University of West Attica
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Diploma Thesis

Metallographic analysis of the microstructure of a high – Mn plastic mold tool steel after heat treatment alternatives

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Διπλωματική εργασία

Μεταλλογραφική ανάλυση μικροδομής μετά από εναλλακτικές μεθόδους σκλήρυνσης εργαλειοχάλυβα για καλούπια πλαστικού υψηλού Mn

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Η διπλωματική εργασία εξετάστηκε επιτυχώς από την κάτωθι εξεταστική επιτροπή

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Table of Contents

1. Introduction

1.0 Abstract

The present thesis is about studying the heat treatment alternatives on the Uddeholm Nimax tool as far as steel microstructure and the hardness after each heat treatment is concerned. The

microstructure and the chemical composition of the steel will show the results of each heat treatment alternative (subzero heat treatment, carburizing) and if possible, examine possibilities to improve its mechanical properties. The specific tool steel grade has hardness limitations if treated to normal heat treatments. Hardness and its depth, going to be measured, the microstructure is going to observed with the chemical composition alterations in order to decide if it's possible to increase hardness through the heat treatments alternatives. The equipment and the procedure that is going to be used is according on ASTM international standards.

1.1 General aspects

Iron and later on the steel was developed around 4000 years ago in the iron age. Iron started the journey as tool and weapons, and it evolved through the ages. It wasn't long after when people notice that if Iron and carbon were combined, they made a stronger metal, the steel.

Robert Forester Mushet, a British metallurgist, in 1868 was the first that developed the first tool steel that got recognized by the scientific community. It wasn't long after Mushet improved the Bessemer steelmaking process by adding more elements (manganese) in the steel where the mechanical properties were improving. [1]

After that, Mushet started experimenting with various ways to find ways to improve the properties of steels. He started noticing that when different elements (tungsten, manganese) were adding in the steel the hardenability was improving by just cooling in air and even without the need of water or even oil quench. [2]

Mushet steel led to many more important developments. Many scientists used his work to even improve it my adding more elements in the steel as well as by using different ways to heat treat it trying to maximize the desired properties.

Tool steels and heat-treating processes are used all around the world. The applications for tool steels can be found almost everywhere and by using a heat-treating process can improve every characteristic of the steel according to the application.
More advanced heat-treatment methods started to thrive in the mid 19th century when the scientists started to find ways to cool objects below the temperature of the environment.
In 1872, Sir James Dewar invented the vacuum flask. The liquid air was first created in 1883 by the Polish scientist named Olszewski. A few years later Olszewski and a British scientist Sir James Dewar made the world first volume of liquid hydrogen, which begins the modern cryogenic application. [3]

Even though tool steels are a small part of the massive family of steels, they have more strict and complicated manufacturing procedures than the normal unalloyed steels because of the high level quality and homogenity that is required. Heat treatment can change the microstructure of the steels and increase the mechanical properties . There are various techniques of heat treatment for different applications as there are various types of steels. Not all steels can be heat treated and those who can, can't be treated by the same way.

Organizations around the world are research and develop more efficient ways in order to involve the technology of the heat-treating methods of steels. Few organizations that are active in this fiend are:

- *The International Federation for Heat Treatment and Surface Engineering (IFHTSE)*
- *Heat Treating society asm international (HTS)*

- *The Association for the Heat Treatment of Metals (ATZK)*
- *Global Heat Treatment Network*
- *American Society for Metals(ASM)*

1.2 State of the art

HEAT TREATMENT OF METALS

Heat treating processes can play a vital role in the application of steels and that's why almost every tool steel has gone through a heat-treating process in order to achieve the desired properties. But, to understand the benefits of these methods and how they can aid the development of the technology of metallic materials, the categories of heat treatment methods and the advantages that they offer should be referred.

The heat treatment method is an operation of heating and cooling a metal or alloy steel in solid form and with that operation the microstructure of the metal can change and also change the properties of the metal without changing the chemical composition.

The heat treatment processes can be categorized into two main types: surface heat treatment and bulk heat treatment. The differences between these two methods are the following:

1: Surface Heat Treatment:

Surface heat treatment focuses on altering the properties of the outer layer or surface of a metal component while leaving the core unaffected.

The primary objective of surface heat treatment is to improve specific surface-related properties such as hardness, wear resistance, and corrosion resistance.

Surface heat treatment methods include:

o   Case Hardening: This process involves adding carbon or nitrogen to the surface layer of the metal to create a hard, wear-resistant case while maintaining a tough core. Common techniques include carburizing, nitriding, and carbonitriding.
o   Induction Hardening: It utilizes electromagnetic induction to selectively heat the surface of the metal component and then rapidly quench it. This results in a hardened surface layer with a softer core, providing a combination of hardness and toughness.
o   Flame Hardening: In this method, the surface of the metal is heated using a high-temperature flame and then quenched. It is typically used for localized hardening of specific areas.
o   Laser Hardening: A laser beam is used to heat and rapidly cool the surface of the metal, creating a hardened layer. It offers precise control over the heat-affected zone and is often used for complex geometries.

2: Bulk Heat Treatment:

Bulk heat treatment involves subjecting the entire metal component to heating and cooling processes to modify its overall microstructure and properties throughout its volume.

The purpose of bulk heat treatment is to achieve desired properties such as improved strength, hardness, toughness, and dimensional stability.

Bulk heat treatment methods include:

- Annealing: It involves heating the metal to a specific temperature and then slowly cooling it to relieve internal stresses, refine the microstructure, and improve machinability and ductility.
- Normalizing: Similar to annealing, normalizing involves heating the metal to a specific temperature, followed by air cooling. It refines the grain structure, improves mechanical properties, and enhances uniformity.
- Quenching and Tempering: This two-step process consists of rapid cooling (quenching) the metal to achieve a hardened structure, followed by tempering to reduce brittleness and improve toughness while retaining a desirable level of hardness.
- Solution Heat Treatment: It is primarily used for alloys, where the metal is heated to a specific temperature to dissolve elements in a solid solution, followed by rapid cooling to retain the desired properties. Precipitation Hardening: This treatment involves a sequence of heating, quenching, and aging to promote the formation of fine precipitates, resulting in increased strength and hardness in certain alloys. [4],[5]

It's important to note that the specific heat treatment method chosen depends on factors such as the desired properties, material composition, component geometry, and intended application.

