN Aerospace has been constantly looking for new materials that can be beneficial to boost the performance of the GTEs as well as keep the manufacturability and forming process of the components as feasible as possible. Inconel 718 (IN718) and Waspaloy are two of the most established high-temperature superalloys in the aerospace industry. However, each has their own drawbacks. Phase stability is relatively poor in IN 718 at the operating temperatures above about 650 °C due to the transformation of the metastable γ'' phase to the more stable δ -phase which has limited strengthening effect [9]. On the other hand, Waspaloy is stable above 650 °C but the fine grain forging window is extremely tight. This makes the formability of this alloy difficult. Compared to these well-known alloys, the novel nickel-based superalloy VDM Alloy 780 has shown promising results in the previous studies in terms of high temperature stability of its main strengthening phase, gamma prime (γ') phase, and having a wider fine grain forging window than Waspaloy, which makes it possible to achieve similarly fine grains as in IN718 [23]. The thermal background via successful heat treatment of the material holds the key to achieving both of these desired properties. There is a fairly standard heat treatment process developed for this alloy in the literature, however, the selection of different parameters like heat treatment temperature and time is not covered in depth to allow a proper understanding of how the microstructure changes under different conditions. Hence, the intended work for this thesis, is to provide insight into the microstructural evolution of VDM Alloy 780 under different isothermal heat treatment conditions.

1.4 Goal

The first goal was to investigate the microstructure of VDM 780 under various isothermal heat treatment conditions, characterize the main strengthening phase, γ' , and the high temperature phase, and correlate their precipitation kinetics to hardness measurements on the samples. The second objective was to understand the standard long heat treatment cycle for this alloy by using the insight gained from the isothermal heat treatments.

1.5 Limitations

- Different cooling rates from the isothermal heat treatments will not be looked into and only water quenching will be done after each heat treatment since the main objective is to freeze and observe the microstructure developed at that exact isothermal temperature and time.
- Due to the limited time frame of this thesis work, mechanical properties of the heat treated samples such as creep and tensile strengths will not be studied.

- The exact temperature values for the isothermal heat treatments will not be given due to the confidentiality agreements with GKN Aerospace.

1.6 Thesis Structure

This report is divided into 6 chapters; Introduction, Theoretical Background, Methods, Results and Discussion, Conclusion and lastly Future Work.

2

Theoretical Background

This chapter presents the essentials that are required to comprehend the project understanding. A general background of the working condition of a gas turbine engine, followed by the current knowledge of superalloys, VDM Alloy 780 and the isothermal transformations are presented in this chapter.

Typical gas turbine engines work with the same principle, including four main stages. The process commences with air being suctioned by turbo-fans on the engine's rear side. The airflow is then routed to a compressor in the engine's midsection, which is squeezed by rotors and stators, raising the system's temperature. The compressed air is mixed with fuel and fired in the combustion chamber, where its combustion and expansion are later used for thrust. The engine's last portion comprises turbines that rotate using the expanding gases from the combustion chamber. The spin of the turbines is subsequently transmitted to the front of the engine by a shaft, resulting in the rotation of the front turbo-fans, allowing the process to continue.[4]

An important point to consider is the rise in temperature that takes place from the beginning to the end of burning the fuel and passing it through a series of turbines. The engine is split into two distinct categories as a direct consequence of this temperature rise. Because of this, the engines are typically separated into a cold section and a hot section, as seen in 2.1, with each section having its own unique set of needs and, consequently, its own set of materials.

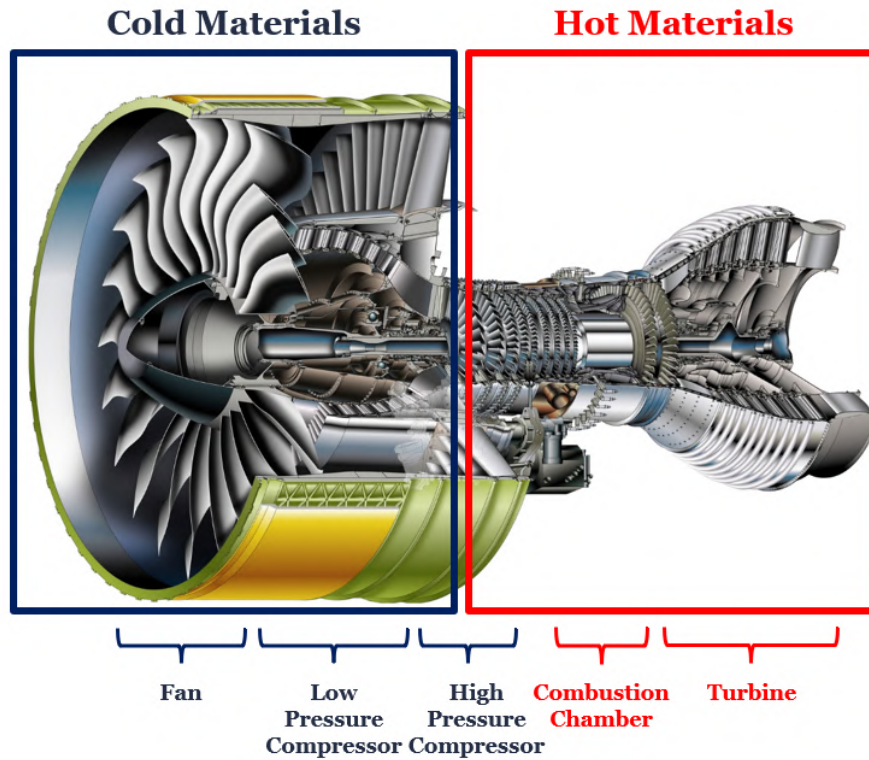


Figure 2.1: Two different sections in terms of temperature and materials in a gas turbine engine.

The impact of the turbine engines' design on the aircraft's overall performance is incomparable to any other structure since it directly affects how the fuel is used[5]. Hence, one of the most critical aspects of the aerospace industry is the ongoing research and development of new turbine engine designs and materials that will enhance the performance of these new engines. As the part of the engine responsible for determining how efficient the whole system is, the service temperature of the hot section in which it operates is a significant consideration during the design process of these engines. As a result of the fact that the service temperature of this section is inversely proportional to the amount of fuel that the engine consumes [6], there is a persistent attempt on the part of the industry to elevate that temperature. When the operating temperature of the turbine engine is raised, the requirements that each component must fulfill to function correctly are rethought. These new requirements concern the mechanical properties of the component, as well as its resistance to corrosion and thermal stability.

Different types of materials, such as superalloys based on nickel, have been created and put to use in the industry as a means of conforming to these standards. These alloys are distinguished by their superior performance at high temperatures as well as their resistance to the debilitating effects that are brought on by operating in such conditions. In addition, they are distinguished by the difficulties that their

manufacturing procedures entail to achieve the desired end-product component design. These alloys must strike a compromise between the desired qualities and the capacity to manufacture the component; as a result, there is little potential for the alloys to advance in their quality.

2.1 High-Temperature Materials and Superalloys

The "high-temperature materials" material group is notable for its capacity to preserve its functionalities even when subjected to elevated temperatures [7]. These materials make an excellent choice for the components in the hot section of the GTEs. To a certain extent, the performance characteristics of these applications are constrained by the operating conditions that the materials employed can withstand.

There are three important characteristics desired from a high-temperature material [7]:

- an ability to withstand loading at an operating temperature close to its melting point, often in excess of 0.7 of the absolute melting temperature, T_m
- a substantial resistance to mechanical degradation over extended periods of time
- tolerance of severe operating environments

Superalloys are an essential group of high-temperature materials utilized in the sections of jet and rocket engines that experience the highest temperatures, reaching temperatures between 1200 and 1400 degrees Celsius [8]. Because of their high thermo-mechanical fatigue resistance, high creep strength, and high hot corrosion and erosion resistance, this group of materials has emerged as the materials of choice for high-temperature applications.

Based on the primary alloying element in the matrix or on their fundamental strengthening mechanisms, superalloys can be divided into three central systems: iron-nickel, nickel, and cobalt base alloys which are discussed in the following Sections 2.1.1 to 2.1.3 [9, 10].

2.1.1 Iron-Nickel-base Superalloy

Iron-nickel-base superalloys are types of superalloys that contain 25-60% Ni and 15-60% Fe [11]. These alloys are also classified as precipitation and solid-solution strengthened alloys. Strengthening and increasing the performance of these superalloys can be achieved by intermetallic phases:

- Precipitation of gamma prime phase (γ') during heat treatment
 - Coherant, ordered $Ni_3(Al, Ti, Nb)$ FCC structure
- Nucleation and coarsening of gamma double prime phase (γ'') on (γ') particles and in the matrix.
 - Coherant but misfitting, ordered metastable $Ni_3(Nb, Al, Ti)$ BCT structure
- Strengthening of grain boundary carbides

Although Fe-Ni-base superalloys provide high creep strength with the possibility of fine grain forging, which makes the forming processes more manageable, the application temperatures are limited. In the case of IN718 under the extended duration in temperatures beyond 650 °C, the metastable γ'' phase transforms into the more stable δ phase. This phase is an incoherent, orthorhombic nickel and niobium-rich phase that precipitates as a needle-like structure. The transformations between γ'' and δ phase lead the main strengthening phase to become brittle; hence it causes a significant decrease in the creep strength. [12]

2.1.2 Cobalt-base Superalloys

Cobalt-base superalloys are metals whose significant mass fraction is cobalt, and it is generally alloyed with elements such as chromium, tungsten, nickel, and iron. Typical compositions for this class of superalloy consist of 40-60% Co, 20-30% Cr, around 20% Ni, and the rest can be W, Mo, and C [10].

Compared to other superalloys, cobalt-base alloys are not strengthened by a coherent, ordered precipitate. Instead, they are distinguished by a minor amount of carbides scattered throughout a solid solution strengthened austenitic (FCC) matrix. Below 417 °C, cobalt crystallizes in the HCP form. Higher temperatures cause it to change into FCC. Almost all cobalt-base alloys are alloyed with nickel to stabilize the FCC structure between ambient temperature and the melting point to prevent this transformation throughout the operation. [13]

Cobalt-base superalloys are corrosion, wear, and heat resistant. Compared to Ni-base superalloys [14], they exhibit superior weldability and thermal-fatigue resistance [15]. On the other hand, they are more prone to precipitate undesired plate-like σ and TCP phases than nickel-base alloys, which have a high tolerance for alloying elements in solid solution.

2.1.3 Nickel-base Superalloys

Ni-base superalloys are the most intricate and frequently employed for the components located in the engine's hot section. Currently, they make up more than 50% of the weight of modern aircraft engines. [16]

These superalloys are solution- and precipitation-strengthened alloys that contain various alloy elements. The solid solution mechanism strengthens the alloy by dissolving one element in another, which causes a disturbance within the crystal lattice and hinders the movements of dislocations, decreasing the rate of plastic deformation. In the case of precipitation strengthening mechanism, these complex engineered materials involve the precipitation of intermetallic phases, similar to Fe-Ni-base superalloys. A precipitated phase develops when the soluble atoms move by diffusion to concentrate at locally favorable locations or specific crystallographic planes. When the first precipitate occurs, it is coherent with the matrix, and this coherence leads to internal strains. Coherency strains trap dislocations, hardening

the alloy. [17]

The precipitated phases are gamma prime (γ'), gamma double prime (γ''), carbides such as MC (rich in Ti and Mo), $M_{23}C_6$ (rich in Cr) and other carbides such as M_6C and M_7C_3 . These micro constituent phases are largely responsible for these alloys' excellent strength, strong oxidation, corrosion resistance, and creep qualities. The (γ') phase is coherent with the gamma matrix (γ) and is the main contributor to the strength. At the same time, the carbides can be observed in the grain boundaries and intergranular in these alloys, incoherent to the matrix [7]. While the alloying elements determine the composition and the phases that can form, the properties of the superalloy can be optimized by heat treatment. Table 2.1 shows the contribution of the most alloyed elements in Nickel-base alloys.

Table 2.1: Effect of alloying elements in Nickel superalloys. [6, 18]

Elements	Main Contribution to Microstructure
Ni	Stabilization of γ phase Formation of the hardening precipitates
Cr	Formation of carbides and solution hardening
Co	Contributes to amount of precipitation Raises γ' solvus temperature
Al	Precipitation of γ' phase Suppresses the precipitation of η phase
Ti	Precipitation of γ' phase
Mo-W	Formation of carbides and solution hardening
Nb	Formation of carbides and solution hardening Precipitation of γ'' and δ phases
Ta	Formation of carbides and solution hardening
C	Forms carbides
B-Zr	Suppresses the precipitation of η phase

2.1.3.1 Heat Treatment of Ni-base superalloys

The first step in developing an alloy with the desired properties is to design its elemental composition. According to this elemental composition, specific phases will form and precipitate in the microstructure after the correct heat treatment is performed on the material. It is vital to manage the microstructure through the use of precipitated phases to derive the high-temperature strength and characteristics of the material when it is subjected to conditions of service [18]. Fedorova et al. have studied the invention and development of the alloy system that later took the name of VDM Alloy 780 [22,23,24]; therefore, this study will not evaluate the discussion further around the design of the elemental composition.

The next step is to design a proper heat treatment to tailor the microstructure. The term "heat treatment" refers to the process of heating metal without allowing it to approach its molten, or melting, state and then cooling the metal in a manner that is under controlled conditions in order to select desirable mechanical characteristics.

In Ni-based superalloys, the precipitation of the main strengthening phases and the secondary phases are controlled with this heat treatment. Because they offered the ideal mix of characteristics for both room temperature manufacturing and elevated temperature service, Ni-based superalloys are often provided in solution-treated [9]. On the other hand, a properly designed heat treatment cycle that is specific to the alloy makes it feasible to accomplish:

- Precipitation of strengthening phases
- Precipitation of carbides
- Control of grain growth

Figure 2.2 shows a representative schematic of the heat treatment cycle of Haynes 282 [9], which is fairly new Ni-based superalloy.

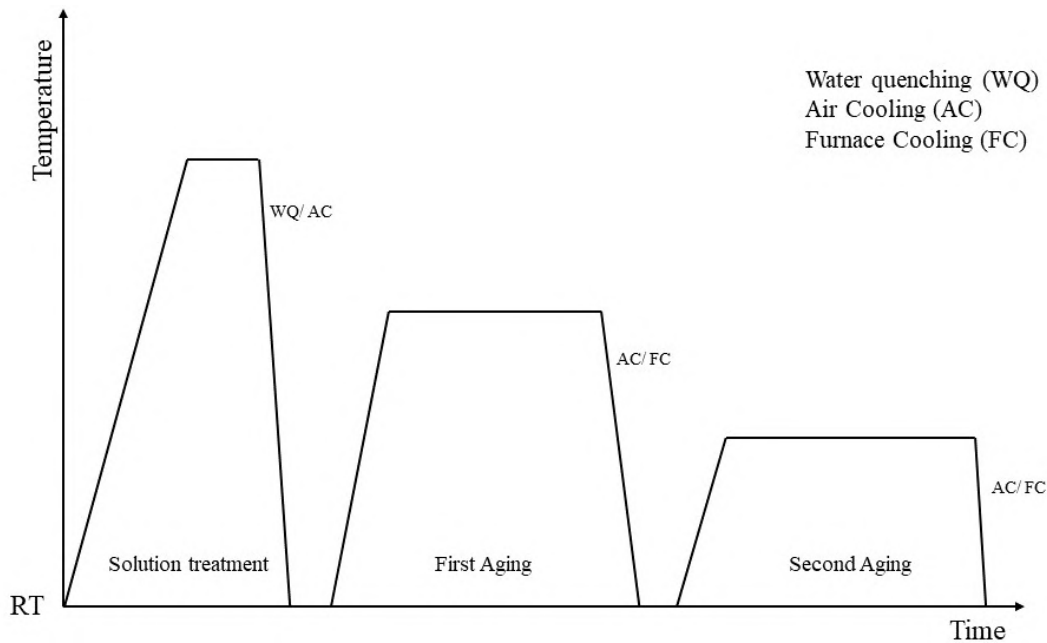


Figure 2.2: Schematic diagram of heat treatment steps in Haynes 282.

The first step of solution treatment between the temperature range of 1050 °C to 1200 °C ensures the dissolving of all strengthening phases such as γ' and carbides except for the stable MC carbides [9]. The first step of the dual-stage aging process for γ' is the carbide stabilization process, whereas the second step completes the precipitation of the γ' phase. It should be noted that the 3 step heat treatment shown in this figure is particular to Haynes 282. Generalization for all Ni-base superalloys should not be made because the heat treatment steps can be changed depending on what is desired from the final microstructure and the property requirements for the application.

2.1.3.2 Phases in Ni-base Superalloys

Successful completion of the heat treatment process will result in the phases of γ , γ' , and carbides. MC carbides form from melt due to the strong segregation of C and N.

The carbides present at the grain boundaries contribute to the creep strength and ductility by inhibiting grain boundary sliding [9]. However, after heat treatments and long operating duration, it has been observed that they decompose to other types of carbides. Most common reactions include [19]:

- $MC + \gamma \longrightarrow M_{23}C_6 + \gamma'$
- $MC + \gamma \longrightarrow M_6C + \gamma'$

Table 2.2 describes the phase details, crystal structure and morphology of the observed phases.

Table 2.2: Phases observed in Ni-base superalloys. [5, 20]

Phase	Crystal Structure	Morphology
γ	FCC	Matrix phase of Ni-base superalloys. It is a solid solution austenitic phase of the alloying elements.
γ' $Ni_3(Al, Ti)$	FCC (ordered $L1_2$)	Variation in shape from spherical to cuboidal. Size varies with aging time and temperature. In recently developed Ni-base alloys gamma prime has cuboidal morphology.
MC	Cubic	Globular, irregular shaped particles.
$M_{23}C_6$	FCC	Can precipitate as films, globules, platelets and cells. Commonly precipitates at grain boundaries.
M_6C	FCC	Discrete, platelet.

2.2 VDM 780

VDM®Alloy 780, represented as VDM 780, is a novel wrought Ni-base superalloy family developed in 2014 specifically for high temperature applications (above 650 °C) while keeping the good workability of IN718 [19]. Although this alloy was initially promoted as "a new 718-type superalloy", the only similarity between IN718 and VDM 780 is the good workability. Apart from that, VDM 780 inherits its high strength from γ' - $Ni_3(Al, Ti)$ precipitation, whereas IN718 gets it from γ'' precipitation. It is essential to understand the difference between the main strengthening phase. Because the intended application temperatures of VDM 780 are beyond 650 °C and at these temperatures, the main strengthening γ'' -phase of IN718 becomes unstable and results in loss of the alloy's strength [12]. Due to this reason, it can be said that this alloy is more similar to another γ' -strengthened alloy, Waspaloy, which also has similar application temperatures. VDM 780 also eliminates the main disadvantage of Waspaloy, which is the very limited formability of the alloy due to its tight grain forging window between γ' and $M_{23}C_6$ solvus temperatures, which is around 15 °C [23]. The developers of VDM 780 have studied many different compositions [23,24] and came up with the composition in Table 2.3 that enables both stability at elevated temperatures and fine grain forging:

In addition to the main strengthening phase of γ' , there is also a high-temperature phase for grain refinement. The initial studies on VDM 780 interpreted this phase

Table 2.3: VDM 780's chemical composition (wt%) compared with Waspaloy's and IN 718's.