Heat Treatment of Metals :Advantages:

- Improved Mechanical Properties: Heat treatment can significantly enhance the mechanical properties of metals, including increasing their strength, hardness, toughness, and ductility. This leads to improved performance and durability of the material. [6][7]
- Altered Microstructure: Heat treatment processes can modify the microstructure of metals, such as refining the grain size, improving grain distribution, and aligning grain boundaries. This results in improved material properties, such as increased strength and reduced brittleness. [6][7]
- Tailored Material Properties: Different heat treatment techniques, such as quenching and tempering, can be utilized to achieve specific material properties required for particular applications. This allows for customization and optimization of the material to meet specific requirements. [8][9]
- Stress Relief: Heat treatment can effectively relieve internal stresses within the metal that may have developed during manufacturing processes, such as casting, forging, or welding. This helps to minimize the risk of deformation or failure under service conditions. [8][9]
- Improved Machinability: Certain heat treatment processes, such as annealing and normalizing, can improve the machinability of metals by enhancing their cutting characteristics, reducing tool wear, and improving surface finish. [7][8]

Heat Treatment of Metals: Disadvantages

- Distortion: Heat treatment can induce distortion in the shape and dimensions of the metal part due to non-uniform heating and cooling rates. This can require additional machining or corrective measures to achieve the desired final dimensions. [8][9]

- Potential for Cracking: Rapid cooling during quenching, especially in large or complex-shaped components, can increase the risk of crack formation or distortion. Proper quenching techniques and control are necessary to minimize these issues. [8][9]
- Reduced Dimensional Stability: Some heat treatment processes may cause dimensional changes in the metal, leading to challenges in maintaining tight tolerances and fitment requirements. This requires careful consideration and compensatory measures during the design and manufacturing stages. [8][9]
- Increased Material Cost: Heat treatment adds an additional step to the manufacturing process, requiring energy, specialized equipment, and skilled personnel. This can increase the overall cost of the metal component compared to non-heat-treated alternatives. [10][11]
- Limited Applicability: Not all metals and alloys can be effectively heat treated due to their chemical composition or specific metallurgical constraints. Factors such as alloy alloy composition, phase stability, and the presence of certain elements limit the range of materials suitable for heat treatment. [10][11]

CRYOGENIC TREATMENT

During the past few years, Universities and Industry studied the cryogenic treatment techniques for improving tool steel properties that heat treatment or other methods couldn't achieve. During these studies, different steel grades are chosen to be researched and based on their

characteristics a number of responses is acquired. Depending on the process applied, the material properties, maybe improved including hardness, toughness, wear resistance, relief of residual stresses and transformation of retained austenite.

Cooling materials to sub-zero temperatures have the advantage of phenomena such as superconductivity that can only occur at low temperatures that used on cryogenic treatment. Other phenomena is during these extreme cold temperatures the free energy of the crystal structure reduced and create more orderly structure.

Cryogenic treatment has been applied to a wide range of materials (mostly metals). The results of this treatment provide plenty of uses.

Cryogenic treatment is widely accepted within the metallurgical profession and engineers, as a treatment that can maximize or improve many of the materials properties. Nevertheless, cryogenic treatment should not be regarded as the best way of treatment for all materials and in all circumstances do not yield the best results. [12]

SHALLOW CRYOGENIC TREATMENT

Shallow cryogenic treatment (SCT) of steels is a sub-zero process that occurs after quenching and before tempering either impart or enhance specific conditions or properties of the material. The benefits of the shallow cryogenic treatment are among others increased strength, better dimensional or microstructural stability, relief of the residual stress and wear resistance increase.

In general hardening and complete transformation from austenite to martensite is desired prior to tempering but 100% transformation rarely occurs.

Retained austenite tends to be present in varying amounts depending on the chemical composition and the heat treatment process.

Some applications require materials such as tool steels, carburized alloy steels and high-carbon martensitic stainless steels for which the retained austenite may result in excessive wear and may cause cracking and/or dimensional changes that can promote failure.

Shallow cryogenic treatment can improve wear resistance in such materials under specific circumstances.

For maximum transformation to martensite, a proper design of heat treatment is required. Considering the part, the chemical composition, the shape and size as well as the application, another treatment may be more practical and improve more properties.

Minimize the risk of cracking due to thermal warpage or cracking cause of the increase volume of the surface or even sharp corners and abrupt changes in sections can create stress concentration and promote cracking.

There are advantages of the (SCT), unlike heat treatment of austenitizing which requires the temperature to be controlled precisely in order to avoid reversal or unsuccessful transformation of the phases, the SCT depends only on the attainment of the minimum low temperature. Lower temperature may affect the transformation, but additional chilling cause any reversal phenomena.

However, cooling rate of a heated piece have a definite influence on the part. Depending on the cooling rate, austenite can transform to bainite or cementite instead of martensite.

Another note of caution is the dimensional changes caused by the difference of temperatures and may contribute to part failure.

The equipment for SCT can be a deep freezer with temperature approximately at -18°C. Dry ice surface is -78°C but the part that is inside of a container can approach -60°C. Mechanical refrigeration units with circulating air approximately -87°C to -95°C can also be an option. [13] [14]

DEEP CRYOGENIC TREATMENT

The Deep Cryogenic process in differentiated from Shallow Treatment. It uses lower temperatures than ahallow treatment, it requires different cooling rate and its application to materials other than steel.

Deep Cryogenic Treatment (DCT) can increase wear resistance, several times related to those achived by Shallow Treatment with hardened steels.

Use of the process onother metals and alloys produces related affects as with steel. The results of the process in ferrous metals include reduced retained austenite, relief of residual stresses, the precipitation of fine carbides, increased wear resistance, hardness dimensional stability, fatigue life, corrosion resistance and electrical and thermal conductivity.

One considerable difference from Shallow Treatment is that Deep Cryogenic processing requires a slow drop of temperature in order to gain all benefits of the process. The cooling rate is slow trying to avoid high temperature gradients that can create harmful stresses, allowing time for the crystal lattice structure to accommodate the changes that occurring and to avoid thermal shock that can lead to part failure.

Tempering after DCT is performed for the same reasons as to the conventionally heat treated materials.

Deep Cryogenic temperatures are the temperatures under -150°C (-238°F or 123K). usually temperatures used in Deep Cryogenic Treatment are generally -185°C but it can also include temperatures down to -268°C. Cooling media fo this process is liquid nitrogen, liquid helium, liquid hydrogen and neon.[13] [14]

TEMPERING

Cryogenic Treatment is not the final stage of the Heat Treatment process. Since successive stress relieving or tempering is absolutely necessary to attain the requisite stress relieved martensite.