	C	Cr	Ni	Mn	Si	Mo	Ti	Nb	Fe	Al	Co
VDM 780	0.021	17.95	Bal.	0.01	0.03	2.91	0.3	5.39	0.47	2.19	25.08
Waspaloy	0.07	19.5	Bal.	0.03	-	4.3	3.0	-	1.2	1.4	13.5
IN 718	0.04	18.5	Bal.	0.09	-	3.0	0.9	5.1	18.5	0.5	-

as the δ -Ni₃Nb phase with D0_a crystal structure. However, recent studies with high-resolution transmission electron microscopy (HRTEM) analysis suggest that it is an ordered type of η -Ni₆AlNb phase and layered structure within individual particles, consisting of η and δ phases [25,26,27]. This high-temperature phase was noted to be sluggish compared to the main strengthening γ' -phase, which means a longer duration of heat treatment will be necessary if precipitation of this phase is desired [28]. It is important to note that no mention of carbides in any study related to VDM 780 was made. Therefore a sufficient amount of precipitation for the high-temperature phase is necessary after the heat treatments if the grain boundaries are to be strengthened and grain growth to be hindered.

2.2.1 Standard Heat Treatment

VDM 780 is still an alloy in development, it has not been adopted by industry, and the heat treatment process is a development field. A couple of studies [26, 27, 28] have applied different heat treatments by varying the temperature, time, and cooling rate of the heat treatment steps, and the most established one seems to be the heat treatment cycle that can be seen in Figure 2.3. This heat treatment is studied in this thesis work as the second objective, and the results are presented in Section 4.4.

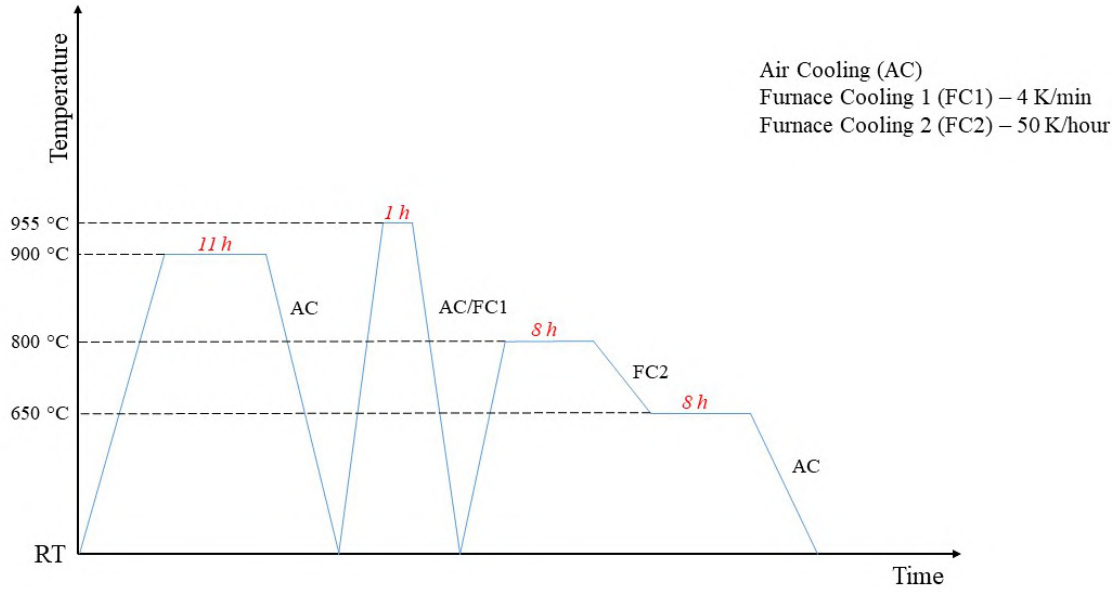


Figure 2.3: Schematic diagram of the standard heat treatment cycle in literature [28].

This "standard" heat treatment cycle consists of 4 stages; 2 at high and two at low temperatures. According to Bergner et al, (2018). [28] the initial stage at 900 °C for 11 hours is required to precipitate a sufficient amount of the high-temperature phase at grain boundaries. This precipitation will prevent grain growth in further stages of heat treatment and during the actual operating environments. The second high-temperature stage at 955 °C for 1 hour is applied to dissolve any existing γ' , and the third and fourth stages at 800 °C and 600 °C for 8 hours each are used to precipitate γ' while not resulting in any morphological change in the high-temperature phase. After this complete heat treatment cycle, the resultant microstructure consists of a bi-modal distribution of γ' precipitates with heterogeneous distribution of the high-temperature phase along the grain boundaries.

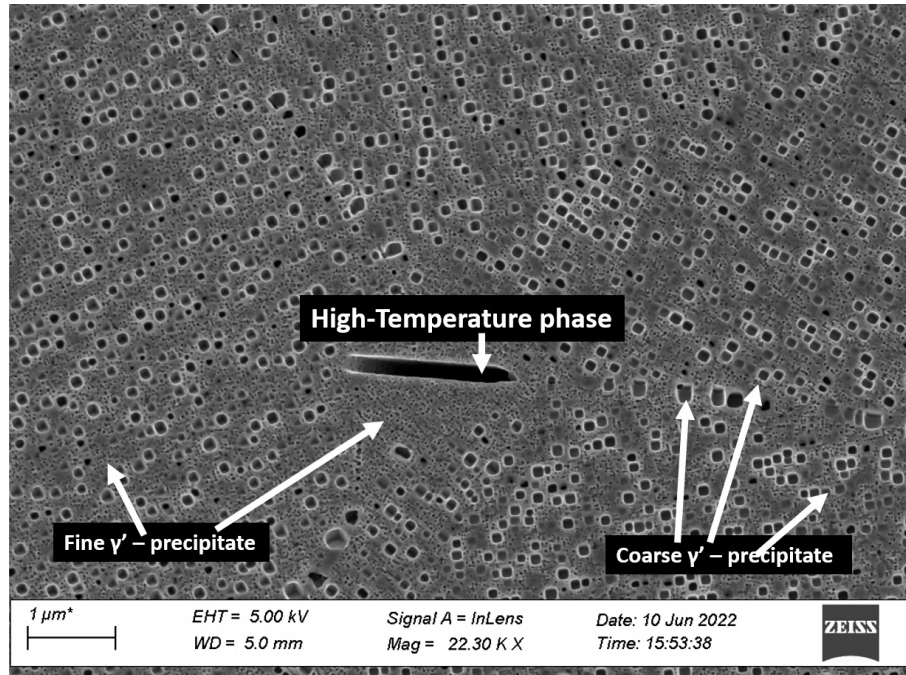


Figure 2.4: VDM 780 sample subjected to full heat treatment cycle and etched with Canadas etchant (γ' and high-temperature phases are etched away).

As it can be observed in Figure 2.4 after the complete heat treatment cycle, the resultant morphology of the high-temperature phase is plate and needle-like. For the bi-modal precipitation of γ' , the coarse precipitates are cuboidal or almost cuboidal, whereas the fine precipitates are in spherical morphology.

2.3 Isothermal Transformations

The microstructural features forming as a result of superalloys' heat treatment are metastable, meaning that their percentage amount and structure will change with the variations of time and the heat treatment temperature [9]. Isothermal heat treatment is performed by controlling either time or the temperature and changing only one of these variables in this heat treatment. The result of the microstructural evolution after the isothermal heat treatments shows the phases and their percentage amounts after each isothermal heat treatment are completed. When the transformation data about the phases are collected for each temperature and time, a time-temperature-transformation (TTT) diagram can be plotted. These diagrams are generated from percentage transformation vs. time measurements and help understand the transformations of an alloy at elevated temperatures. Understanding a TTT diagram is a basis for understanding the design behind a complete heat treatment cycle and can further open the way to optimize them.

In the case of the isothermal transformations in VDM 780, only one study [28] has studied the isothermal transformations by varying the temperature from 850 °C

to 975 °C and the time from 1 hour to 16 hours. However, this study does not fully reveal what happens in each specific temperature and time studied and only gives a general understanding. Hence, a TTT diagram is also not available for VDM 780.

3

Methods

This chapter describes the design of the heat treatment process, sample preparation and characterization. Metallography steps have been conducted at the GKN Aerospace Sweden AB facilities in Trollhättan. Polishing, etching and imaging with Optical Microscopy has been performed at both University of West and GKN Aerospace.

3.1 Simulations

Computational simulations were done before starting the experimental procedure to determine; the solvus temperatures of the phases from equilibrium diagrams, the time and temperature required to precipitate each phase from TTT curves, and the amount of each phase forming at each isothermal temperature from phase evolution diagrams. This work was carried out using JMatPro, simulation software that estimates alloy characteristics and is especially powerful in multicomponent alloys.

In the user interface of the JMatPro software, after entering the material composition of VDM 780 as an input, which can be found in Table 2.3, three different modules will be used to obtain the simulation results.

3.1.1 Equilibrium Diagram

The phase content of the alloy can be calculated at any temperature and composition using "equilibrium diagrams," also known as "constitutional and phase diagrams." When the alloy is heated and cooled under equilibrium conditions, when every process in the system is reversible, they make it possible to see the phase changes.

Equilibrium diagrams for VDM 780 were obtained from "Step Temperature" calculations under the "Thermodynamic Properties" module of JMatPro. This selection automatically considers the phases that can be obtained from this elemental composition. However, based on the literature, only four phases are expected to be observed: γ , γ' , a high-temperature phase containing both δ and η [26, 27], and a liquid phase. Even though carbides were not mentioned in the previous studies, an option for MC , M_2C_6 , and M_7C_3 carbides was still turned on to see if the simulation matched up with the data from experiments.

According to Figure 3.1, the microstructure lacks carbides, which correlates with

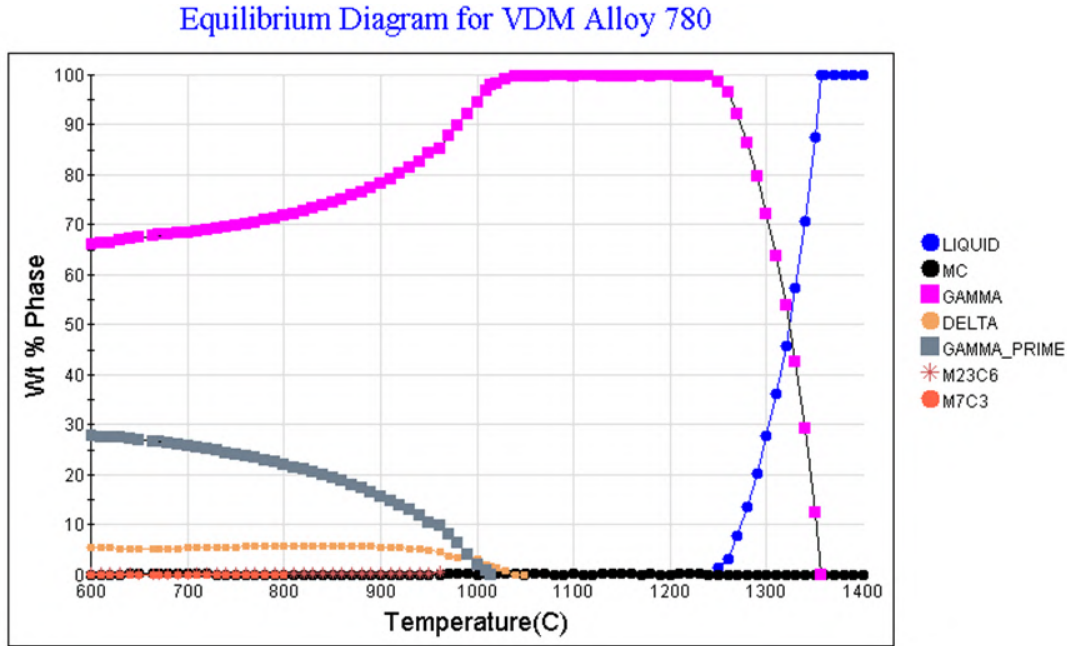


Figure 3.1: Equilibrium diagram from JMatPro for VDM780 composition.

the literature data. Even though both δ and η phases were chosen to represent the high-temperature phase, according to the JMatPro database, only δ phase appears to be forming.

3.1.2 TTT Diagram

A TTT diagram is necessary to predict the temperature and time necessary for transforming γ' and the high-temperature phase under the isothermal conditions. A TTT diagram was obtained from JMatPro's "Quick TTT/CCT diagram" calculation under the "Phase Transformations" module, and the results are presented in Section 4.1. However, in these calculations, only the δ phase is considered the high-temperature phase since the calculations did not generate an η phase.

3.1.3 Isothermal Heat Treatment (IHT)

The last section of the simulations focuses on the thermodynamic evolution of the phases under certain isothermal heat treatment conditions. Phase evolutions for γ' and δ phases at each IHT temperature are calculated, and the respective diagrams are obtained from JmatPro's "Isothermal" calculation under the "Phase Transformations" module. The results from these simulations are shown in Section 4.1.3, and then they are compared to the experimental results in Section 4.3.3.1.

3.2 Material

The starting material was a wrought VDM 780 that was rolled into the plate form. The detailed material composition can be seen in Table 3.1. The size of the as-received state plates was 60 mm x 300 mm x 3 mm, which was above the size limits of the heat treatment furnace. The condition of the as-received material was identified as solution annealed in the material specification sheet. However, the temperature of the solution annealing process was not specified. Therefore, a consensus regarding if the γ' phase was completely dissolved or not could be established. For this reason, the received group of materials consisted of 4 plates divided into two groups; two remained in the as-received initial state, and for the other two, a solutionizing heat treatment (SoHT) prior to the IHTs was performed. The details of this solutionizing heat treatment will be given in Section 3.3. Overall, the plates were subjected to two cutting sequences before the IHTs; the first one to reduce the size of the 2 plates subjected to the SoHT, and a second one for all the plates with both as-received and solutionized initial states where they were cut into squares of 30 mm x 30 mm. The material cutting process in Grand Premium X-series model water jet cutter by WJS.

Each sample was individually heat treated under different isothermal conditions (time and temperature). After the completion of the IHTs, samples with the initial state of as-received and solutionized were compared. Microstructural characterization and hardness evaluations were done on all samples to determine the phase change introduced during different conditions of the IHTs.

Table 3.1: Complete element composition of VDM 780.

VDM 780 Chem. wt.%							
C	S	N	Cr	Ni	Mn	Si	Mo
0.021	0.0005	0.008	17.95	45.56	0.01	0.03	2.91
Ti	Nb	Cu	Fe	P	Al	Mg	Pb
0.3	5.39	0.01	0.47	0.01	2.19	0.0006	0.0002
Sn	Ca	V	Zr	W	Co	B	As
<0.0010	0.002	0.01	<0.01	0.02	25.08	0.003	<0.0010
Ta	Se	Te	Bi	Sb	Ag	O	
0.01	<0.0003	<0.00005	<0.00003	<0.0005	<0.0001	0.001	

3.3 Heat Treatments

As previously stated, two heat treatments were performed in this study; a SoHT for half of the samples and IHTs for all samples. SoHT was performed at 1050°C for 30 minutes. Following that, the IHTs were performed on samples with an initial state of as-received and solutionized. As a result, after the completion of IHTs, two samples with both initial states were obtained for each isothermal condition. Schematic illustrations of complete heat treatment cycles for samples with two initial states can be observed in Figures 3.2 and 3.3

Both SoHT and IHTs were performed in a calibrated Muffle furnace, Nabertherm s/n 2401209, at GKN Aerospace. This furnace is accurate to within 10°C between 600°C and 850°C and within 8°C between 850°C and 1140°C.

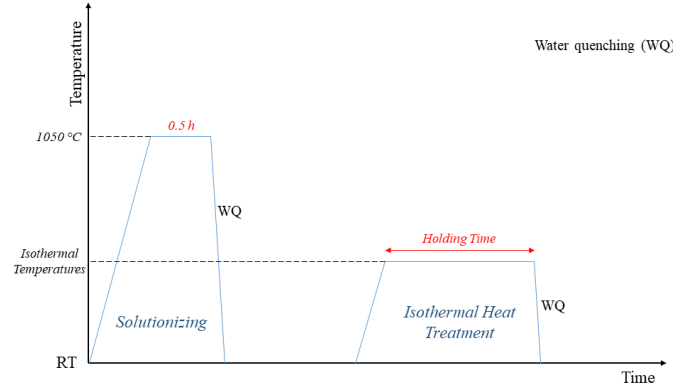


Figure 3.2: Schematic illustration of complete heat treatment cycle with 'solutionized' starting condition.

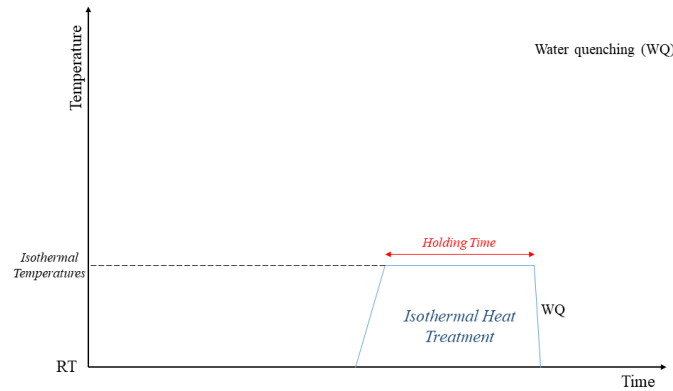


Figure 3.3: Schematic illustration of complete heat treatment cycle with 'as-received' starting condition.

3.3.0.1 Isothermal Heat Treatments

The IHTs were performed between the temperature range of 600 °C and 1000 °C. The holding time ranged from 1 hour to 16 hours. Table 3.2 contains the complete list of IHT parameters defined for this study and the nomenclature for the samples. The samples were labeled as follows:

- In the first letter, the solutionized initial state of the material is identified with "S", and the as-received initial state of the material is identified with "A".

- In the following letter, the six different IHT temperatures are identified with letters from (a) to (f) in the systematically increasing order between the range of 600 and 1000 °C.
- The last one or two letters show the heat treatment duration. For 16 hours, both numbers were necessary; otherwise might have been confused with 1-hour heat treatment.

Table 3.2: IHT conditions.

Temperature (°C)	Holding Time			
	1 hour (1)	4 hour (4)	8 hour (8)	16 hour (16)
(a)	S(a)1	S(a)4	S(a)8	S(a)16
	A(a)1	A(a)4	A(a)8	A(a)16
(b)	S(b)1	S(b)4	S(b)8	S(b)16
	A(b)1	A(b)4	A(b)8	A(b)16
(c)	S(c)1	S(c)4	S(c)8	S(c)16
	A(c)1	A(c)4	A(c)8	A(c)16
(d)	S(d)1	S(d)4	S(d)8	S(d)16
	A(d)1	A854	A(d)8	A(d)16
(e)	S(e)1	S(e)4	S(e)8	S(e)16
	A(e)1	A(e)4	A(e)8	A(e)16
(f)	S(f)1	S(f)4	S(f)8	S(f)16
	A(f)1	A(f)4	A(f)8	A(f)16

In addition to the 48 samples described in Table 3.2 there are two more samples: A000, and S000. These two specimens are sample cuts from the original as-received and solutionized plates, which have not been subjected to IHTs for reference and comparison purposes. The grain size and hardness measurements with the analysis on the γ' phase for these two samples will be discussed in Chapter 4.

3.3.0.2 Cooling Rates

The selection of the cooling rate carries as much importance as the selection of the heat treatment temperature and the duration. The cooling rate is vital in tailoring the precipitates' microstructure, size, and volume fraction. Suppose a slow cooling such as furnace cooling is applied. In that case, the heat treatment will continue because the heat surrounding the material will continue its effect on the phase transformations. In order to observe the microstructure as it was at the IHT temperature, the cooling needs to be swiftly performed with water quenching. This will freeze the microstructure as it was during the heat treatment and reveal the correct microstructural to be observed. Hence, in this study, only one type of cooling method, water quenching, was preferred for all IHTs, and the other cooling methods were not studied.