Tempering may reduce hardness, but also it reduces residual stress, increases ductility and toughness and provides dimensional stability. [15]

MARTENSITIC TRANFORMATION

The martensitic structure is a unique crystalline structure that can form in certain metals during a specific type of heat treatment known as martensitic transformation. It gives metals an exceptional level of hardness and strength and is characterized by a highly distorted lattice. The martensitic structure of metals and its essential features will be discussed in this conversation. [16]

Definition and Formation:

- o The martensitic structure is a metastable phase that forms when certain metals are relatively rapid cooled from a high temperature to below a critical point, called the martensite start temperature (Ms).
- o The transformation occurs due to the rearrangement of atoms, resulting in a distorted lattice structure without diffusion or phase changes.
- o Martensitic transformation is commonly observed in iron-based alloys (e.g., carbon
- o steels and stainless steels), as well as some non-ferrous alloys (e.g., titanium alloys). [16]

Martensitic Transformation:

- Martensitic transformation is a solid-state phase change that occurs in certain metals during rapid cooling from a high temperature to below the martensite start temperature (Ms).
- The transformation involves a rearrangement of atoms without diffusion, resulting in a distorted lattice structure known as martensite.
- The martensitic start temperature (Ms) is the temperature at which the transformation begins, while the martensitic finish temperature (Mf) is the temperature at which the transformation is completed. [16]

Martensitic Start and Finish:

- The martensitic start temperature (Ms) is influenced by various factors, including alloy composition, cooling rate, and carbon content. Higher carbon content increased alloy composition and faster cooling rates generally lead to lower Ms temperatures.
- The martensitic finish temperature (Mf) is the temperature at which the transformation is complete. It is typically lower than the martensitic start temperature.
- The difference between the Ms and Mf temperatures is known as the martensite transformation temperature range and can affect the resulting microstructure and properties. [16]

Key Characteristics of Martensitic Structure:

- o Needle-Like or Plate-Like Structure: The martensitic structure typically exhibits a needle-like or plate-like morphology. These structures are formed due to the rapid and non-uniform rearrangement of atoms during the transformation.
- o High Hardness: Martensite is known for its exceptional hardness, making it desirable for applications where wear resistance and strength are critical. The hardness arises from the distorted lattice structure and high carbon content in some alloys. [16]

Benefits of Martensitic Structures:

- High Hardness: Martensitic structures have a high degree of hardness, which makes them ideal for applications requiring wear resistance and surface durability.
- Increased Strength: Martensitic alloys offer high strength due to the distortion of the lattice structure during transformation, providing excellent load-bearing capabilities.

- Improved Wear Resistance: The combination of hardness and strength in martensitic structures enhances their resistance to wear, minimizing surface damage and extending the component's lifespan.
- Dimensional Stability: Due to their low susceptibility to thermal expansion and contraction, martensitic structures offer dimensional stability, making them appropriate for applications with stringent dimensional requirements.
- Magnetic Properties: In ferrous alloys, martensitic structures can exhibit ferromagnetic characteristics, making them suitable for use in applications requiring magnetic response or magnetic field generation. [16]

Factors Affecting Martensitic Transformation:

- Composition: The chemical composition of the alloy, particularly the carbon content in steels, greatly influences the formation and stability of martensite. Higher carbon content generally promotes martensitic transformation.
- Cooling Rate: The cooling rate plays a crucial role in martensitic transformation. Rapid cooling rates are required to prevent atom diffusion and achieve the necessary distortion of the lattice. Quenching in oil, water, or air is commonly employed to achieve high cooling rates. [16]

Austenite Stability: Martensitic transformation occurs from a specific parent phase called austenite. The stability of austenite is influenced by alloy composition, temperature, and cooling rate. Higher carbon content and lower Ms temperatures encourage martensite formation.

Applications of Martensitic Structure:

- Cutting Tools: Martensitic steels are widely used in the manufacturing of cutting tools such as knives, blades, and saws due to their high hardness and wear resistance.
- Automotive and Aerospace Components: Due to their combination of strength, corrosion resistance, and magnetic characteristics, martensitic stainless steels are used in the automotive and aerospace industries.
- Springs and Fasteners: The high strength and hardness of martensitic alloys make them suitable for use in springs, fasteners, and other mechanical components that require excellent strength and dimensional stability.
- Structural Components: In particular applications, the unique combination of strength, hardness, and magnetic properties offered by martensitic structures can be advantageous, such as in magnetic resonance imaging (MRI) equipment. [16]

It's important to note that the martensitic structure can also exhibit some challenges, including potential brittleness, susceptibility to hydrogen embrittlement, and dimensional changes due to thermal expansion or contraction. Proper understanding of these characteristics is crucial for the successful application of martensitic alloys.

TOOL STEELS

High-strength alloy steels known as "tool steels" are specifically made for use in dies, molds, cutting, drilling, and shaping tools. The aerospace, automotive, and construction industries, as well as businesses engaged in manufacturing and metalworking, are just a few of the areas in which they

are used. Tool steels are used because they have superior cutting, shaping, and forming capabilities, which have substantially increased the efficiency and productivity of various industries.

High hardness, wear resistance, and toughness are the defining characteristics of tool steels, which make them the perfect choice for use in demanding applications requiring accuracy and sturdiness. In order to attain specified qualities and performance characteristics, the chemical composition, microstructure, and heat treatment of tool steels are meticulously tuned. [17] [18]

The hardness, wear resistance, and toughness of tool steels are significantly influenced by the amount of carbon in the steel. Low-carbon tool steels are utilized in applications that call for excellent toughness and impact resistance and typically contain less than 0.6% carbon. Applications that call for strong wear resistance and moderate hardness use medium-carbon tool steels, which have a carbon content of 0.6% to 1.4%. Applications that require high hardness and wear resistance use high-carbon tool steels, which contain more than 1.4% carbon. [17]

Chromium, molybdenum, tungsten, vanadium, and cobalt are further alloying substances frequently found in tool steels. The steel is given these additions to increase its tensile strength, toughness, resistance to wear, and heat resistance. The type of tool steel and its unique qualities depend on the concentration of these components and how they combine with carbon. [19]

Due to their tendency to harden after cooling in air, air-hardening tool steels, such as A2 and D2, are easier to work with and less likely to deform after heat treatment. These steels often have higher concentrations of chromium and tungsten, which enhances their superior toughness and wear resistance. Cutting tools and dies are two examples of applications that frequently use air-hardened tool steels for good wear resistance. [20]

The microstructure of tool steels is a key factor in determining their qualities and performance traits, in addition to their chemical make up. To attain the necessary hardness, toughness, and wear resistance in tool steels, heat treatment is commonly used. The steel passes through a number of phase transitions during heat treatment, creating a particular microstructure as a result. [21]

The martensitic transformation, which transforms austenite into martensite, is one significant phase change that takes place during heat treatment. Tool steels' high hardness and wear resistance are a result of martensite, a particularly brittle and hard phase. The capacity of tool steels to generate hard surfaces can be improved by the addition of alloying components like manganese. [22]

PLASTIC MOLD TOOL STEELS

Steels made especially for use in the production of plastic components and goods are known as plastic mold tool steels. For these types of steel to withstand high pressures and temperatures involved in the plastic injection molding process, they need to have high wear resistance, and

hardness as well as good dimensional stability. [23]   .

Pre-hardened steels like AISI P20 and P21, which are frequently referred to as low-alloy tool steels, are the most widely used for plastic molds. These steels have high hardenability, good dimensional stability, and good machinability. They are excellent materials to employ in plastic injection molds because they have outstanding thermal conductivity and wear resistance. [24]

Powder metallurgy (PM) steels can be employed for applications that call for even more wear resistance and hardness. These steels are created by combining tiny tool steel particles with a binder and then sintering the combination under high pressure and temperature. PM steels are ideal for high-volume plastic molding applications because of their outstanding performance in terms of wear resistance, toughness, and dimensional stability.[25]

In general, plastic mold tool steels are necessary for producing plastic components and goods. The type of tool steel used will depend on the particular requirements of each application; among the most popular types are low-alloy tool steels, , and powder metallurgy steels.

SURFACE HARDENING OF METALS

Surface hardening is a process in which a number of different techniques is used to improve some properties of the material without affecting others. Specifically, it can improve the properties of the

surface of the material or a selective part of the surface without interfering with the properties of the core.

For example, when there is a need for higher wear resistance or fatigue resistance of the surface with retaining ductile but tougher interior of the part, this method is more suitable to use.

Surface hardening is usually done for improving wear resistance, resistance to high contact stresses, fracture toughness, fatigue resistance and sometimes corrosion resistance.

Furthermore, surface hardening has the advantage to keep the costs at low levels because less expensive steels like medium-carbon steels and low-carbon steels can be surface hardened without the limitation of distortion and cracking associated with the through hardening of thick sections. [26] [27]

Carburizing

Carburizing is the addition of the carbon to the surface of low-carbon steels at high temperature (generally around 900 °C) at which austenite with high solubility for carbon is the stable crystal structure of the metal.

Hardening is accomplished when the surface (with high-carbon concentration) is quenched to form martensite. The high-carbon martensitic case is improved having good wear resistance on a tough, low-carbon steel core.

There are different methods of carburizing according to the requested properties. The methods are the following:

1. Pack Carburizing

2. Salt Bath Carburizing

3. Gas Carburizing

4. Vacuum Carburizing

5. Plasma Carburizing

Nitriding

Nitriding is a method similar to carburizing. Nitriding is a surface hardening heat treatment that introduces nitrogen into the surface of steel, but in comparison with carburizing, nitriding occurs at temperature range (500 to 550 °C), while the structure is in ferritic phase.

The difference between those methods is that carburizing adds carbon into austenite, but nitriding adds nitrogen into ferrite. Because of this nitriding does not involve heating into austenite phase field and a subsequent quench to form martensite but nitriding can be accomplished the minimum amount of distortion with excellent dimensional control.

The mechanism of nitriding and the different nitriding media may have unsimilar results, depending on the different grade steel. As carburizing, nitriding have different methods according on the application. The methods are the following:
1. Gas (box furnace or fluidized bed) Nitriding

2. Liquid (salt bath) Nitriding

3. Plasma (ion) Nitriding   [27]

Carbon and Nitrogen

Surface hardening with carbon and nitrogen offers processing temperatures between those of carburizing and nitriding.

The methods that use carbon and nitrogen for surface hardening are:

1. Carbonitriding

2. Austenitic Nitrocarburizing

3. Ferritic Nitrocarburizing

All the methods have higher processing temperatures than nitriding does. One of the advantages is that they have is been suitable for plain carbon steels.
The Austenitic Nitrocarburizing and the Ferritic Nitrocarburizing techniques rely on the formation of a thin layer of ε carbonitride, while carbonitriding use nitrogen for hardening the carburized austenite. [27]

There are more methods of surface hardening of metals:

1. Flame Hardening: Flame hardening involves heating the surface of a component using a high-temperature flame, followed by quenching. The localized heating and subsequent rapid cooling create a hardened surface layer.
2. Laser Hardening: Laser hardening utilizes a high-energy laser beam to selectively heat the surface of a metal component. The surface is then rapidly cooled, resulting in a hardened layer.
3. PVD/CVD Coatings: Physical Vapor Deposition (PVD) and Chemical Vapor Deposition (CVD) techniques are used to deposit thin layers of hard coatings onto the metal surface. These coatings provide improved wear resistance and surface hardness. [27]

The method of surface hardening is chosen based on the specific requirements of the component, the desired depth of hardening, and the material being treated.

There are several reasons for hardening the surface of metals:

1. Wear Resistance: By hardening the surface, the metal becomes more resistant to wear caused by friction, abrasion, or contact with other surfaces, extending the component's lifespan in demanding applications.
2. Fatigue Strength: Surface hardening can improve the fatigue strength of a component by reducing crack initiation and propagation, enhancing its resistance to cyclic loading.
3. Corrosion Resistance: Some surface hardening methods, such as certain types of coatings or treatments, can provide improved corrosion resistance, protecting the metal from chemical reactions or environmental factors.
4. Dimensional Stability: Surface hardening methods can minimize distortion or dimensional changes during subsequent manufacturing processes or during service, ensuring the component retains its desired shape and dimensions. [26]

MANGANESE

Manganese is a chemical element hard, brittle metal of group 7 (VIIb) of periodic table with the symbol Mn and atomic number 25. Since 1770 many scientists were studying on this . It was in 1774 that it was first isolated by the Swedish chemist Carl Wilhelm Scheele using the method of mineral pyrolusite. [28]

PROPERTIES OF MANGANESE
Manganese is a common metal with many advantages founded in almost every steel. It has similar chemical and physical properties to iron and is a useful deoxidizer and desulphuriser as S. ans P. are particularly ductile and harmful for the steel. It is common addition and is often omitted from the specifications of steels unless it is present in quantities of more than 2%.
Manganese is a grey-white metal that resembles iron, it's harder than iron but it's very brittle, its chemical reactive and it decomposes slowly in water. This metal is not found as a free element in nature but it's in a combination of other elements.
Manganese is acquired either through electrolysis of manganese sulfate or by reducing the oxide with magnesium, sodium or aluminum.
The manganese is found mostly in the form of ferromanganese and silicomanganese alloys.
Manganese can become ferromagnetic after suitable treatments. It also reacts with many nonmetals at high temperatures but its stays unreactive at room temperatures.[29]