3.4 Metallographic Preparation

All heat-treated samples were cut down into smaller square sizes of 15 mm x 15 mm to fit them into a 30 mm diameter mount. The process was operated on a Struers Secotom-50 precision cutting machine with a 30A15 cutting wheel. Subsequently, the pieces were mounted with PolyFast according to Struers' instructions. This type of mount is selected since it is electrically conductive; therefore, Scanning Electron Microscopy (SEM) evaluation for high-resolution imaging is possible. The following grinding and polishing steps were performed on the Struers Tegramin-30 machine, but a program adapted from Buehler's instructions [30] was used. The consumables and the selected parameters during these steps can be seen in Table 3.3.

Table 3.3: Sample preparation procedure for Ni-base superalloy specimen. Adapted from Buehler's instructions [30].

Sample Prep. Step	Equipment(s)	Consumable	Parameter		
Cutting	Water-jet cutting machine				
Mounting	Citopress 30	Polyfast	Struers' program		
Grinding and Polishing	Tegramin 30	MD-Piano disks 3A27 #150 & Grinding water	500 micron		
		MD-Dur & 9-micron diamond abrasive	5 min	30 N	100/150 Comp.
		MD-Dac & 3-micron diamond abrasive	4 min	30 N	100/150 Comp.
		MD-Nap & 1-micron diamond abrasive	3 min	30 N	100/150 Comp.
		MD-Nap & 0.2-micron diamond abrasive	1 min	Hand held	100 rpm.

Etching was done using two techniques: electrolytic etching with 10% Oxalic acid and immersion etching with Canadas etchant, see Table 3.4. Using two etchants was to observe and analyze different microstructural features. Electrolytic etching with Oxalic acid is a well-established method to reveal γ' , γ'' phase, and carbides by etching off the matrix. However, no particular study shows the effect of revealing the high-temperature phase. For this reason, immersion etching with Canadas etchant, suggested by VDM-Metals, was used to reveal the high-temperature phase and γ' phase. However, these phases are revealed by phase removal because of the hydrofluoric acid in Canadas etchant's composition. Therefore, Canadas was efficient since it revealed more high-temperature phase compared to etching with Oxalic acid and helped visualize the morphology of this phase. However, to reveal the elemental composition of the specific phases with energy-dispersive X-ray spectroscopy (EDS) analysis, SEM images obtained from etching with Oxalic acid were used.

Table 3.4: VDM 780 etchants.

Etchant	Compostion	Method	Parameters
Oxalic Etching Solution	10 g Oxalic acid, 90 ml H2O [28]	Electrolytic	2.5 V and max current for 2 seconds
Canadas Etchant	80 ml distilled water, 20 ml H2SO4, 20 ml HF, 10 ml HNO3	Immersion	Immerse fully for 10-15 minutes Etchant should be fresh

3.5 Characterization Methods and Hardness

This section will systematically discuss the various material characterization methods used during this study. These methods include grain size measurements, the precipitate size and volume fraction of the γ' phase, qualitative study of the high-temperature phase, and hardness measurements.

3.5.1 Light Optical Microscopy (LOM)

LOM images obtained from the Nikon Eclipse LV150 model microscope were utilized to observe the microstructure, including carbides and precipitation of high-temperature phases that fall under the LOM range. The grain size measurements were performed using the Zeiss Axioscope 5 model microscope images.

3.5.1.1 Grain Size Measurement

The measurements on samples were performed with Jeffries planimetric method according to the ASTM E112 standard, and NIS Elements software was used to enhance the image digitally and count the grains. In this method, a circle with a known area, denoted as A , is inscribed on the LOM image. While doing this, the magnification of the image should be adjusted so that at least 50 grains can be counted in the field. The images were captured in 100x magnification for this purpose. The number of grains that fall entirely inside the circle is then counted, denoted as N_{Inside} , followed by the number of grains that intercept the circle, denoted as $N_{Intercepted}$. Figure 3.4 shows an example of this process.

After that the following Equation 3.1 is then used in the ASTM standard to calculate grain size, N_A :

$$N_A = \frac{N_{Inside} + 0.5 * N_{Intercepted}}{A} \quad (3.1)$$

A minimum of three images from each sample were taken for an accurate grain size calculation, and the average of these three results was the final ASTM grain size for the sample.

3.5.2 Scanning Electron Microscopy (SEM)

The size of the γ' precipitates is so small in the nano-scale that even the highest magnification in LOM cannot give an image that can lead to quantitative analysis.

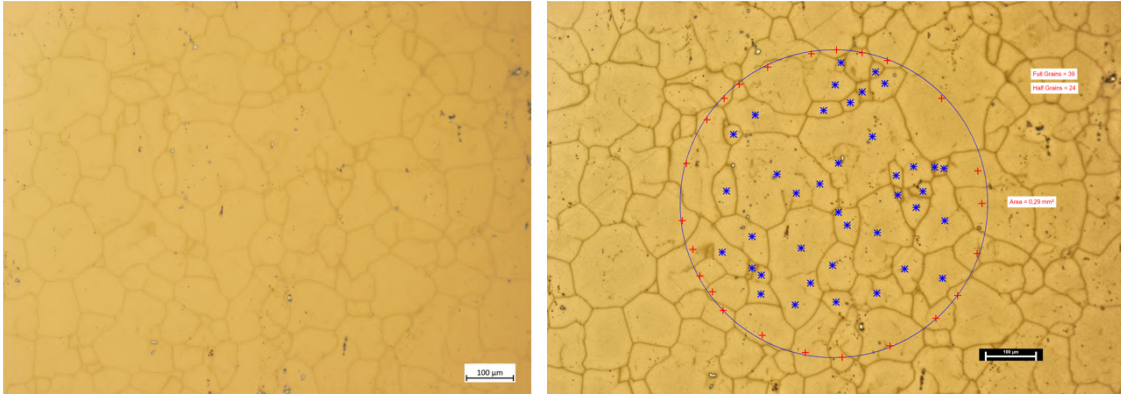


Figure 3.4: Grain size measurement of S000 with Jeffries planimetric method. Left is the original OM image of the sample electrolytic etched with Oxalic acid and on the right is the processed image by using NIS Elements software.

Therefore, SEM was utilized to obtain microstructure images at various magnifications. For the γ' precipitate size and volume fraction measurements, images were captured at 50000x and 20000x magnifications. Next, these images are used as an input for MIPAR and ImageJ, two image processing software, and precipitate size and volume fractions of the γ' phase are analyzed. Furthermore, the elemental composition of each phase was identified by an elemental micro-analysis with an EDS probe by Oxford Instruments, which was integrated into the SEM machine that directly transfers the data to software AZtec to be analyzed.

3.5.2.1 MIPAR and ImageJ

MIPAR and ImageJ are both powerful image analysis software. With the operation of two different software packages, the capabilities of both software and the results obtained from these analyses could be compared. As shown in Figures 3.5 and 3.6, both software uses a premade recipe that includes cropping the image, adjusting the contrast, having an adaptive threshold, and so on. The preparation of a recipe makes the batch analyses faster. However, depending on the original image contrast and the resolution obtained from SEM, these values in the recipe needed an update, so it was not a constant recipe for all images. In order to have an accurate measurement, five SEM images from each sample were analyzed, and the average results of these five analyses for both γ' precipitate size and volume fraction were taken as the final values.

3.5.3 Hardness

The hardness of the samples was measured with a Vickers type macro hardness test with 10 kg of load according to the ASTM E384-17 standard. The testing equipment was a calibrated Vickers Armstrong Pyramid Hardness Tester. This test machine was also calibrated with test blocks before each use to ensure the reliability of the

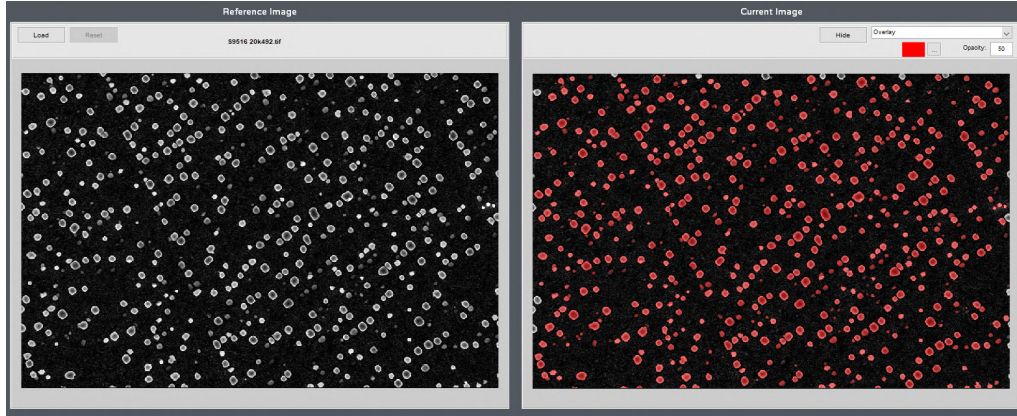


Figure 3.5: Left is the original image input during MIPAR recipe construction, while right is the processed image highlighting the γ' precipitates.

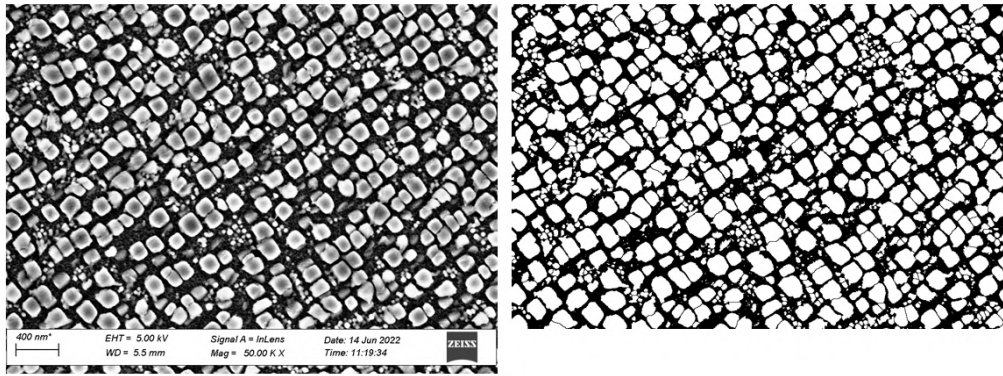


Figure 3.6: Left is the original image input during ImageJ macro-code development, while right is the processed image highlighting the γ' precipitates.

results. Since the machine was manual, not digital, the diagonal lengths of each indentation were measured, and the average of the two diagonal lengths was used to calculate the average diagonal length for that indentation. The following Equation 3.2 is used to obtain the hardness value in Vickers for a single indentation. P is the load applied, and d is the average diagonal length of the indentation.

$$HV = 1.8544 * \frac{P}{d^2} \quad (3.2)$$

Hardness measurements were taken from 2 indentations at each corner and the center, resulting in 10 indentations, as seen in Figure 3.7. The average of these measurements was the accepted hardness of the sample.

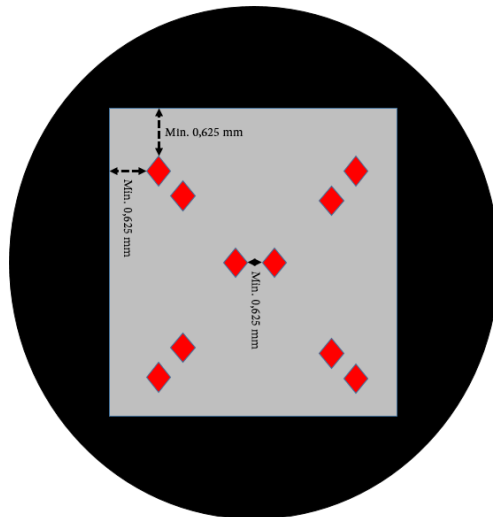


Figure 3.7: Schematic representation of hardness indentations and the minimum distances.

4

Results and Discussion

This chapter summarizes the most important findings from the experiments. The section simply includes the most important data and tables, with the appendix serving as a supplement.

4.1 Simulation Results

The equilibrium diagram previously shown in Figure 3.1 is important for two parameters; the solvus temperature of the phases and the amount of wt% of the phase at room temperature (RT) can form after sufficient heat treatment. In Figure 4.1, these two values are marked for both γ' and δ phases. As previously stated in Section 3.1.1, simulation was not producing an η phase, which, along with the δ phase, constitutes the high-temperature phase in VDM 780. Therefore, in the following sections related to the simulations, δ phase will represent the high-temperature phase.

4.1.1 Equilibrium Diagram

In the equilibrium diagram, the wt% of γ' phase for each temperature from 600 °C to its solvus temperature is drawn with the grey line. The δ phase is represented by the orange line. The solvus temperatures for these phases are visible from the intersection points of the grey and orange lines with the x-axis. The solvus temperature of the γ' phase is 1014 °C, while it is 1048 °C for δ phase. Furthermore, the wt% of these phases at RT is 27,8% for γ' phase and 5,6% for δ phase in the γ matrix.

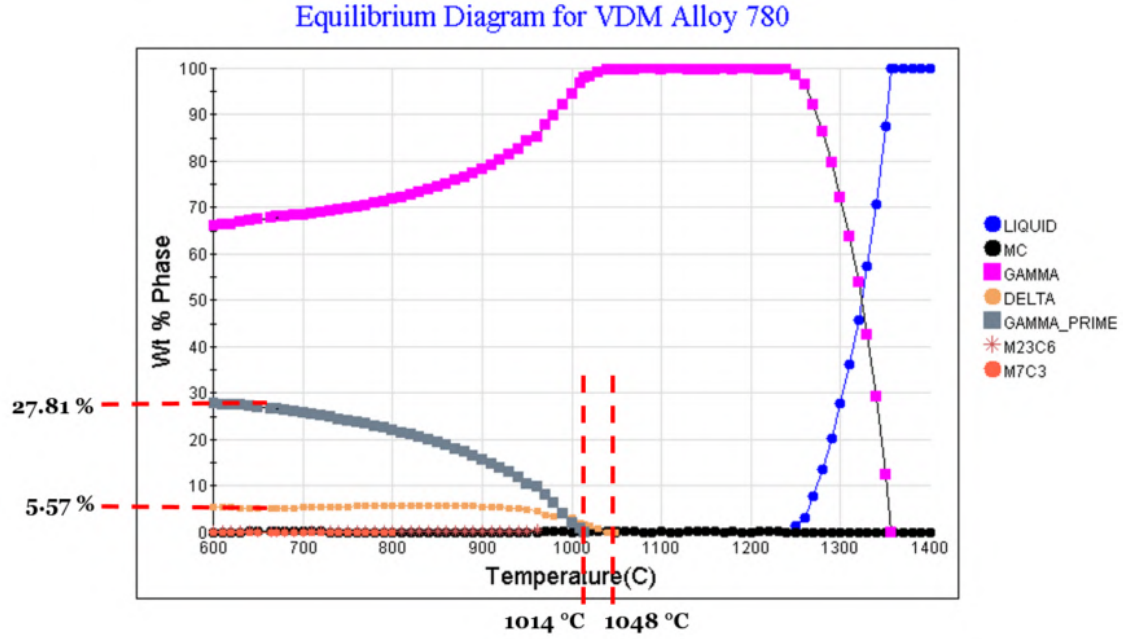


Figure 4.1: Equilibrium diagram highlighting the solvus temperature of the selected phases and their wt% at RT.

4.1.2 TTT Diagram

The TTT diagram in Figure 4.2 displays the time required to start the transformation of both phases between the 600 and 900 °C temperature range. For the γ' phase, the transformation starts promptly after 7 minutes when the temperature is around 840 °C. Above and below this temperature, the time it takes to start the transformation increases. A similar trend is also seen for the δ phase, but on contrary to the γ' phase, the precipitation kinetics of the δ phase is much slower, which is referred to as "sluggish" in the literature [25]. The minimum time to start the transformation is almost 12 hours at 790 °C.

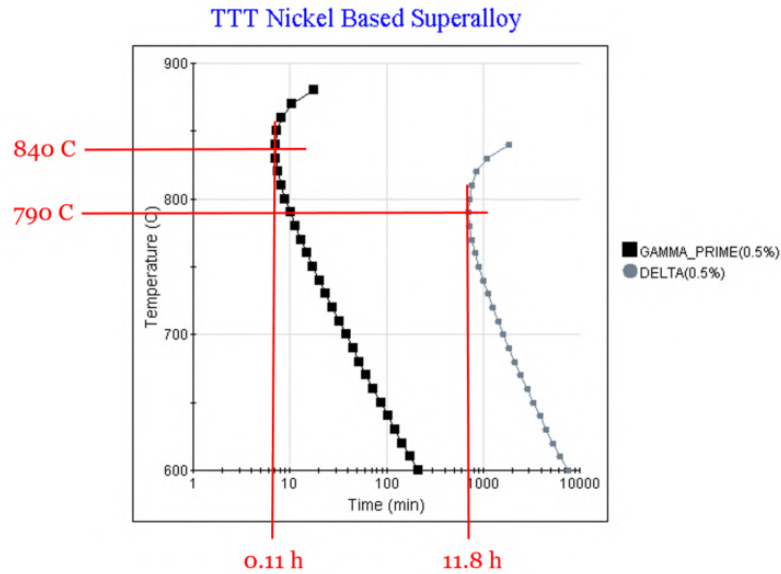


Figure 4.2: TTT diagram for γ' - and δ -phases in VDM 780.

4.1.3 Phase Evolution Diagrams

The IHT simulation results are presented in phase evolution diagrams, which show the amount of phase forming in atomic percentages (at.%) for the γ' and δ phases at each isothermal temperature. In these diagrams, the x-axis displays the time passed from the start of the heat treatment, and the y-axis displays the fraction (at.%) of the phase forming. The results of these calculations are presented in Table 4.1. The IHT at (a) °C in Figure 4.3 shows the highest at.% of the γ' phase after the most prolonged heat treatment duration of 16 hours with 31,39%. On the other hand, the maximum at.% for δ phase is reached after the IHT at (b) °C for 16 hours with 9,5% in Figure 4.4. The rest of the phase evolution diagrams can be found in Appendix A.1.

Table 4.1: Amount of precipitating phase at each isothermal heat treatment temperature.

Isothermal Heat Treatment Temperature °C	Atomic Percent of γ' -phase (at.%)	Atomic Percent of δ -phase (at.%)
(a)	31,39	1,02
(b)	28,87	7,08
(c)	26,72	9,08
(d)	23,58	8,34
(e)	18,91	6,72
(f)	11,94	4,72

Simulation automatically generates the phase fraction in the at.% unit. In order to

compare these results with the experimental results, they need to be converted to the weight percentage (wt.%). This transformation is calculated using the atomic weight (at. wt.) and the at.% values obtained from the simulations. The results of these calculations will be shown in comparison to experimental results in Section 4.3.3.1.

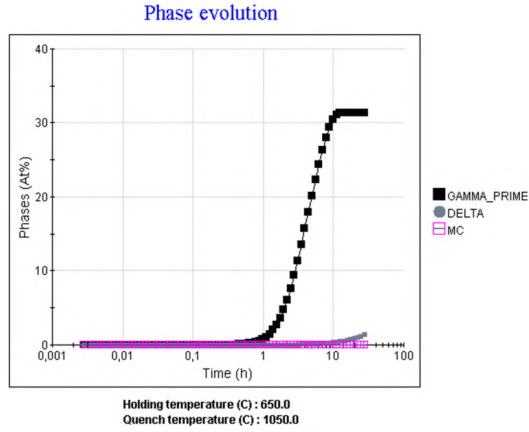


Figure 4.3: Phase evolution at isothermal temperature at (a) °C.

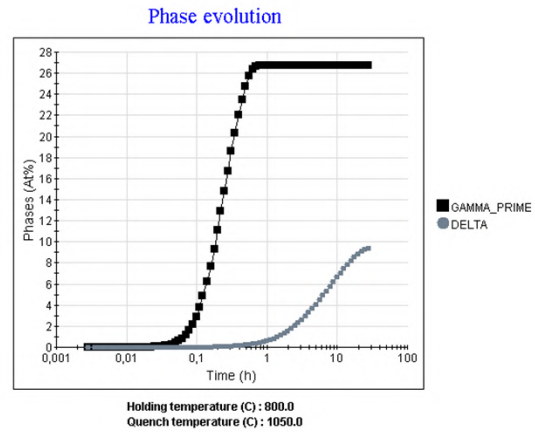


Figure 4.4: Phase evolution at isothermal temperature at (c) °C.

4.2 Initial States

Before the IHTs were performed, two initial states of the material were introduced; the as-received and the solutionized state. In Section 3.3.0.1, it was stated that the samples for these materials without any heat treatments were made under the names A000 for the as-received state and S000 for the solutionized state. The results of the microstructural evaluation of these samples are presented in this section.

4.2.1 As-Received State

4.2.1.1 Grain Size Measurement

The grain size measurement using the LOM images of sample A000 indicated that the material in the as-received state has a grain size of 4,5 ASTM.

4.2.1.2 γ' Phase Analysis

The microstructure of the sample A000 was observed with SEM. However, none of them showed noticeable precipitates of γ' phase. Figure 4.5 shows one of the taken images. A few white particles can be noticed from this image, but they are found to be remains of the etchant that could not be cleaned away, and they should not be misinterpreted with γ' precipitates.

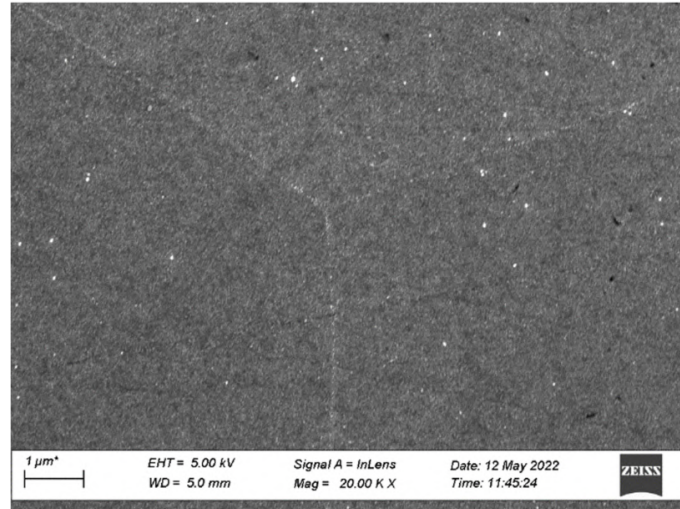


Figure 4.5: SEM image of microstructure of initial as-received state before IHTs.

4.2.1.3 Hardness Measurement

The hardness for the sample A000 is measured to be 340 HV.

4.2.2 Solutionized State

4.2.2.1 Grain Size Measurement

The grain size measurement using the LOM images of the sample S000 indicated that the material in the solutionized state has a grain size of 4,5 ASTM, the same as the as-received state.

4.2.2.2 γ' Phase Analysis

The analyzed SEM images for the sample S000 did not show any precipitation of γ' phase, as can be seen in Figure 4.6. This was expected as the material was subjected to the SHT beyond γ' solvus temperature, which should dissolve any precipitate inside the matrix.

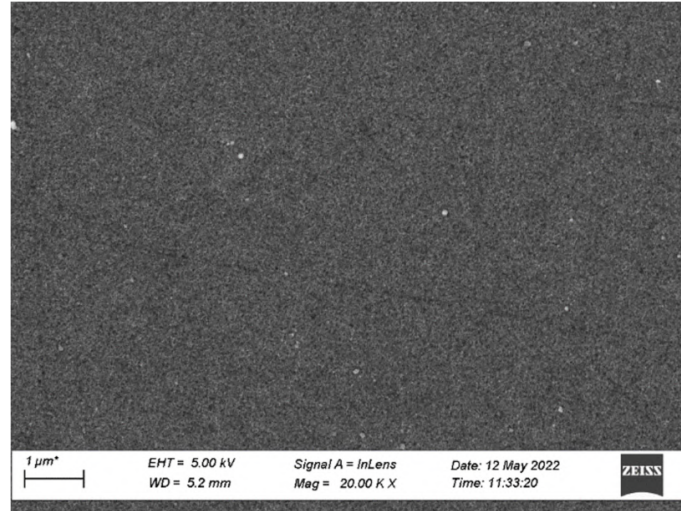


Figure 4.6: SEM image of microstructure of initial solutionized state before IHTs.

4.2.2.3 Hardness Measurement

The hardness for the sample S000 is measured to be 216 HV.

4.2.3 Comparison of Initial States

In both initial states, the grain size was measured to be 4,5 ASTM, and, as a result of the microstructural observation on both samples, γ' precipitates were not observed. However, there is a significant difference in the hardness values; 340 HV in the as-received initial state and 216 HV in the solutionized initial state. This difference between the two values indicates that γ' precipitates are present in the as-received state. However, their size is so small that the resolution of SEM cannot capture these precipitates.

4.3 Isothermal Heat Treatments

The microstructural characterization to understand the effect of different IHT conditions on VDM 780 are in five sections:

1. Grain size measurement from the LOM images.
2. Hardness measurements for all samples.
3. Analysis of γ' phase with the SEM images.
4. Qualitative analysis on the high-temperature phase with LOM and SEM images.
5. Elemental study of the phases with EDS.

4.3.1 Grain Size Measurements

The grain size measurements were possible after the electrolytic etching of the samples with Oxalic acid, as shown in Figure 4.7 (a) for sample A(c)1. This technique revealed the grains by etching off the grain boundaries. Although this technique worked well for some of the samples, this was not the case for all of them, such as for the sample A(c)16 in Figure 4.7 (b). The only difference between Figure 4.7 (a) and (b) is the heat treatment duration of 1 hour and 16 hours. In most cases it is seen that the samples which were subjected to IHTs above the temperature of (c) °C for more than 1 hour did not react in the same way to the etching as the rest of the samples because the grains were not distinguishable even after the image quality was enhanced with NIS Elements software. This issue can be visually understood in Figure 4.7. Regrettably, for these samples, a new etching method could not be studied due to the limited time frame of the thesis. Thus, the grain size measurement could only be performed for a few samples. These measurements are plotted in Figures 4.8 and 4.9 where the x-axis represents the IHT duration in hours, and the y-axis represents ASTM grain size. In these plots, the black lines show the grain size of the initial states, and their value is not changing with increasing duration since they were not subjected to the IHTs. The red, green, and purple lines are for IHT temperatures of (a), (b), and (c) °C, respectively.

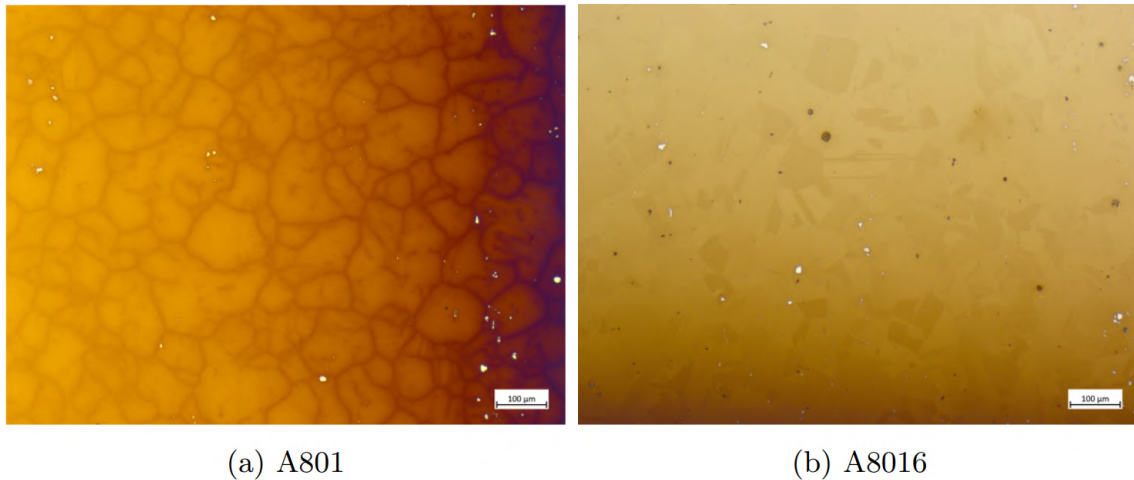


Figure 4.7: Electrolytic etching of the two samples with the same etching parameters that can be seen in Table 3.4.

The grain size measurements show that the grain size fluctuates around 4,5 ASTM until the IHTs at (c) °C for 1 hour. Although the trend suggests that the remaining IHTs also result in the same ASTM grain size, it cannot be said that the rest also have the same grain size without the correct measurements. However, to relate the influence of γ' precipitates and their effect on the hardening of the alloy after the IHTs in Section 4.3.3.2, an assumption of no change in the grain size must be made. Otherwise, the hardening of the material can be related to a change in the grain size, which will limit the correlation between hardening and the γ' phase.

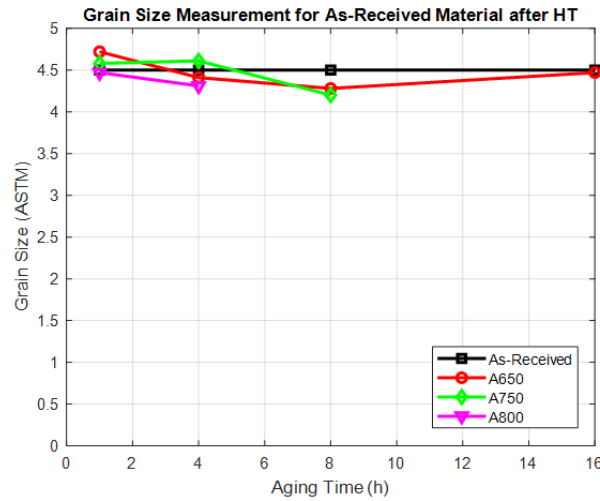


Figure 4.8: Grain size measurement of samples that have the initial state of as-received.

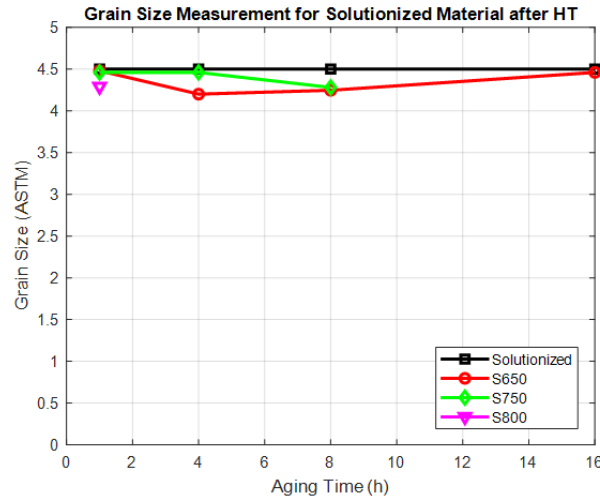


Figure 4.9: Grain size measurement of samples that have the initial state of solutionized.

4.3.2 Hardness Measurements

The hardness of the as-received and solutionized initial states was stated in Section 4.2. The hardness of the samples that were subjected to the IHTs was measured using the same method. Their results are presented with a line plot in Figure 4.10. The x-axis of this plot represents the increasing IHT duration in hours, while the y-axis represents the range of hardness values in Vickers units. In these plots, the solid and dashed black lines show the hardness of the initial states, and their measurements are not changing with increasing duration since they were not subjected to the IHTs. The red, green, blue, purple, cyan, and yellow lines constitute IHTs at the temperatures of (a) °C, (b) °C, (c) °C, (d) °C, (e) °C and (f) °C, in the same order. While the solid lines are for the samples with an initial state of as-received, the dashed lines are for the samples with an initial state of solutionized.

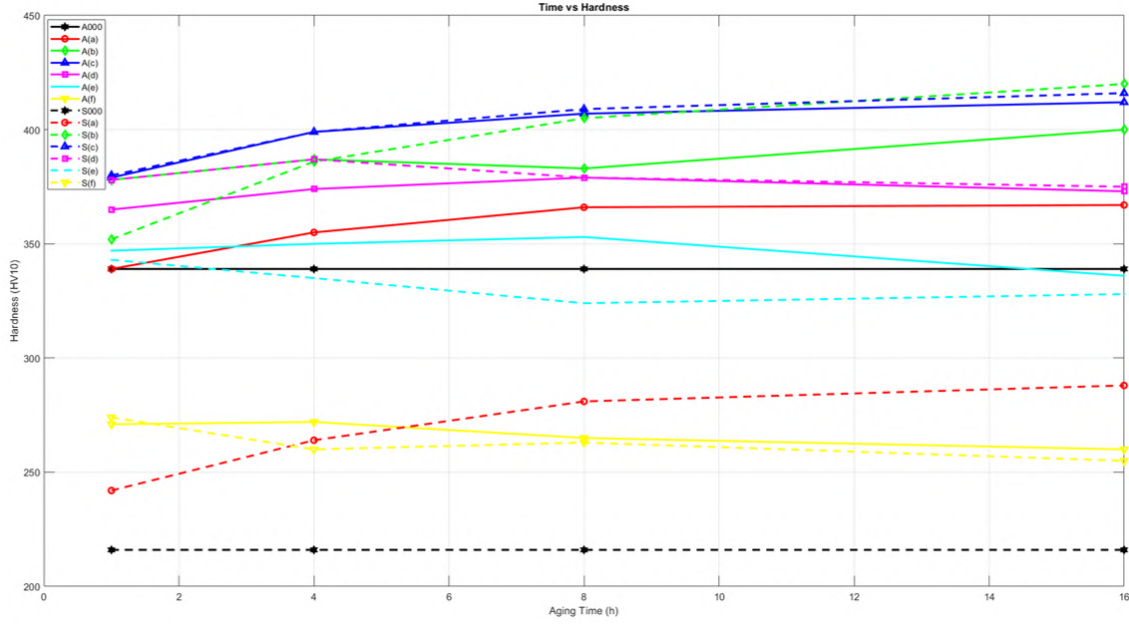


Figure 4.10: Hardness versus time for samples with different IHT conditions. Solid lines indicate samples with an as-received initial state, and dashed lines indicate samples with the solutionized initial state.

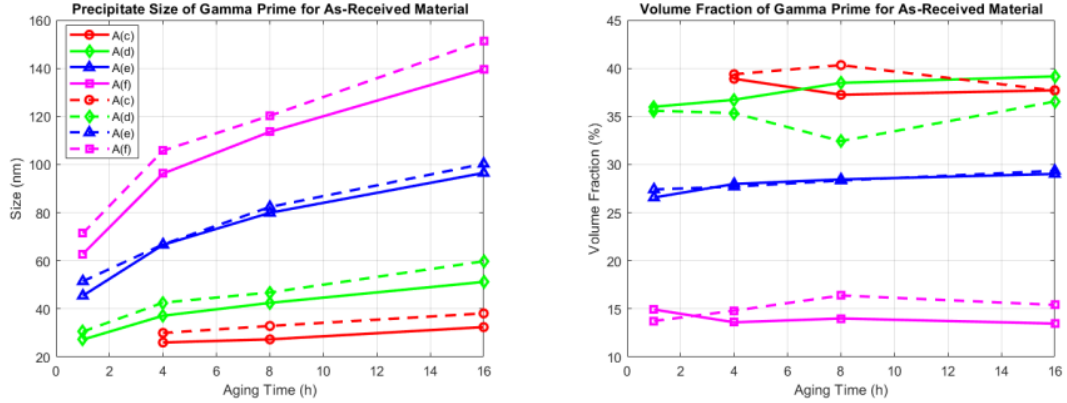
From Figure 4.10, it can be seen that the IHTs at (a) °C, (e) °C, and (f) °C for any IHT duration resulted in higher hardness values for the samples that have an as-received initial state compared to the samples that have solutionized initial states. However, for the IHTs at (b) °C, (c) °C and (d) °C, the samples with the initial state of solutionized showed greater values than the as-received ones. The highest hardness results are obtained from the solutionized samples subjected to IHTs at (b) and (c) °C for 8 and 16 hours. Further increasing the IHT temperature beyond these two temperatures resulted in decreasing hardness. Furthermore, below (d) °C, there is a trend of increasing hardness values with the increasing heat treatment duration. However, above this heat treatment temperature, the hardness values decrease after 1, 4, and 8 hours for IHTs at (f) °C, (e) °C and (d) °C, respectively. These findings indicate that at around (c) °C, the main hardening phase, γ' phase, is being optimized so that its precipitation size and volume fraction results in the highest hardening of the material. A more precise correlation between hardness and γ' phase is discussed after the results of the γ' precipitation size volume fraction are presented in Section 4.3.3.2.

4.3.3 Analysis of γ' phase

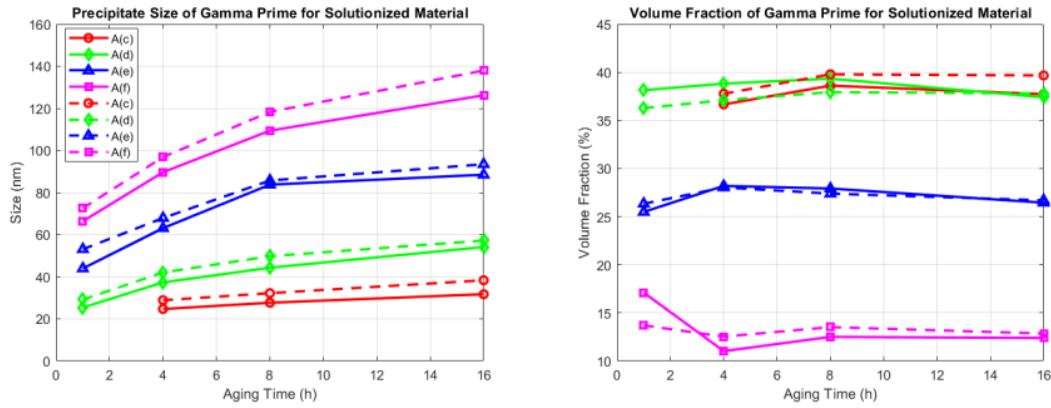
The SEM images were captured at magnifications of 20000x and 50000x and analyzed using MIPAR and ImageJ. The volume fraction and the size of the γ' precipitates were measured separately. The image analysis focused on two aspects: precipitation size measurement and the phase volume fraction at each IHT condition.

In Figures, 4.11 (a) to (d), the results of γ' precipitation size and volume fraction analysis are plotted. Figures 4.11 (a) and (b) are for the samples which have the initial material state of as-received, whereas Figures 4.11 (c) and (d) are for the samples which have the initial material state of solutionized. In both sets of plots, the x-axis represents the IHT duration in hours, and the y-axis represents the precipitation size in nanometer scale for the plots on the left and the volume fraction (%) for the plots on the right. For all plots, the red, green, blue, and purple colors constitute IHTs at the temperatures of (c) °C, (d) °C, (e) °C and (f) °C, sequentially. The solid lines represent the results obtained from the MIPAR analyses, while the dashed lines represent the ImageJ analysis results.