EFFECT OF MANGANESE

Manganese has notable effects on alloy metals, influencing their properties in various ways. The impact of manganese on specific alloys can differ depending on factors such as composition and application. Here are the effects of manganese on alloy metals, along with the corresponding sources:

1. Strength and Hardenability Enhancement: Manganese is commonly utilized as an alloying element in steels and cast irons to increase their strength and hardenability. It forms solid solution strengthening with iron, leading to improved overall strength and hardness. The promotion of fine-grained structures during solidification contributes to enhanced toughness and wear resistance [28].
2. Improved Workability and Machinability: The addition of manganese to certain alloys, including aluminum and copper alloys, can enhance their workability and machinability. By reducing brittleness, manganese increases the ductility of these materials, making them more pliable and easier to shape through various processes such as rolling, extrusion, and forging [29].
3. Enhanced Corrosion Resistance: In stainless steels, manganese is frequently incorporated to enhance corrosion resistance. It helps stabilize the austenitic phase, which exhibits high resistance to corrosion. Additionally, manganese aids in the formation of a protective oxide layer on the alloy's surface, acting as a barrier against corrosive substances [30].
4. Solid Solution Strengthening: Manganese has the ability to form solid solutions with different metals, including aluminum, copper, and nickel. This mechanism of solid solution strengthening increases the strength and hardness of alloys. For example, in aluminum alloys, manganese contributes to the formation of intermetallic compounds, reinforcing the material and improving its mechanical properties [31].
5. Deoxidation and Desulfurization: Manganese possesses strong deoxidizing properties, making it useful for removing oxygen during the steelmaking process. Additionally, it aids in desulfurization by forming manganese sulfides, which can be eliminated from the alloy. These actions contribute to the production of cleaner and more homogeneous alloys [28].
6. Thermal Stability and Creep Resistance: Manganese plays a role in enhancing the thermal stability and creep resistance of alloys, particularly at elevated temperatures. By improving

strength and resistance to deformation under prolonged high-temperature exposure, manganese-containing alloys are well-suited for applications in high-temperature environments [32].

EFFECT OF MANGANESE ON MARTENSITIC TRANFORMATION

Manganese can have an influence on the start and finish temperatures of the martensitic transformation in alloy steels. The exact effects of manganese on these temperatures can vary depending on factors such as the specific alloy composition and processing conditions. [33] [34]

The addition of manganese to alloy steels generally tends to decrease the start temperature (Ms) of the martensitic transformation. This means that with higher manganese content, the martensitic transformation will begin at lower temperatures.

On the other hand, manganese can raise the finish temperature (Mf) of the martensitic transformation in alloy steels. Consequently, the martensitic transformation will conclude at higher temperatures when manganese is present in the alloy.

These effects of manganese on the martensitic transformation temperatures are influenced by its impact on the crystal structure and lattice parameters of the alloy. Manganese atoms can occupy interstitial sites within the crystal lattice, leading to changes in the atomic arrangement and affecting the transformation behavior.

It's important to note that the precise influence of manganese on the martensitic transformation temperatures can be complex and depend on various factors such as the alloy composition, processing conditions, and the presence of other alloying elements. Therefore, it is recommended to refer to specific research papers, metallurgical textbooks, or academic sources that delve into the topic of phase transformations in alloys to gain a more detailed understanding of the effects of manganese on the start and finish temperatures of the martensitic transformation. [33] [34]

Experiment

All actions and equipment employed in this experiment conform to the standards set by the American Society for Testing and Materials (ASTM). This ensures that the findings and conclusions derived from this research hold credibility within the scientific community. The equipment utilized in the experiment undergoes rigorous testing and calibration procedures, while the actions performed by individuals involved in the experiment strictly adhere to the prescribed guidelines outlined by ASTM. By following these established standards, the validity of the findings obtained from this study is upheld, thereby enhancing the reliability and acceptance of the research outcomes within the scientific community.

1.3   *Justification of the research*

The rapid progression of technology has generated a need for high-quality equipment characterized by exceptional mechanical attributes. Tool steels play a pivotal role in fulfilling these demands. When selecting a tool steel for a specific application, careful consideration must be given to several factors. These factors encompass the properties of the part itself, including its chemical composition,

shape, size, and intended application. Each of these aspects significantly contributes to the final product's quality and usability.

Nimax is the trade name of a low-carbon plastic mold tool steel developed by Uddeholm. It is highly regarded for its remarkable machinability, easy weldability, and impressive polishing and texturing properties. It exhibits exceptional resistance to indentations and possesses high impact and fracture toughness, rendering it a dependable and efficient choice.

Nimax is designed to be utilized in its as-delivered condition and does not exhibit any significant enhancement in hardness or other properties through heat treatment (14). Nevertheless, the demands for plastic products has garnered considerable attention towards plastic mold tool steels. Consequently, enhancing the mechanical properties of these steels has become an utmost priority in order to meet the ever-expanding demand for plastic products.

The primary objective of this thesis is to conduct an experiment utilizing alternative heat treatment methods on Uddeholm Nimax tool steel. The aim is to investigate the feasibility of augmenting its hardness through advanced heat treatment techniques. The successful attainment of this objective would broaden the range of applications for Uddeholm Nimax, enabling its utilization in scenarios with higher requirements. Additionally, the experiment has the potential to challenge the presumption, put forth by the manufacturing company, that the hardness of this steel cannot be increased. Such findings may also hold implications for other steels exhibiting similar properties, thus opening new roads for the application of tool steels in diverse fields. (14)

1.4. Research questions

This thesis aims to address the following questions:

1. Can heat treatment alternatives improve the hardness of the specific tool steel grade, which the producing company, Uddeholm, claims that cannot be achieved?
2. Will heat treatment alternatives successfully enhance the hardness of Nimax tool steel without compromising other mechanical properties?
3. Can the application of heat treatment techniques prolong the lifespan of plastic mold tool steel after undergoing heavy usage?
4. Are there any alternative methods that can yield the desired results in improving the properties of the specific tool steel grade?

These questions form the core focus of this thesis, as they seek to explore the potential of heat treatment alternatives, their impact on hardness improvement, the maintenance of other mechanical properties, and the overall durability of the plastic mold tool steel. The findings from this investigation will contribute to the knowledge base regarding the suitability and viability of different approaches in enhancing the performance of Uddeholm Nimax.