In Figures 4.11 (a) to (d), the analysis results starting from the IHT at (c) °C for 4 hours could be presented, while this analysis for any IHT at (a) °C and (b) °C as well as (c) °C for 1 hour could not be shown because the precipitate size and volume fraction in these conditions could not be analyzed. The precipitate sizes were very small, and the image resolution was not suitable due to the SEM resolution limitations on the very small precipitates, which led to further image processing producing undependable results.



(a) Variation of precipitate size with aging duration for as-received initial state. (b) Variation in volume fraction with aging duration for as-received initial state.



(c) Variation of precipitate size with aging duration for solutionized initial state. (d) Variation in volume fraction with aging duration for solutionized initial state.

Figure 4.11: Gamma prime analysis results visualized in line plots for precipitate size and volume fraction of γ' under different IHT conditions. Solid lines are representing the MIPAR results and dashed lines are for ImageJ results.

It is observed that as the IHT duration and the temperature increase, the precipitate sizes also increase, which is displayed in Figures 4.11 (a) and (c). The difference between the solid and dashed lines in these figures indicates that the image processing with ImageJ gives marginally larger precipitate sizes than MIPAR analysis. The precipitate sizes range from 25 nm at (c) °C IHT to 150 nm at (f) °C. The samples with the initial as-received material state show slightly but not significantly larger precipitate sizes compared to the solutionized initial state. Furthermore, a morphological development from sphere precipitates to almost cube-like precipitates can also be observed, as exemplified in Figure 4.12 as the IHT duration increases. The rest of the SEM images displaying the γ' precipitation varying to IHT temperature and time and to the initial state of the material can be found in the Appendix A.3.1

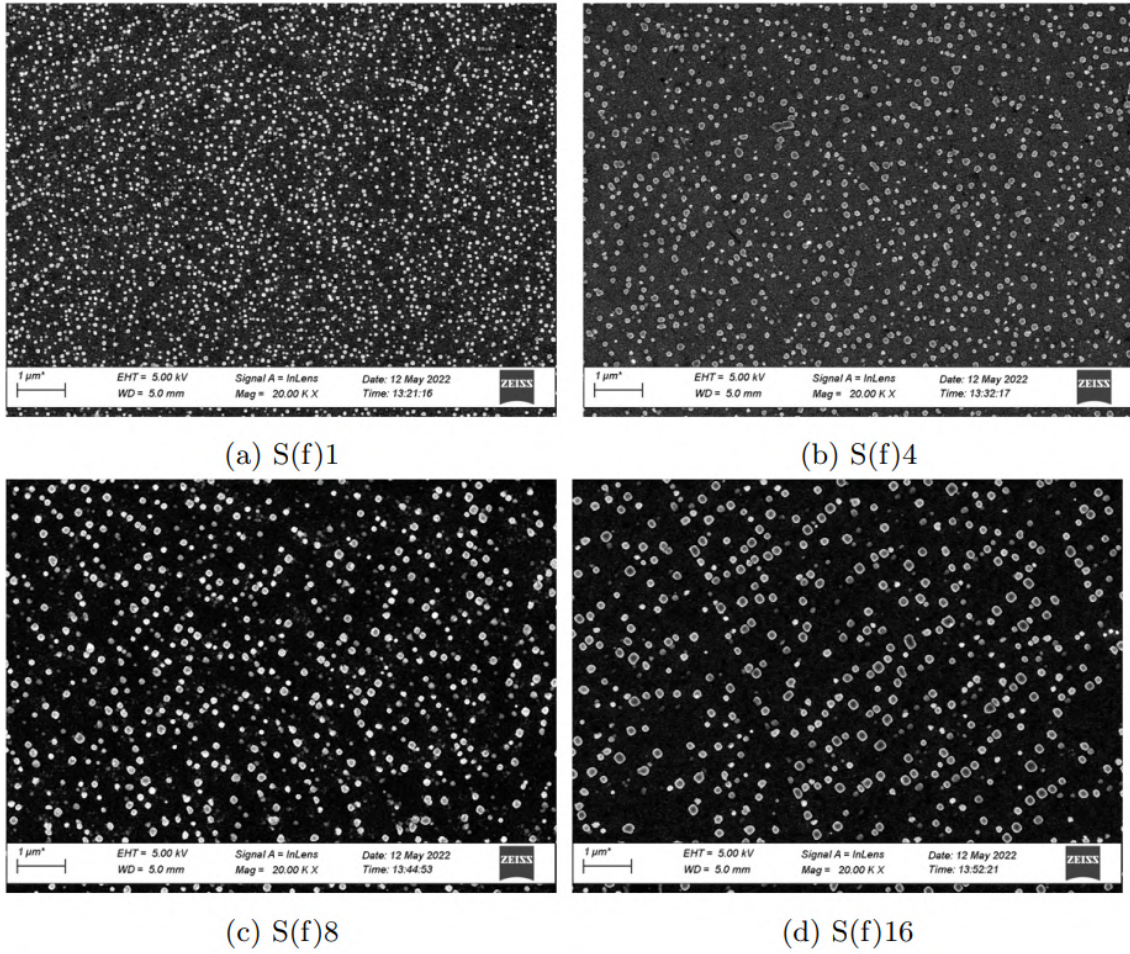


Figure 4.12: Observation of γ' -precipitates for solutionized and isothermally heat treated samples at (f) °C for 1-16 hours.

For the volume fraction analysis of the γ' phase, Figures 4.11 (b) and (d) are used. These figures demonstrate that an opposite trend to precipitate size is valid for the volume fraction as the IHT temperature increases. The volume fraction of both as-received and solutionized initial state samples is decreasing. For both as-received and solutionized initial state samples, the highest volume fraction of nearly 40% was obtained after IHT at (c) °C for 8 hours. Similar to the precipitation size analysis, MIPAR and ImageJ results are slightly different. This is assumed because, as the precipitate size gets smaller and the volume fraction gets larger, the identification and separation of features in the microstructure become more complicated for the software. Hence, this variation in the results is due to the software's capacity.

4.3.3.1 Comparison of Simulation and Experimental Results

The VDM 780 is a novel alloy; therefore, the database of the thermodynamic and kinetics simulation software might not be capable of giving reliable results that correlate with the experimental data. However, the results acquired from those sim-

ulations might still give a trend line that might match the experimental results. In Table 4.2, calculated at.% and wt.% of γ' phase results from simulations that were previously presented in Section 4.1.3 are compared with the experimental volume fractions as a result of image analyses.

Table 4.2: γ' - precipitate volume fraction comparison for simulation and experimental results.

Temperature (°C)	At% of γ' Phase (%)	Wt% of γ' Phase (%)	Volume Fraction of γ' for Solutionized Material (%)	Volume Fraction of γ' for As- Received Material (%)
(a)	31,39	36,5	-*	-
(b)	28,87	33,57	-	-
(c)	26,72	31,07	37,67	37,97
(d)	23,58	27,42	38,43	37,59
(e)	18,91	21,99	27,01	28,01
(f)	11,94	13,88	13,25	14,00

According to the IHT simulation results, the highest wt.% of γ' phase is obtained at (a) °C with 36,5%. According to the experimental results, the highest volume fraction for samples with a solutionized initial state is 38,43% at (d) °C. The highest volume fraction for samples with an as-received initial state is 37,97% at (c) °C. The experimental results for the volume fractions at (a) and (b) °C could not be obtained due to the previously mentioned problem with image analyses in Section 4.3.3.

This difference indicates that, as was predicted, JMatPro is not yet fully capable of predicting the actual phase transformation kinetics of VDM 780. Instead, the current simulation result is based on the general Ni-base alloys database. For the software to correctly predict the phases appearing on the equilibrium diagram and the phase evolution diagrams, the database created from the experimental results on the VDM 780 by studies such as this work should be introduced to the software's systems.

4.3.3.2 Relationship between γ' Precipitate Size, Volume Fraction and Hardness

The most important finding from this study has been the correlation between the γ' phase precipitate size, its volume fraction, and the hardness results of the corresponding IHTs. In order to visualize this correlation easier, the γ' precipitate size and volume fraction line plots that can be seen in Figure 4.11 in Section 4.3.3 are transformed into heat map plots by using the measurements obtained from MIPAR analyses. At the same time, the hardness measurement in Figure 4.10 in Section 4.3.2 is transformed into a heat map of Time-Temperature-Hardness (TTH). These new heat maps for isothermal heat treated samples with both initial conditions of as-received and solutionized are illustrated between Figures 4.13 and 4.18.

The heat maps for the γ' precipitate size and the volume fraction are presented in the Figures 4.13, 4.14, 4.16 and 4.17. In these figures, above the white dashed line shows the results of measurable data, and below this line, analysis results were

inconclusive. Hence measurements could not be presented. However, infinitesimally small values were still entered to substitute these inconclusive measurements to plot the heat map so that all heat maps, including TTH, have the same y-axis range, which is the IHT temperature, and an x-axis that is the duration of the IHT.

The TTH plot, as seen in Figures 4.15 and 4.18, shows that the highest hardness results are seen at the end of the IHTs at (c) °C for 8 hours and 16 hours. The 8-hour IHT at this temperature resulted in 406 HV for as-received and 409 HV for solutionized initial states. Whereas the 16 hours gave slightly higher results of 412 HV for as-received and 415 for solutionized initial states. Although increasing the duration from 8 hours to 16 hours results in a gain in the hardness values, this gain is minimal compared to the energy spent for eight more hours of heat treatment. As a result, IHT at (c) °C for 8 hours, as indicated by a blue circle in Figures 4.15 and 4.18, is determined to be the most efficient heat treatment for achieving desired hardness levels. But the question at this point is how does this relate to the γ' precipitate size and volume fractions.

In the Figures 4.13, 4.14, 4.16, and 4.17, the precipitate size and volume fraction measurement results that correspond to IHT at (c) °C for 8 hours are also marked with red circles. At this IHT condition, the precipitate size is measured to be 27 nm for both as-received and solutionized initial states, and the measured volume fractions are 37% and 39%, respectively. These values show that at this IHT condition, almost the smallest precipitate size is measured as this size only reaches 1,14% of the measurement ranges for the as-received and 2,93% for the solutionized initial states in their group whilst reaching nearly the highest volume fraction percentages with the as-received sample reaching 92,56% and solutionized sample reaching 97,42% of the analysed range group.

Samples with both initial states reached similar hardness values with the same IHT condition of (c) °C for 8 hours. However, the solutionized samples reached a higher result with 409 HV than the as-received 406 HV. The main reason for this increase in hardness is that, although both initially stated samples have the same precipitate size of 27 nm, the volume fraction for the solutionized sample is 2% more than the as-received sample. Ali et al., 2020 have shown that the size and volume fraction of γ' precipitates significantly impact the material's hardness and that, at a given constant precipitate size, the volume proportion of γ' precipitates directly affects hardness [32]. The concept of precipitation hardening can also explain this phenomenon. The precipitations strengthen the material, γ' precipitation in this case, as the dislocation traveling inside the γ matrix cannot enter the γ' phase without the formation anti-phase boundary (APB). This APB holds energy, representing a barrier that needs to be overcome if particle cutting occurs. Therefore, dislocations must travel through the γ/γ' structure in pairs, with a second dislocation removing the anti-phase boundary introduced by the first. This overcoming of the APB energy by the second dislocation indicates that substantial 'order strengthening' is expected [7]. Consequently, as the γ' precipitate size remains the same for both initial states, the higher volume fraction in the solutionized sample will require more energy to be applied to the sample to overcome the APB energy by the second dislocation. Hence, the hardness of the solutionized sample increases.

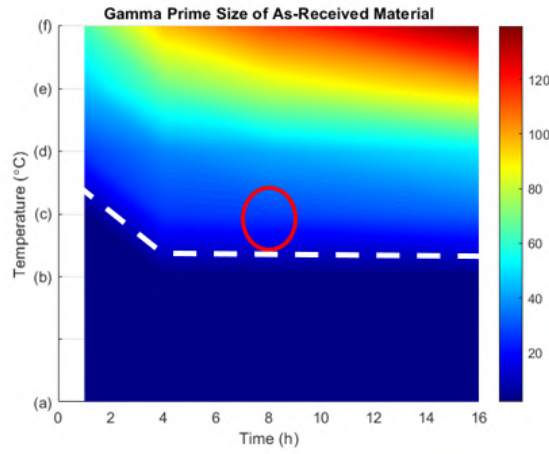


Figure 4.13: Heat map of γ' precipitate size for sample with initial state of as-received.

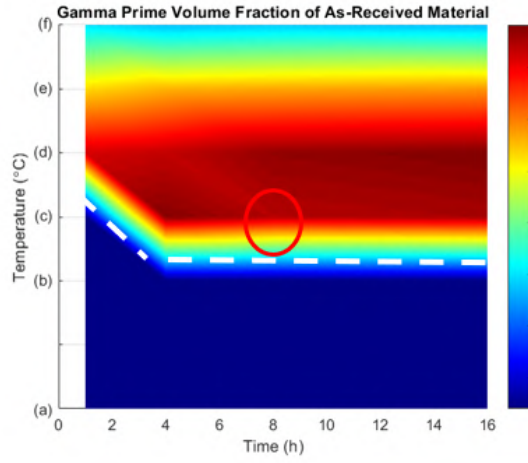


Figure 4.14: Heat map of γ' volume fraction for sample with initial state of as-received.

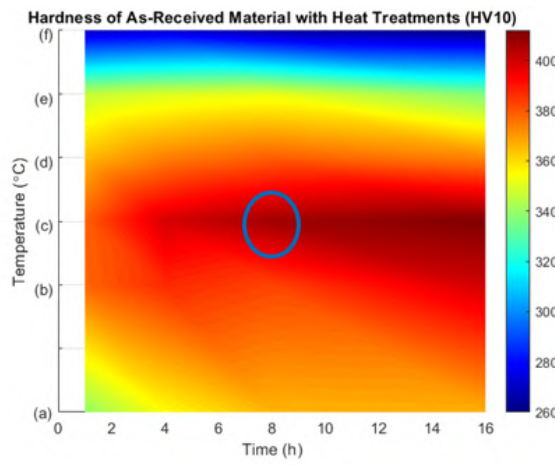


Figure 4.15: Heat map of TTH relation for sample with initial state of as-received.

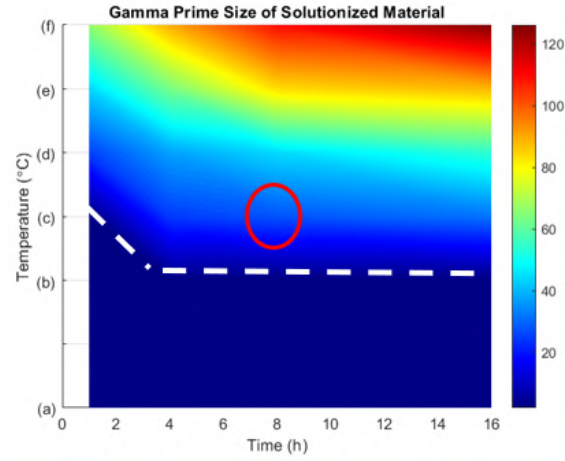


Figure 4.16: Heat map of γ' precipitate size for sample with initial state of solutionized.

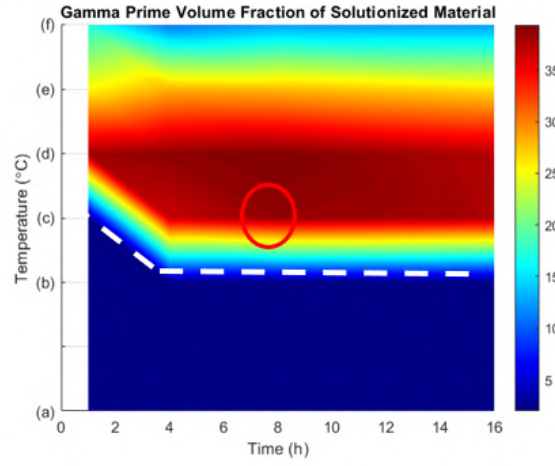


Figure 4.17: Heat map of γ' volume fraction for sample with initial state of solutionized.

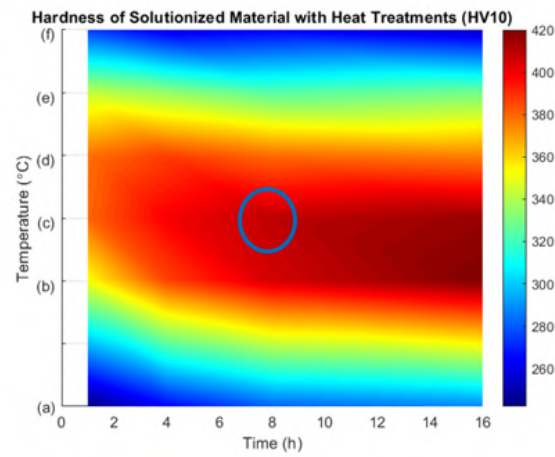


Figure 4.18: Heat map of TTH relation for sample with initial state of solutionized.

4.3.4 Qualitative Analysis on the High Temperature Phase

The importance of the high-temperature phase becomes evident after careful observation of the microstructure with LOM and SEM. In general, the microstructure of VDM 780, particularly the grain boundaries, lacks the important MC, M_6C , and $M_{23}C_6$ (where M stands for metallic elements) carbides found in Ni-base superalloys. Grain boundary carbides are essential because they can reduce or eliminate grain sliding effects, improving the alloy's creep properties [33]. As VDM 780 is lacking these carbide precipitations and, in addition to the high temperature phase, only the γ' precipitation is observed at the grain boundaries, as seen in Figure 4.19, which will not create enough resistance to prevent grain boundary sliding under stress, therefore the precipitation of the high temperature phase in a substantial amount is necessary.

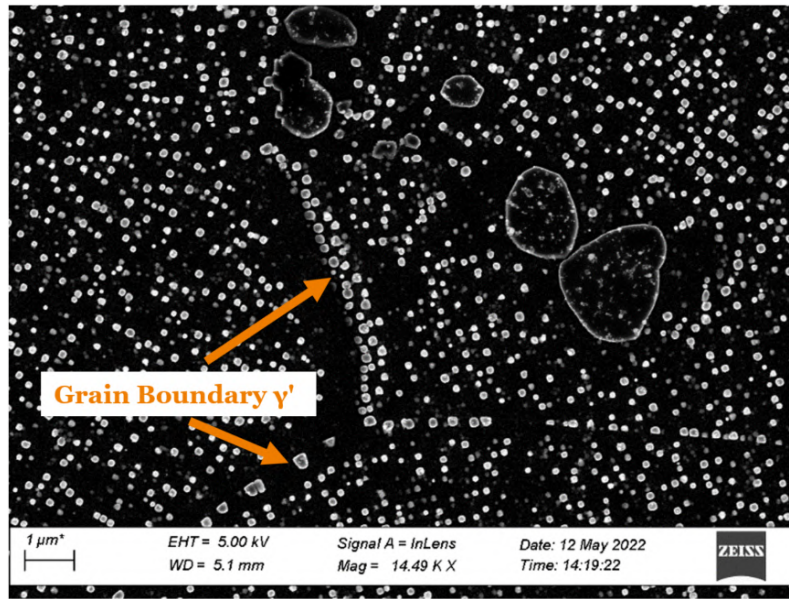


Figure 4.19: SEM image of grain boundary γ' in sample A(f)8.

The morphology of the high-temperature phase is studied from both LOM and SEM images in Figures 4.20, 4.21 and 4.22. The rest of the LOM images displaying the high-temperature phase can be found in the Appendix A.2.1. The precipitates' morphology appears needle-like primarily, but they also appear in small globular and almost triangle-like forms. The major identification method is the signature precipitation-free zone (PFZ) around this phase. In the next Section 4.3.5, EDS analysis on all phases will be presented. Its results will support that precipitates in this morphology are the precipitates of the high-temperature phase.

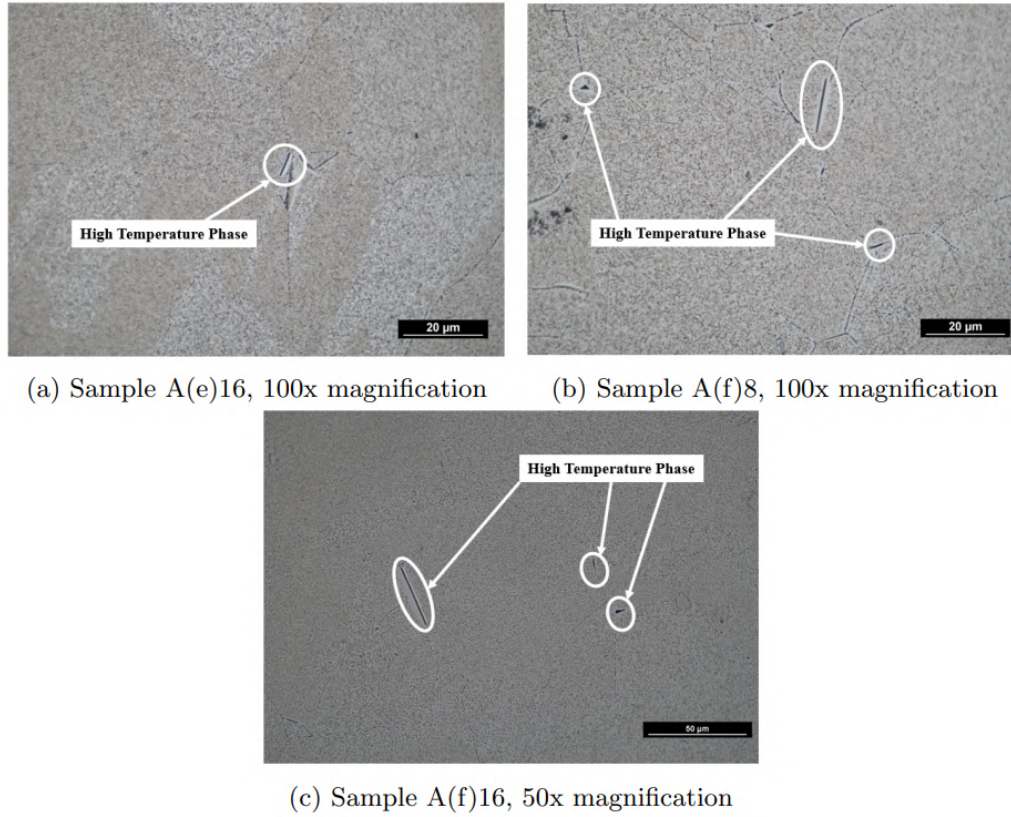


Figure 4.20: Observation of high-temperature phase with LOM in the as-received and heat-treated samples.

After the IHTs, the high-temperature phase was only observed for 8 and 16 hours of heat treatments at (e) °C and (f) °C. This requirement for the long heat treatment duration corresponds with the one obtained from the TTT diagram in Figure 4.2. However, the simulation in the TTT diagram predicted that transformation to the high-temperature phase denoted as the *delta* phase in that calculation should also occur at lower temperatures. It is previously indicated in Section 4.3.3.1 that the database of JMatPro is not yet ready to make exact predictions for VDM 780 but to eliminate the uncertainty for the high-temperature phase, the samples were etched twice. The first etchant, Oxalic acid, was well known to reveal the γ' phase by etching off the γ matrix, but no study showed that it revealed all the precipitates of the high-temperature phase. Therefore, Canadas etchant was utilized. The SEM images showing the precipitation of the high-temperature phase revealed with both etchants are seen in Figures 4.21 and 4.22. Although this new etchant visualized a comparable amount of high-temperature phase by removing the precipitates of the high temperature and γ' phases from the matrix, it didn't reveal any high-temperature phase in the IHT temperatures lower than (e) °C. Although quantification of this phase was not possible due to heterogeneous precipitation along the grain boundaries, IHTs at temperatures ranging from (e) °C to (f) °C for 8-16 hours were found to be necessary and the only option to obtain a sufficient amount of the high-temperature

phase precipitation in the microstructure.

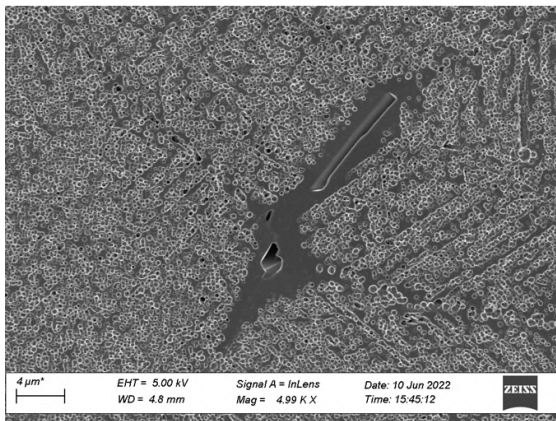


Figure 4.21: High-temperature phase in sample A(f)16 revealed with Canadas etchant.

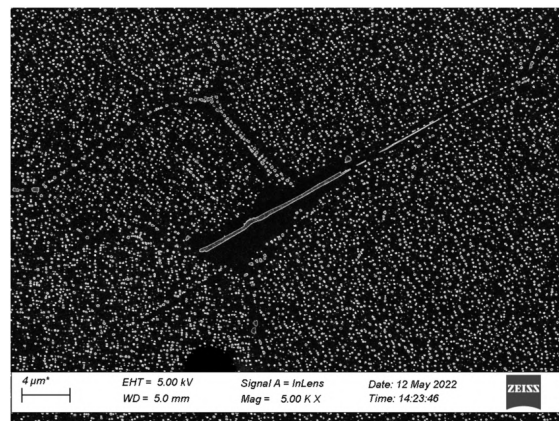


Figure 4.22: High-temperature phase in sample A(f)16 revealed by etching with Oxalic acid.

4.3.5 Elemental Analysis

Performing the elemental analysis was a necessary step of this thesis because without finding what elements constitute the precipitates being observed, confirmation regarding the phases cannot be done just by looking at their morphology. Therefore, EDS analyses were performed on the SEM images. This investigation was carried out in specific areas, shown in Figure 4.23 and the results of this analysis are shown in Table 4.3.

4. Results and Discussion

Table 4.3: Elemental composition of specific phases and VDM 780 composition.

	Element	C	O	Al	Si	Ti	Cr	Fe	Co	Ni	Cu	Nb	Mo	Total:	
MC Carbide	Spectrum 21	Wt%	17.15	0.64	0.20	0.03	3.20	1.62	0.00	1.64	2.97	0.17	71.74	0.66	100.00
γ' - phase	Spectrum 6	Wt%	1.19	0.48	4.67	0.08	2.67	4.12	0.65	13.90	59.98	0.00	11.51	0.74	100.00
High Temp. - phase	Spectrum 4	Wt%	1.11	0.33	0.77	0.10	1.27	2.39	0.00	22.21	45.33	0.00	24.86	1.63	100.00
Base (γ) Composition	Spectrum 19	Wt%	4.70	0.30	2.20	0.06	0.25	17.79	0.58	24.47	42.60	0.00	4.29	2.76	100.00
VDM Composition	Wt%	0.021	0.001	2.19	0.03	0.3	17.95	0.47	25.08	45.56	0.01	5.39	2.91		

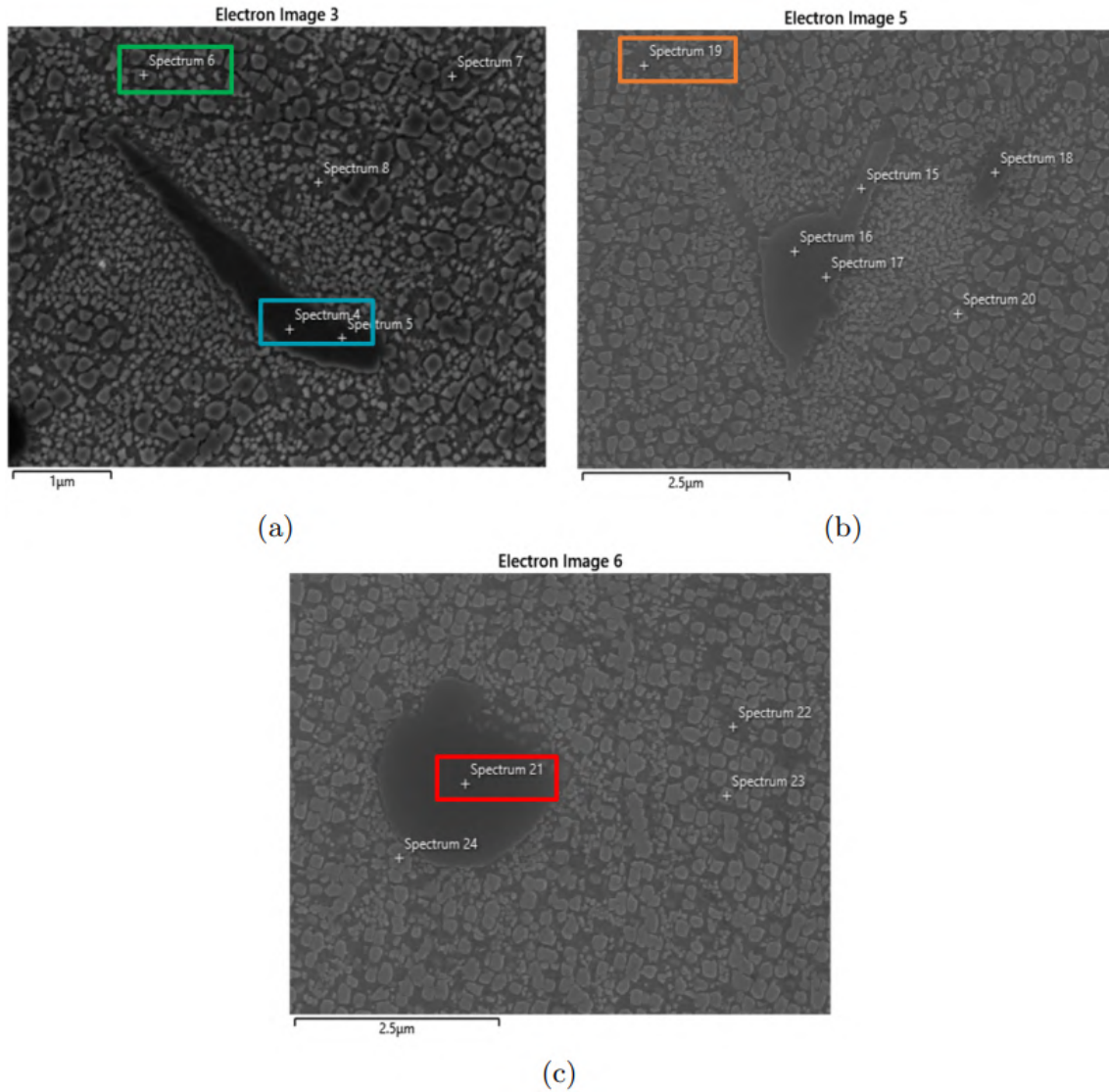


Figure 4.23: EDS analysis on different phases.

The concentration of the elements found in the specific spectrum clarifies which phases exist in the microstructure of VDM 780. First, it was previously mentioned that some carbide particles were observed inter and intragranular but in inadequate volumes. As the elemental study on Spectrum 21 in the Figure 4.23 shows a high concentration of carbon and niobium, and the shape of this carbide appears to be blocky and discrete, it can be confirmed that this is an Nb-rich MC carbide. Sec-

only, elemental composition at Spectrum 6 shows high concentrations of nickel and comparatively high concentrations of Al and Ti. This data, morphology, and high volume concentration of this precipitate prove that this is a γ' precipitate with a composition of $Ni_3(Ti, Al)$.

Furthermore, Spectrum 4 reveals that this precipitate has a medium concentration of titanium and high concentrations of cobalt, nickel, and niobium. This data matches the EDS analysis performed in previous studies [26,29], indicating that this precipitate is a high-temperature phase composed of δ , Ni_3Al , and η , Ni_3Ti phases. The last elemental analysis was performed on the matrix in Spectrum 19 to see if the composition of VDM 780 given in the material data sheet matches the EDS results. As the data shows in Table 4.3, the two compositions match almost entirely, except for the carbon concentration from Spectrum 19. The reason for this is presumably because of the carbon pick-up that is left from the sample preparation. With this analysis, the elemental study of the phases observed in the microstructure of VDM 780 is completed.

4.4 Standard Heat Treatment

The second objective of this study is to understand the STH of the VDM 780 alloy. This section will first present the results of the STH and afterward show the necessity of the long duration of STH by using the findings from Section 4.3.

4.4.1 Results of the Standard Heat Treatment

4.4.1.1 γ' Analysis

The STH cycle on the four samples, of which the conditions of these samples can be seen in Table 4.4, resulted in a bi-modal distribution of the γ' -precipitates. There are finer and coarser precipitates coexisting in the microstructure simultaneously. A significant difference is observed when the cooling method from the second stage of the heat treatment is changed from air cooling to furnace cooling, as seen in Figure 4.24 which is also shown in the study by Bergner et al. [28], which presents the same result.

Table 4.4: Sample descriptions for the standard heat treatment.

Sample	Starting Condition of Samples Prior the Heat Treatment	Cooling of Second Stage
1.1	As-Received	Air Cooling
1.2	As-Received	Furnace Cooling
2.1	Solutionized	Air Cooling
2.2	Solutionized	Furnace Cooling

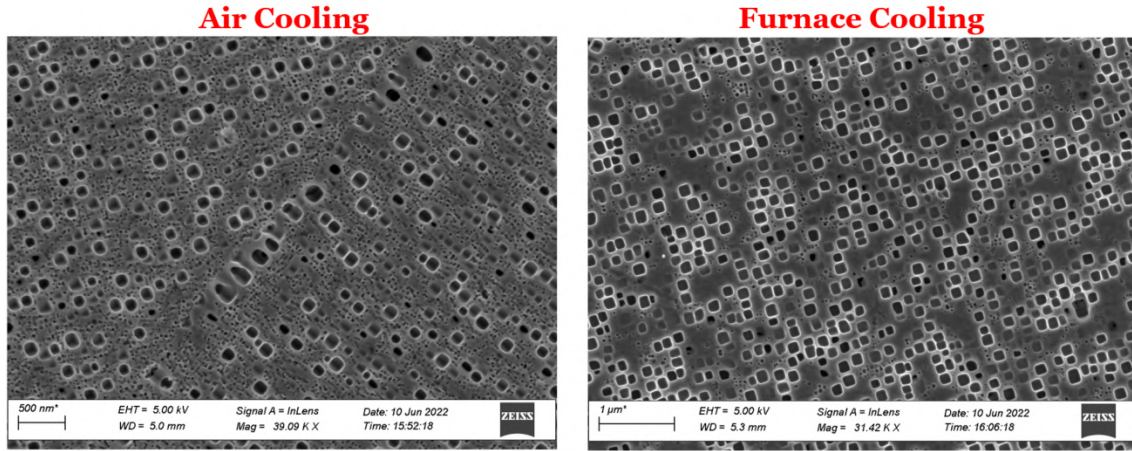


Figure 4.24: Resulting microstructure and the bi-modal γ' .

The quantification of γ' precipitate size and the volume fraction was performed with only ImageJ software, and the results are presented in the bar graphs seen in Figures 4.25 (a) and (b). Some of the SEM images that were used in the measurement for each sample can be seen in the Appendix A.3.2. In Figure 4.25 (a) the average size of the bi-modal γ' precipitation and (b) the average volume fraction of γ' phase are presented. In both figures, there are two sets of bars, one for the samples with the initial state of the as-received state and the other for the solutionized state. These states are again divided into two depending on the cooling method used in the STH's second step. The blue bars show the results of the air-cooled sample, and the orange bars show the results of furnace-cooled samples.

It is essential to consider that the size analysis gives the average size of the bi-modal distribution. Separate size analysis was not performed. Results indicate furnace cooling from the second stage of the heat treatment leads to a larger average size of the bi-modal γ' precipitation with around 78 nm for as-received and 71 nm for solutionized initial states, while air cooling results in an average of 45 nm for samples with both initial states.

The volume fraction analyses also give similar results, with a higher volume fraction observed for the furnace cooling. The highest volume fraction of γ' with approximately 55% in the sample with the initial state of as-received, while the solutionized sample's volume fraction is around 50%. The results from the air cooling give around 48% volume fractions for both initial conditions.

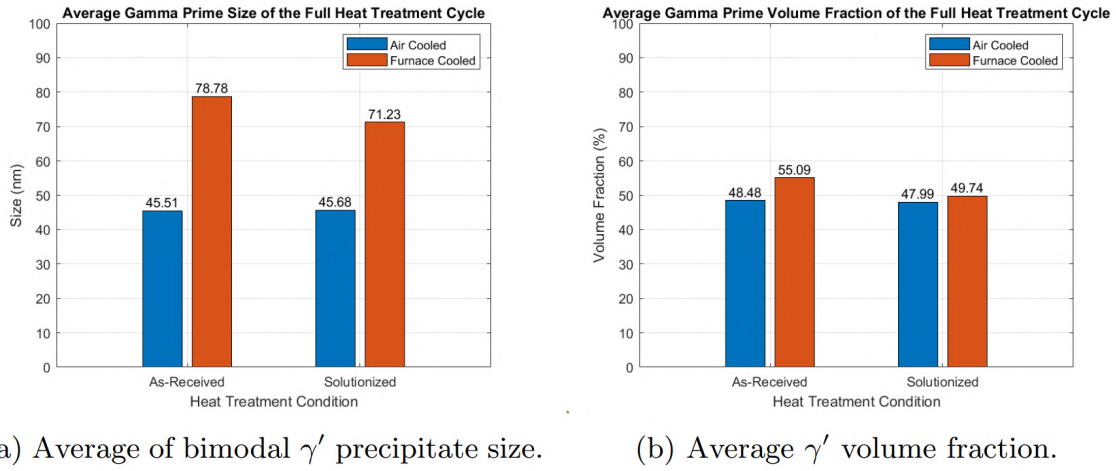


Figure 4.25: γ' size and volume fraction measurements for the standard heat treated samples.

4.4.1.2 Hardness Measurements

The hardness results of samples after the STH are presented in Figure 4.26 with the same plot setting as 4.25. Results indicate that both air and furnace cooling give high hardness values above 400 HV. Nevertheless, the air cooling leads to higher results than the furnace cooling, and the solutionized state gives the highest hardness results with 446 HV compared to the sample with an initial material state of as-received, which gives 432 HV with the air cooling. When the furnace cooling is applied after the second heat treatment stage, hardness measurement results in a hardness of around 402 HV regardless of the initial state.