2. Proposed Methodology

2.1. Methodological approach & assumptions

This thesis requires an experiment from one specific tool steel grade as the test subject (Uddeholm Nimax). The examination of three different specimens of the same steel grade on the microstructure and on the properties. The specimens had gone through three different heat treatments. The specimens will be tested on

the lad and it's going to be adjudicated if the surface of the material is hardened and if so, how much. What other affects may appear and at the end to decide if hardening Uddeholm Nimax bulk heat treatment alternatives is possible.

The first step is the preparation of the specimens and after the test that is required to study the microstructure of the specimens. The steps are the following:

- Cutting of the specimens on smaller parts
- Mounting the specimens
- Plane and fine grinding the surface of the specimens to make a flat and smooth surface using Silicon carbide papers (from 180 to 2400 grains)
- Polishing the surfaces of the specimens (from 6μm to 0.3μm)
- Use microhardness tester to measure the hardness on the specimen's surface using Vickers hardness unit.
- Chemical eching of the surface of the specimens in order to study the microstructure on the microscope.
- Use optical metallographic microscope to observe the difference on the surface of each specimen according to the nature of grains, the size and the number of them

These are the basic steps of the experiment. Everything takes place to the lab of the Quality Control and Technology of Metallic Materials of the University of West Attica. Every part of equipment that is going to be used is calibrated and every operation is following the astm standards.

ASTM INTERNATIONAL standards

The astm standards that is used for this experiment are the following:

- Designation: E140-97e2
Standard Hardness Conversion Tables for Metals Relationship Among Brinell Hardness, Vickers Hardness, Rockwell Hardness, Superficial Hardness, Knoop Hardness, and Scleroscope Hardness

- Designation: E92 – 17
Standard Test Methods for Vickers Hardness and Knoop Hardness of Metallic Materials

- Designation: E112-13
Standard Test Methods for Determining Average Grain Size

Designation E3 – 95
Standard practice for preparation of metallographic specimens

- Designation: E1951-14
Standard Guide for Calibrating Reticles and Light Microscope Magnifications

2.2. *Research infrastructure*

The research infrastructure required is advance knowledge of heat treatments, properties of tool steels, subzero treatments, surface hardening, advance knowledge of metallic microstructure and knowledge for using the proper lab equipment. During this thesis the test results are followed according to astm standards,. The test machines and the equipment used will be the following:

- Cutting machine (3 Struers Model LAbotom)

- o Metallographic specimens mounting machine (Struers CitoPress-1)

- o Metallographic specimen sanding machine (Presi Mecapol P-262)

- o Metallographic specimen polishing machines (Struers Knuthrotor / Buehler Metaserv)

- o Optical metallographic microscope (Zeiss Axio Vert.A1)

2.3.   Case study

The experiment includes the sum of three different heat treatments on three different specimens of the same tool steel.

Each heat treatment is designed to work on a specific pattern of heat treatments. Meaning the first heat treatment is a simple treatment without expecting any changes on the hardness. The other two heat treatments are more complicated with different combinations of treatments.

In the beginning are the designs of the heat treatments going to be presented and afterwards its going to be presented every step of every test that the specimens undergo, with every important detail.

The following are the designs of every heat treatment and the hardness results of each specimen.

1) <u>Carburizing, quenched in water, cryotreatment, tempered</u> (microstructure, carburizing thickness, chemical etching)
   900°C for 180 mins, down to 850°C then water cooled
   Dry over night at -70°C (15:20 – 8:20 17 hours)
   ➔ Top 57 HRC, Side 59+ HRC
   580°C in GS430 for 120 mins
   ➔ 52 HRC

2) <u>Normalizing and double quench hardening</u> (microstructure, carburizing thickness, chemical etching))
   850°C for 30 mins then air cooled
   900°C for 180 mins, down to 850°C then oil cooled
   ➔ 38 – 39 HRC
   780°C for 30 mins then water cooled
   ➔ 46 – 48 HRC

3) <u>Normalizing</u> (microstructure, chemical etching)
   760 °C for 90 mins
   ➔ 39 – 40 HRC

<u>CHAPTER 2.3: Experimental Analysis, and the Preparation of the Specimens</u>

<u>2..1 Observation, Measurement, and Analysis of Specimens</u>
Before taking any further action, the initial step was to measure the dimensions of each original specimen of the three various heat treatments. A mechanical vernier calliper with a millimetre scale was utilized for this assessment. Following measurement, the samples were cleaned using methanol (CH4) to get rid of any residue from the surface.

Photographs were taken of each specimen along with its measurements; furthermore, these images were captured inside a photo box, which diffuses light eliminating dark areas on the specimens. It makes use of cold-coloured LED lighting; a coin was used as a benchmark size for dimension comparisons as portrayed in Figure 2.1.2.
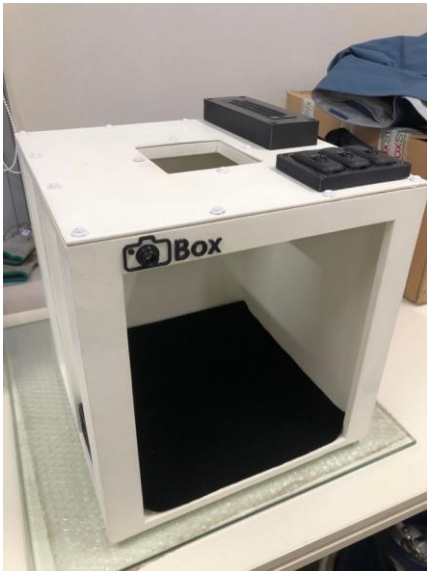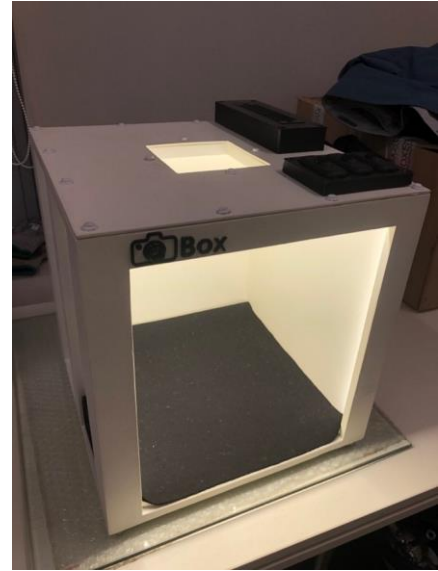
(A)   (B)

Figure 2.1.1: Indicates the photo box used to capture the images of the specimens : (a) Photo box, (B) Photo box diffusing light

The specimens undergo a detailed sequence of preparation steps as per Designation ASTM E3 - 95, which outlines the standardized procedure for metallographic specimen preparation. This includes thorough cleaning, cutting, mounting, grinding, and polishing. Adhering to these standardized procedures is crucial as they significantly impact the quality and accuracy of the subsequent test methods. Proper specimen preparation is of high importance to ensure reliable and consistent results.