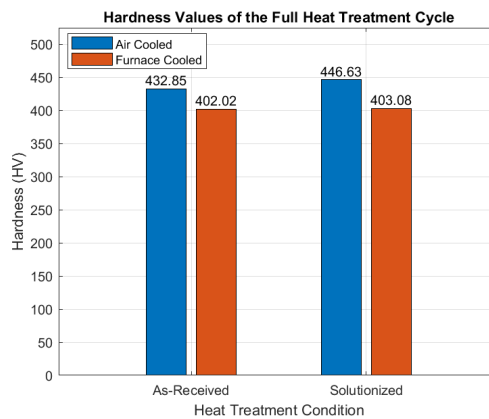


Figure 4.26: Hardness measurements of the standard heat treated samples.

4.4.2 Understanding the Necessity of Long Heat Treatment

VDM 780 has a reasonably long STH procedure, as represented again in Figure 4.27, with 28 hours of furnace heat treatment for just 1 sample without including the time required for cooling between the different stages. It is essential to fully comprehend and show the necessity of each heat treatment step in this STH by relating them to the findings of the microstructural evolution study under IHT conditions. Any future study that tries to develop a shorter standard heat treatment cycle should refer to this reference.

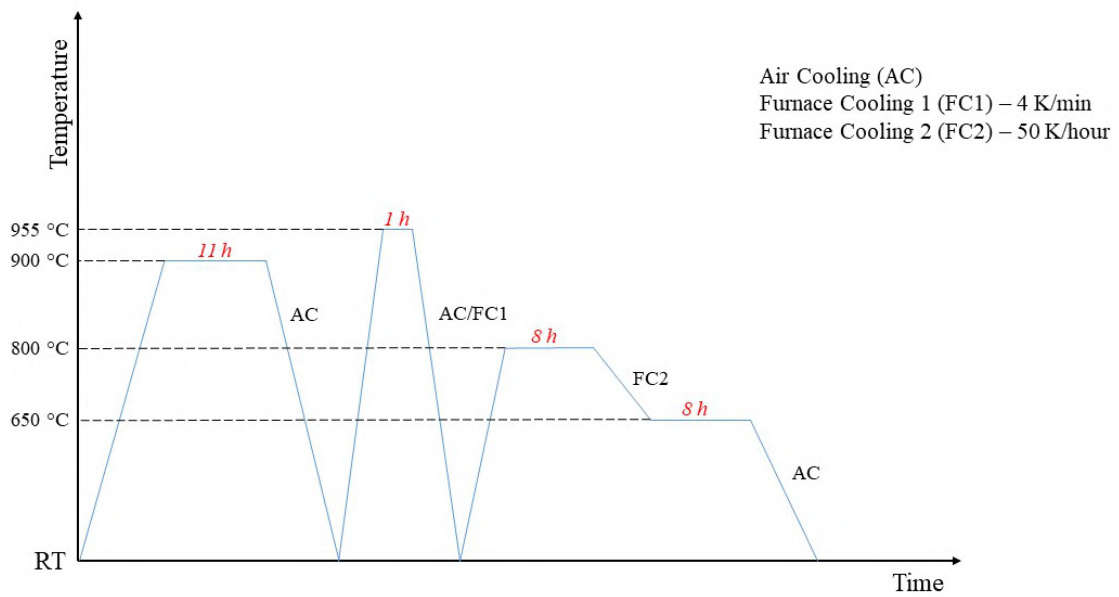


Figure 4.27: Schematic diagram of the standard heat treatment cycle in literature [28].

The first step is heat treatment at 900 °C for 11 hours. According to Section 4.3.4, long heat treatment of more than 8 hours at high temperature is required to precipitate the sluggish high-temperature phase. It is known that this phase will be critical to prevent grain growth in the remaining heat treatment steps and further in the application, hence, allowing fine grain forging. That is why precipitation of the sluggish high-temperature phase becomes the first step of this heat treatment procedure. The SEM and LOM images of the high-temperature phase observed after the STHs can be found in Appendix A.2.2 and A.3.3.

The second step is a one-hour heat treatment at 955 °C. Bergner et al., 2018 [28] suggest that this step ensures the dissolving of any remaining γ' precipitate in the microstructure. The γ' phase and hardness measurements after the IHTs in Section 4.3.3.2 showed that solutionizing the material prior to the IHTs resulted in a higher amount of γ' volume fraction and higher hardness values. The solutionizing temperature for the IHT study was selected to be at 1050 °C, but it was based on

the equilibrium diagram obtained from JMatPro, and now it is known that these simulation results are not accurate. Therefore, a heat treatment at 955 °C can still be above the solvus temperature of the γ' phase and can be enough to dissolve the remaining γ' precipitates in the matrix.

The third and the last steps are heat treatments at lower temperatures of 800 and 650 °C for 8 hours each. Third step is understood to be, correlating to the similar temperature results of Section 4.3.3.2, for the γ' phase precipitation in relatively small size and high volume fraction, leading to comparatively high hardness results. The fourth step is most likely to precipitate the secondary γ' precipitates that, in the end, create the bi-modal distribution of γ' precipitation throughout the observed microstructure in Figure 4.24. As a result of this bi-modal distribution, the hardness results showed around 30 HV higher measurements compared to the IHTs.

5

Conclusion

In this thesis microstructural evolution of VDM Alloy 780 under different isothermal heat treatment condition was performed and standard long heat treatment procedure is understood. Based in the results and analysis obtained in this work, the following conclusions can be made:

- Simulation tools are not giving accurate results to predict precipitation kinetics of VDM Alloy 780 yet, and this should be taken into account for any further study.
- Grain size of the starting material, in both as-received and solutionized initial state, was ASTM 4,5. The grain sizes could be measured until the IHT temperature of (c) °C, and the grain sizes remain unchanged for these measurements. However, the same cannot be confirmed for the heat-treated samples above this temperature.
- The hardness of samples subjected to the IHTs changed from 241 HV at (a) °C to 419 HV at (b) °C. The most efficient IHT to maximize the hardness value was at (c) °C for 8 hours because of the γ' precipitate size and volume fraction obtained in these isothermal conditions.
- The starting condition of the wrought material, either used in an as-received state or solutionizing prior to heat treatments, has an effect on the results after the heat treatment, as seen in the hardness and γ' characteristics observed. Solutionizing the material before starting the IHTs resulted in higher hardness values after the IHTs were completed compared to using the material in an as-received condition.
- Heat treatment of a minimum of 8 hours at higher temperatures, above the temperature of 900 °C is necessary to obtain a sufficient amount of high-temperature phase along the grain boundaries, which will inhibit grain growth and grain boundary sliding.

6

Future Work

This chapter suggests and points out the areas that can be explored in future studies on VDM Alloy 780 and how the works in this thesis work can be improved:

- The grain sizes could not be measured for all the isothermal heat-treated samples due to the etching effect in different samples. One can work on identifying the best performing etching recipe for this alloy and then measure the grain sizes for the remaining samples. The findings will most likely suggest how the grain size changes at higher heat treatment temperature since we expect to find the high-temperature phase there, and this phase's main role is to prevent grain growth, so there must be a variation.
- Since an understanding of the long standard heat treatment is formed, a future study can work on a shorter heat treatment cycle which can open the way to the industrialization of this alloy.
- In this thesis work, apart from the hardness measurement, no mechanical property is tested. Understanding the mechanical properties of this alloy with the isothermal phase evaluations can be performed in the future.
- VDM Alloy 780 is also produced in the powder version. Comparing the properties of wrought and AM products can be an area of interest.

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A

Appendix

In this section it is possible to find complementary information, including graphs, tables and images, in relation to different aspects mentioned in the report.

A.1 JMatPro Isothermal Heat Treatment Diagrams

This section displays the phase evolution diagrams for each IHT temperature.

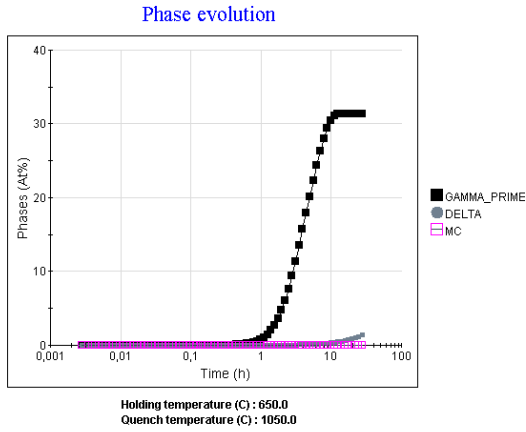


Figure A.1: IHT at (a) °C

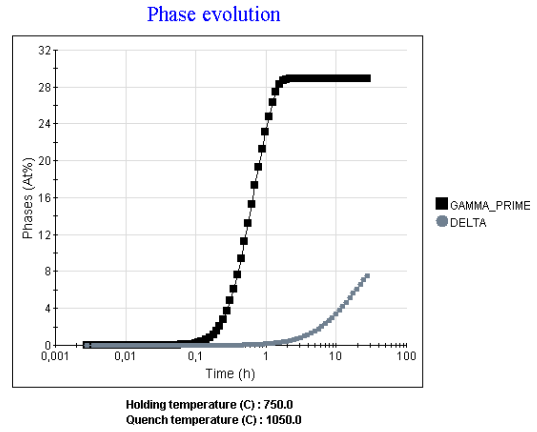


Figure A.2: IHT at (b) °C

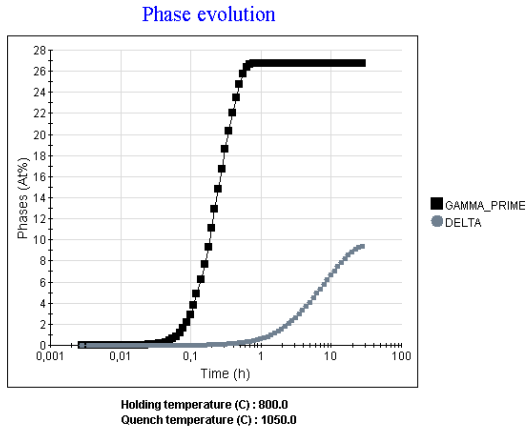


Figure A.3: IHT at (c) °C

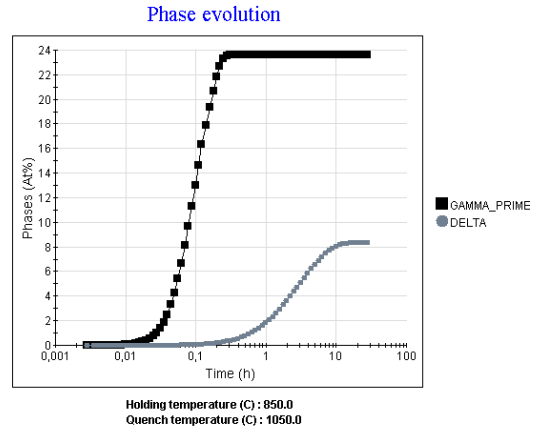


Figure A.4: IHT at (d) °C

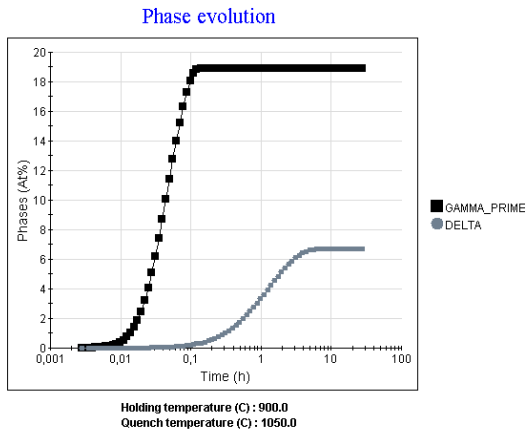


Figure A.5: IHT at (e) °C

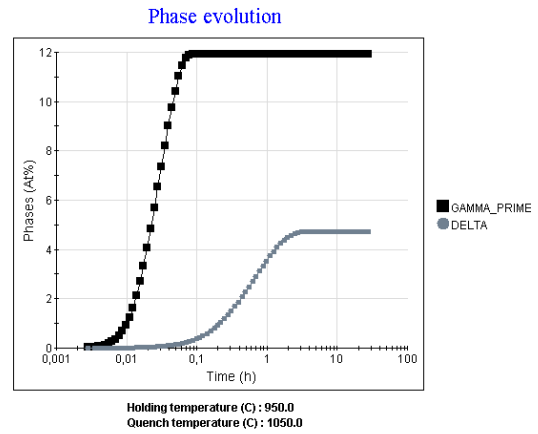


Figure A.6: IHT at (f) °C

A.2 Light Optical Microscopy Images

A.2.1 High Temperature Phase- IHTs

This section presents LOM images of samples where high temperature phase is visible after IHTs.

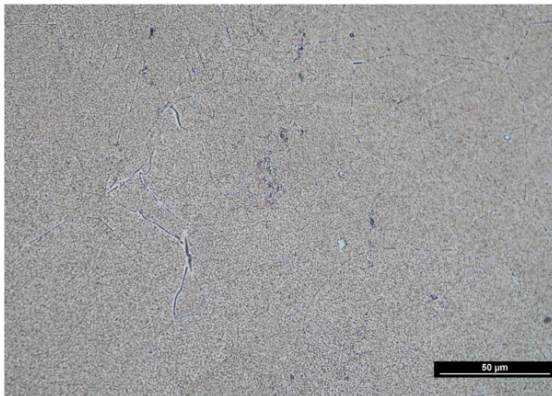


Figure A.7: A(f)8 - x50

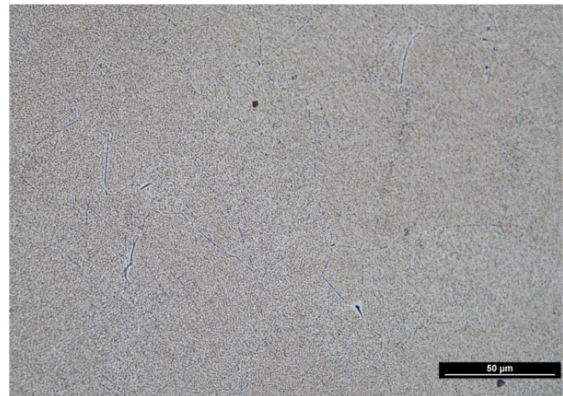


Figure A.8: A(e)16 - x50

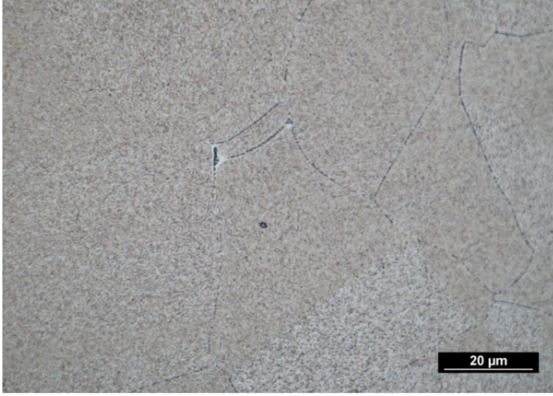


Figure A.9: A(e)16 - x100

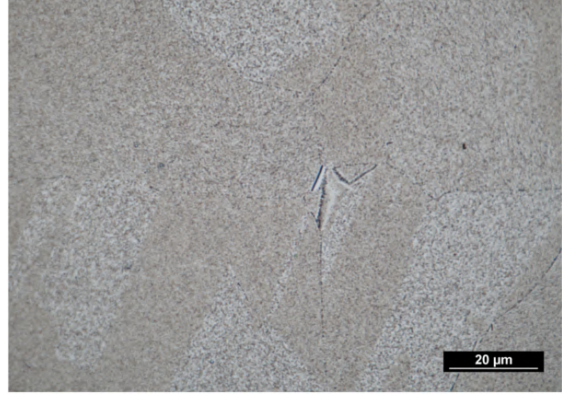


Figure A.10: A(e)16 - x100

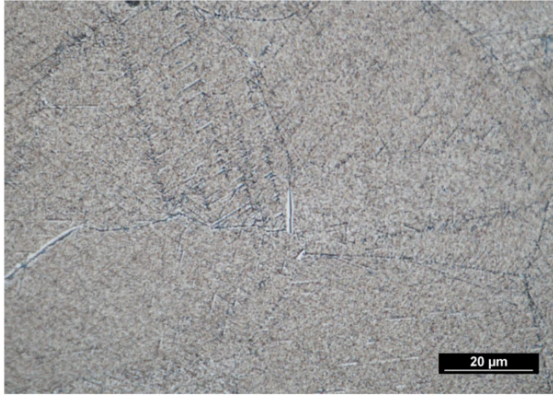


Figure A.11: S(e)16 - x100

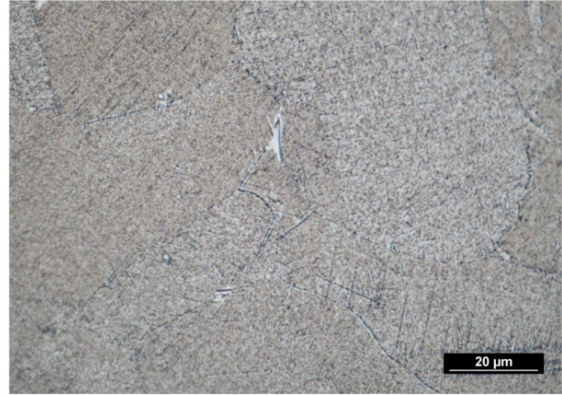


Figure A.12: S(e)16 - x100

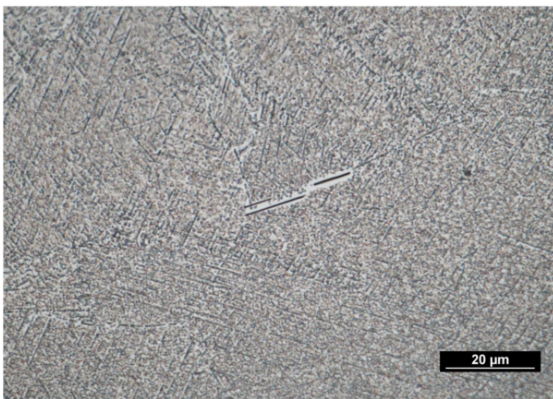


Figure A.13: S(f)16 - x100

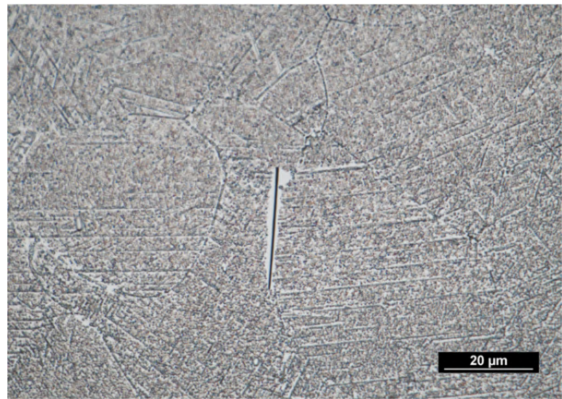


Figure A.14: S(f)16 - x100

A.2.2 High Temperature Phase- SHTs

In this section OM images where high temperature phase can be seen along the grain boundaries are presented. The images are only for the samples 1.1 and 1.2.