(a)   (b)

(c)

(d)

Figure 2.1.2: First Heat Treatment Specimen comparative observations of the first specimen : (a) Top-down perspective, (b) Bottom-up perspective, (c) Lateral perspective, (d) Over-head/aerial perspective

(a)

(b)

(a)

(b)

(c)

(d)

(e)

(f)
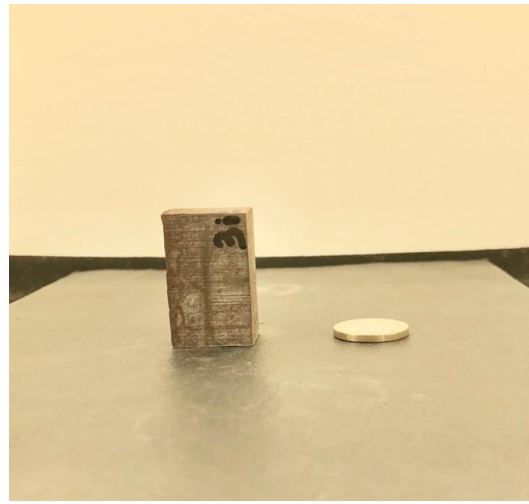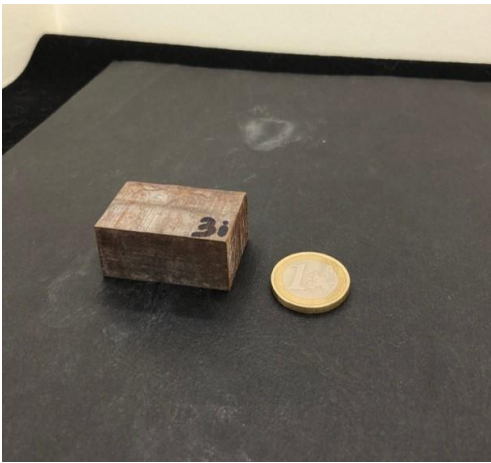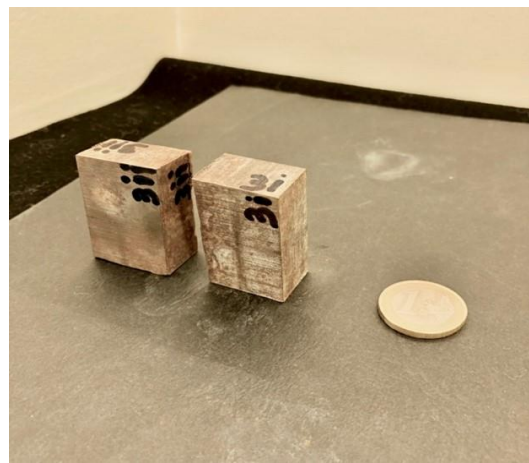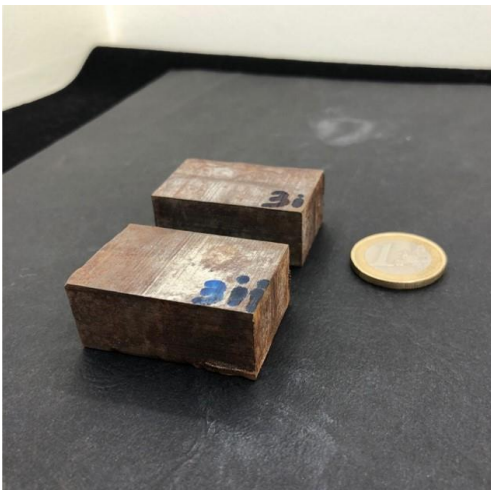
Figure 2.1 ... ond H... ...eatmer... ...ecimer... ...nparati... ...ervatio... of ...seco... ...ple : (...op-down ...rspectiv... (B) ...otton-up ...rspectiv... C) ...Lateral ...spectiv...

Figure 2.1.4: Third Heat Treatment Specimen; comparative observations of the third specimen. (a) Top-down perspective, (b) Lateral perspective, (c) Over-head/aerial perspective, (d) Over-head/aerial perspective, (e) Over-head/aerial perspective, (f) Top-down perspective

Table2.3.1: Measurement Table (Millimetre (mm)).

The table below represents the measurements taken for the different specimens, specifically their length, width, and height, all measured in millimetres (mm).

Specimens	Length (mm)	Width (mm)	Height (mm)
Specimen 1	30.6	51.9	-
Specimen 2	32	26.6	-
Specimen 3.1	40.6	24.9	17.3
Specimen 3.2	39.1	27.1	17.3

The Table provides a concise overview of the measured dimensions for each specimen, allowing for easy comparison and analysis of the physical characteristics of the specimens based on their length, width, and height.

2.2: Cutting and Preparation of Specimens

The cutting of specimens refers to a process in which larger pieces or samples of a material, typically in this context steel, are divided or sectioned into smaller, more manageable pieces for

further testing or analysis. It is a destructive testing method as the original specimen is altered or modified during the cutting process.

The Struers model Labotom – 3 (serial number:5321898)(Figure 2.2.1.a & b) with a cutting disk known as 40A25 (Figure 2.2.1.c), following to the EN 12413 standard which serves as the cutting machine utilized for this method. The choice of cutting disk was selected based on the hardness of the steel being tested. The spindle speed of the disk during the test was 3450 rpm at 60 Hz. To ensure proper cutting and prevent overheating, a cooling fluid Struers Unitclean (Figure 2.2.1.d) was employed during the cutting process. The dimensions of the final specimens comply with the ASTM Designation E3 - 95, as specified in the Standard Practice for Preparation of Metallographic Specimens.

(A)

(B)
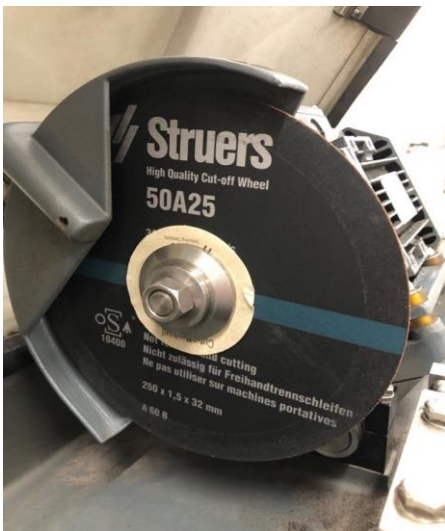
(C)

(D)

Figure 5.2.1: photos of the machine and tools used for the cutting off the specimen (A) Struers Model LAbotom – 3, (B) Struers Model LAbotom – 3 serial number, (C) Cutting Disk 50A25, D) Struers UnitClean cooling fluid

The experiment was conducted to the Quality Control and Technology of Materials lab, University of West Attica[1] [2]. The procedural steps followed in this investigation were as follows:

- The specimens were cleaned with methanol (CH_3OH).