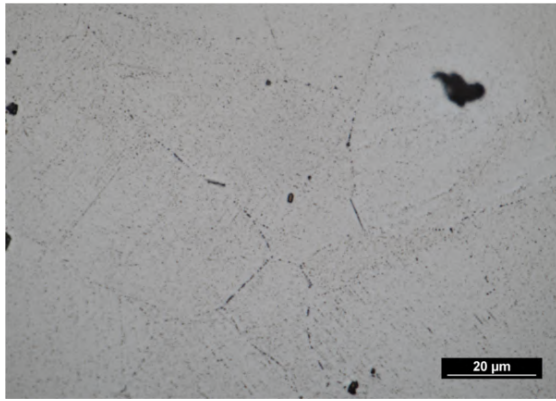


Figure A.15: Sample 1.1 - x100

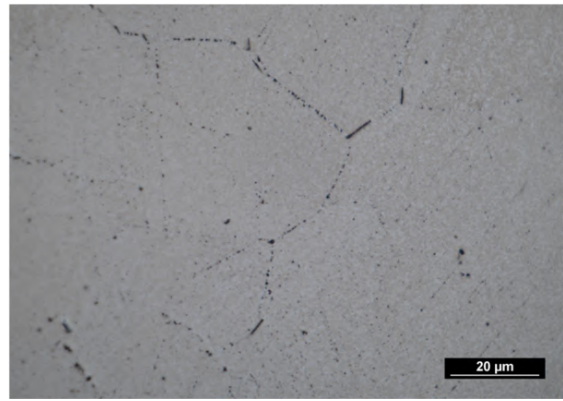


Figure A.16: Sample 1.2 - x100

A.3 Scanning Electron Microscopy Images

A.3.1 Isothermal Heat Treatments

This section puts forward the SEM images of samples subjected to IHTs. Each figure presents 8 images at one IHT temperature. These images vary according to the heat treatment duration, on x-axis, and the initial state of the material prior to heat treatments, on y-axis.

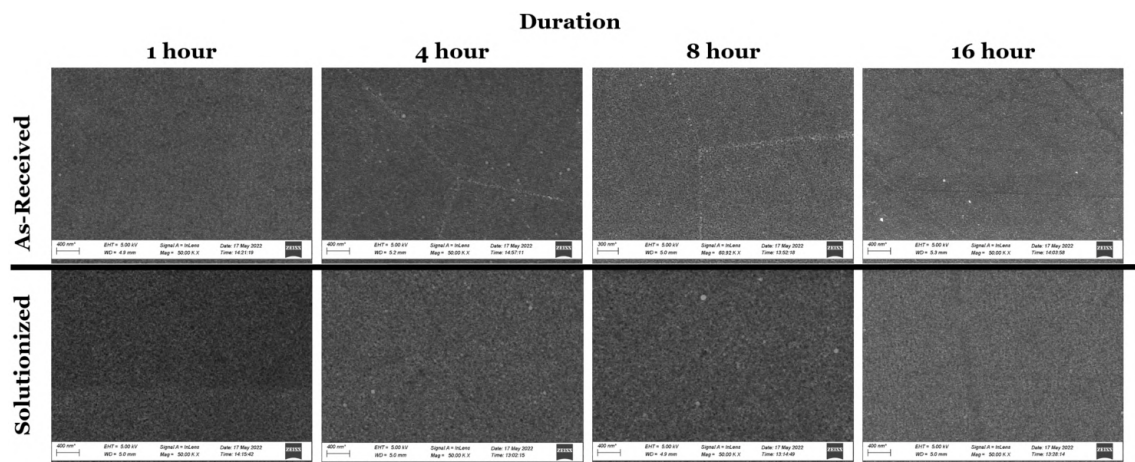


Figure A.17: SEM images of IHT at (a) °C.

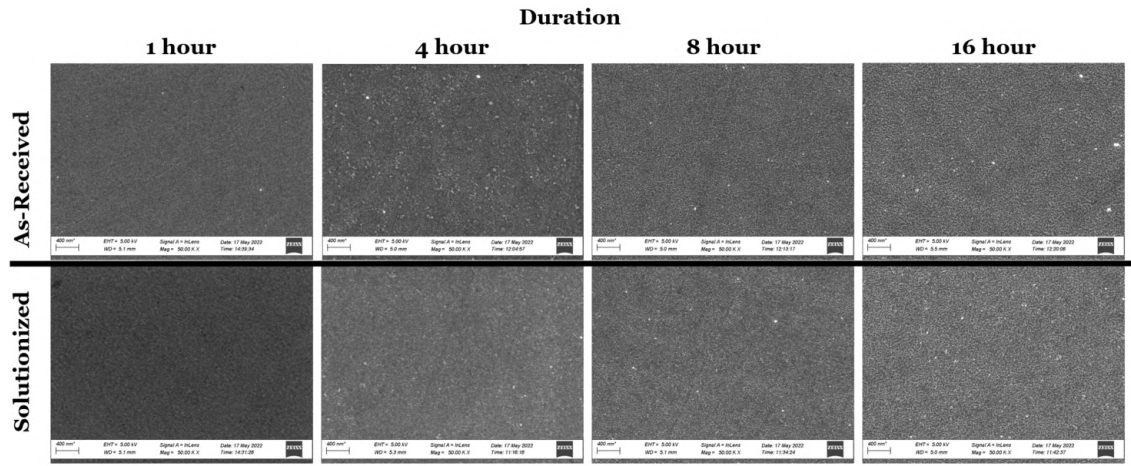


Figure A.18: SEM images of IHT at (b) °C.

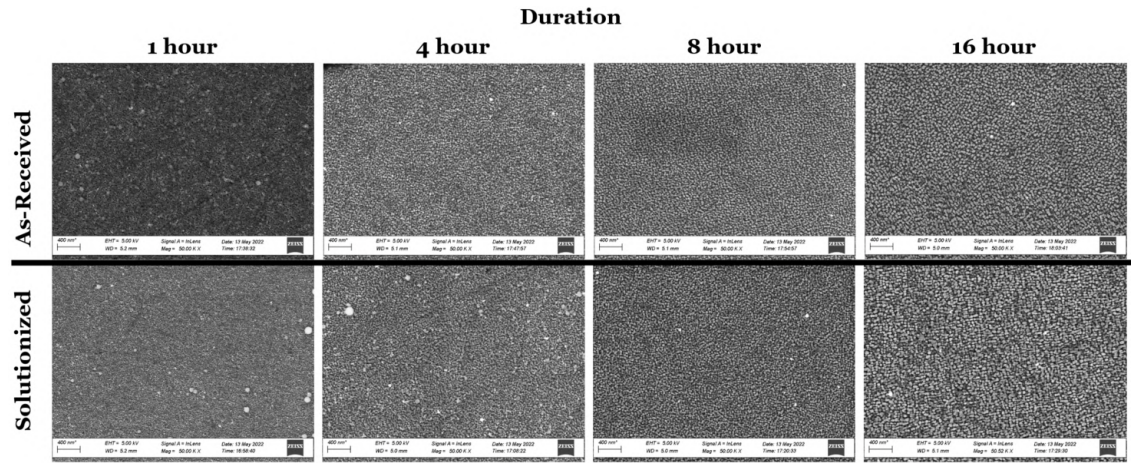


Figure A.19: SEM images of IHT at (c) °C.

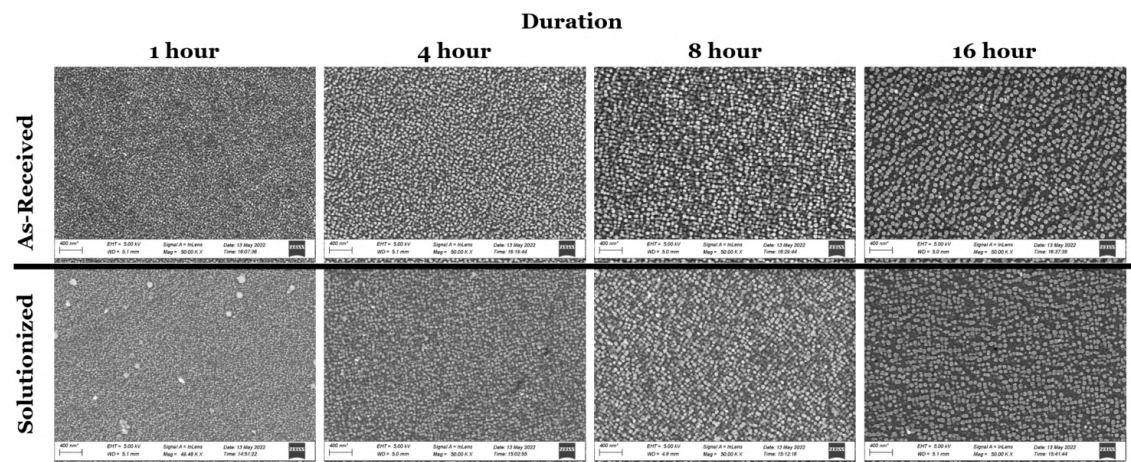


Figure A.20: SEM images of IHT at (d) °C.

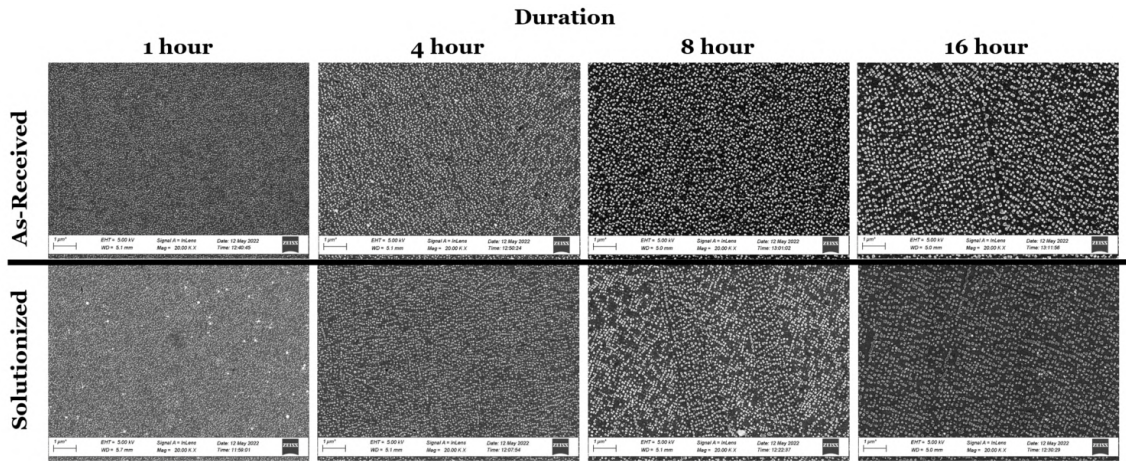


Figure A.21: SEM images of IHT at (e) °C.

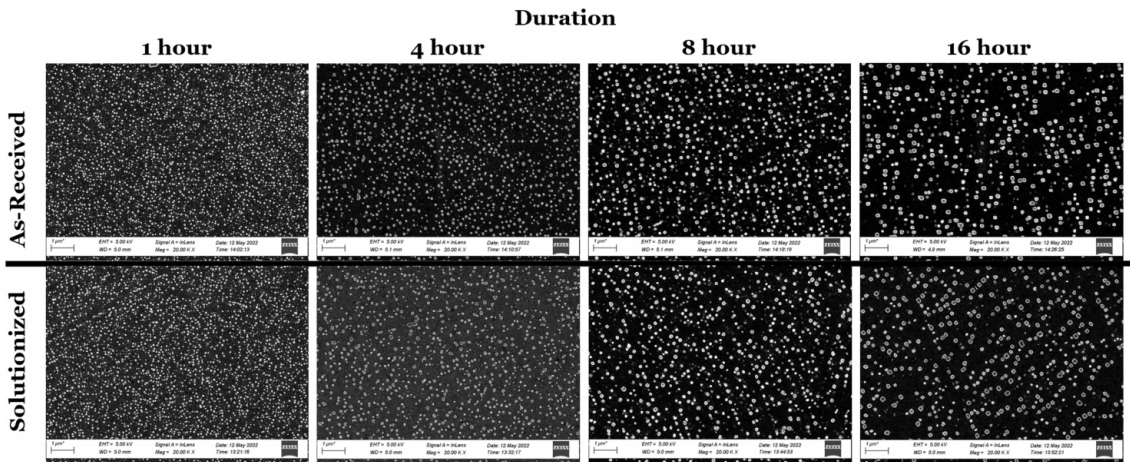


Figure A.22: SEM images of IHT at (f) °C.

A.3.2 Standard Heat Treatment

This section shows one SEM image from each STH condition that was specified in Table 4.4.

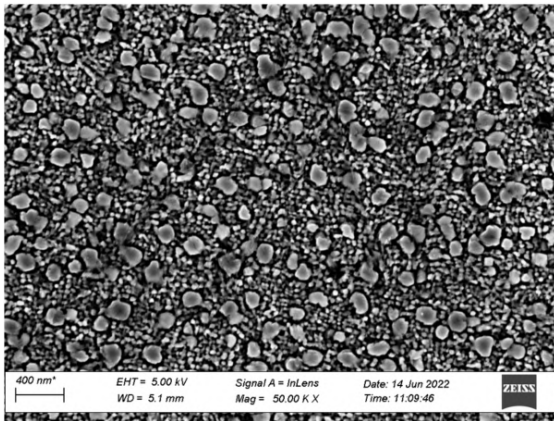


Figure A.23: Sample 1.1 - x50000

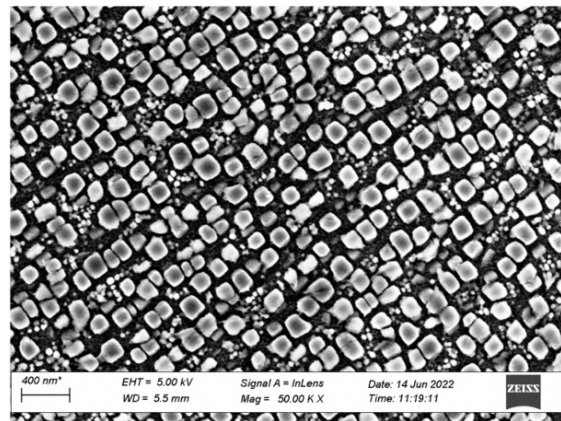


Figure A.24: Sample 1.2 - x50000

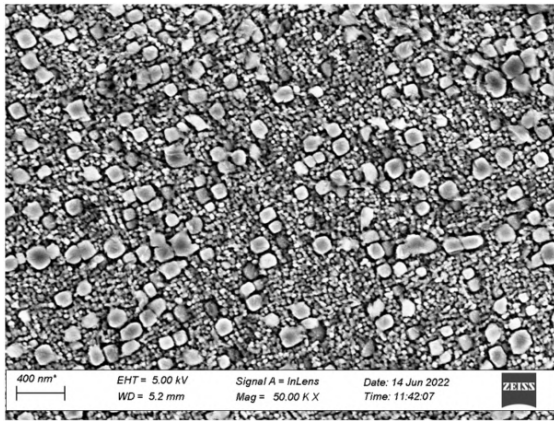


Figure A.25: Sample 1.1 - x50000

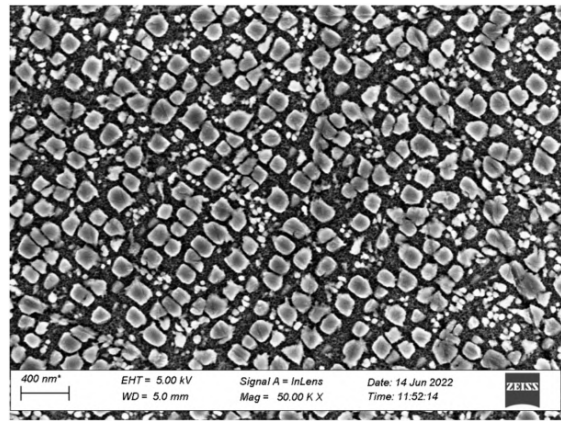


Figure A.26: Sample 1.2 - x50000

A.3.3 High Temperature Phase- SHTs

This section displays some of the SEM images showing precipitation of high temperature phase in the samples that were subjected to the standard heat treatments.

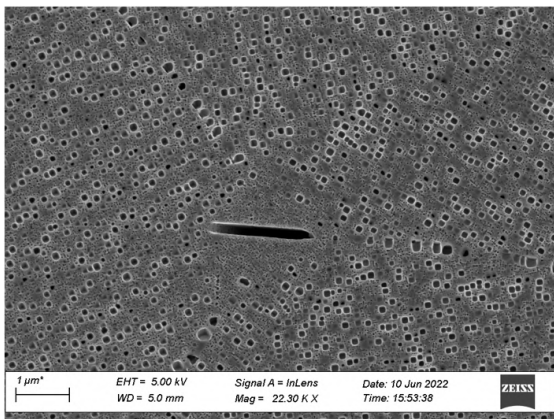


Figure A.27: Sample 1.1 - x22000

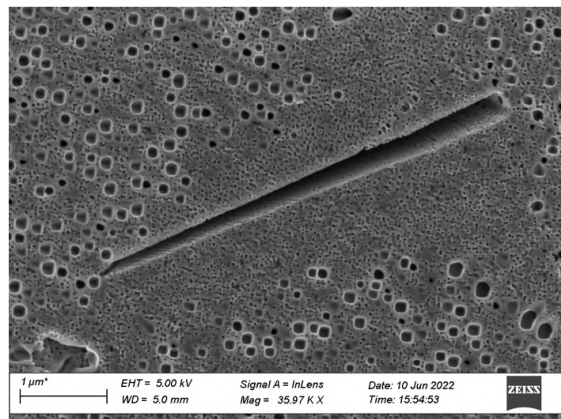


Figure A.28: Sample 1.1 - x35000

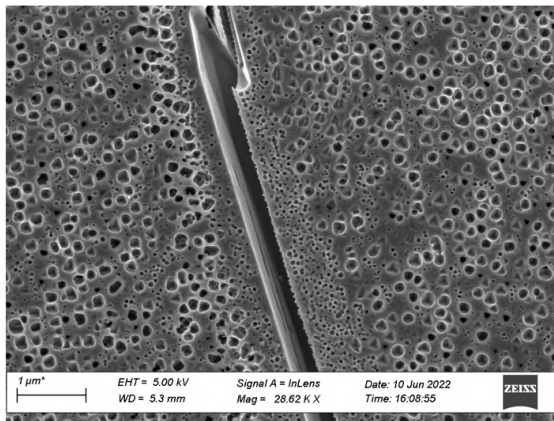


Figure A.29: Sample 1.2 - x29000

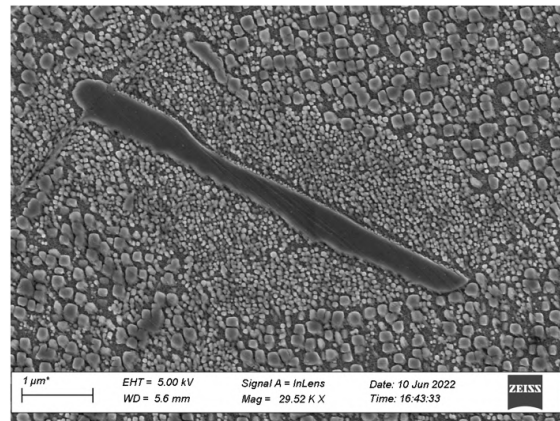


Figure A.30: Sample 2.2 - x30000

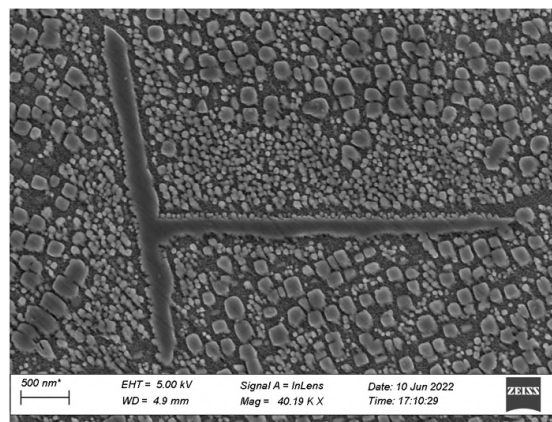


Figure A.31: Sample 2.2 - x40000

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