- The pieces were cut in a manner that generated the final specimens of the desired size and surface characteristics for examination. It was necessary to obtain multiple specimens from each piece.
- Methanol cleaning was conducted on each piece after sectioning, and the cuts were documented through photographic records.

One crucial aspect to consider during the cutting process was the direction of the cut. The specimens were cut to align with the original piece's orientation. This approach aimed to ensure consistent and comparable surface conditions for following experimentation aiming for repeated and reliable results.

The following images illustrate the progression of the test method, capturing each step and cut in detail.

(A)

(B)
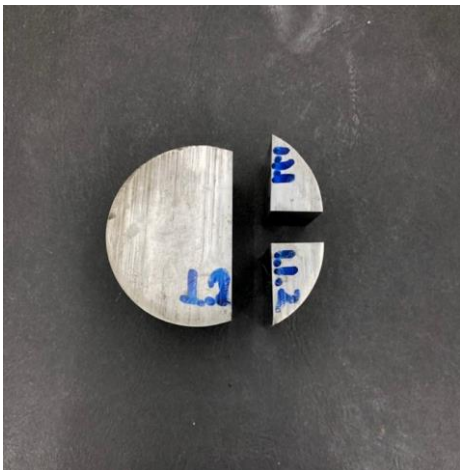
(C)

(D)

Figure 2.2.2: Sectioning and Preparation of the First Specimen: (A) Specimen 1 cut into 1.1 and 1.2, (B) Specimens 1.1 and 1.2 from an aerial perspective, (C) Specimen 1.2 cut into 1.1.1 and 1.1.2, (D) Specimens 1.2, 1.1.1, and 1.1. 2 from an aerial perspective, (E) Specimens 1.1.1 and 1.1.2 from an aerial perspective, (F) Specimens 1.1.1 and 1.1.2 from Top-down perspective, (G) Specimens 1.1.1.1 and 1.1.1.2 from an lateral perspective, (H) Specimens 1.1.2.1 and 1.2 from an lateral perspective, (I) Specimens 1.2, 1.1.1.1, 1.1.1.2, 1.1.2.1. and 1.1.2.2 from an aerial perspective, (J) Specimens 1.2, 1.1.1.1, 1.1.1.2, 1.1.2.1. and 1.1.2.2 from an aerial perspective

Figure 2.2.2.1 illustrates the preparation and cutting procedure employed for the first heat treatment specimen, following the sequential order outlined below:

• 1 -> 1.1 + 1.2
• 1.1 -> 1.1.1 + 1.1.2
• 1.1.1 -> 1.1.1.1 + 1.1.1.2
• 1.1.2 -> 1.1.2.1 + 1.1.2.2

The resulting cut specimens, used from this division, were designated for subsequent test methods, specifically 1.1.1.1, 1.1.1.2, 1.1.2.1, and 1.1.2.2.

(A)

(B)

(C)
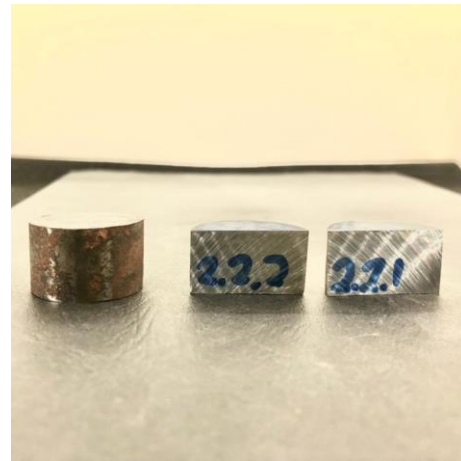
(D)

Figure 2.2.3: Sectioning and Preparation of the Second Specimen : (A) 2 -> Specimens 2.1, 2.2.1, and 2.2.2 from Top-Bottom perspective, (B) Specimens 2.1, 2.2.1, and 2.2.2 from an aerial perspective, (C) Specimens 2.2.1 and 2.2.2 from Top-Bottom perspective, (D) Specimens 2.1, 2.2.1, and 2.2.2 from lateral perspective

Figure 2.2.3.1 portrays the preparation and cutting technique for the second heat treatment specimen, which was cut in the below order:

• 2 -> 2.1 + 2.2
• 2.2 -> 2.2.1 + 2.2.2

The resulting cut specimens used from this division, specifically 2.2.1 and 2.2.2., were utilized for subsequent test methods.
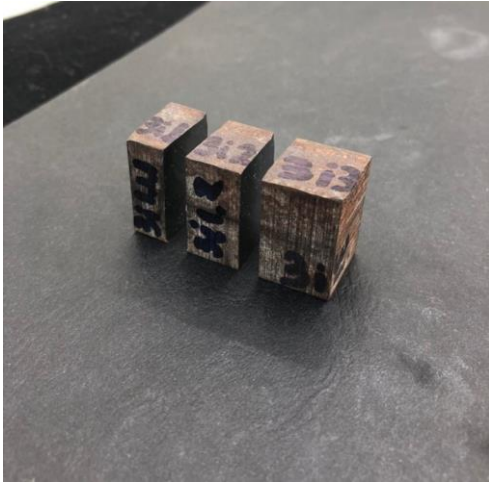
(A)

(B)

(C)

(D)

(E)

(F)

Figure 2.2.4 Sectioning and Preparation of the Third Specimen : (A) Specimens 3.i.2 and 3.i.3 from an aerial perspective, (B) Specimens 3.i.1, 3.i.2 and 3.i.3 from Top-Bottom perspective, (C) Specimens 3.i.1, 3.i.2 and 3.i.3 from an aerial perspective, (D) Specimens 3.i.1, 3.i.2.1, 3.i.2.2 and 3.i.3 from Top-Bottom perspective, (E) Specimens 3.i.1, 3.i.2.1, 3.i.2.2 and 3.i.3 from an aerial perspective, (F) Specimens 3.i.2.1 and 3.i.2.2 from an aerial perspective

The cutting process for the specimens for the third heat treatment were obtained by cutting the original specimen 3i in the following order:

• 3i -> 3i1 + 3i2 + 3i3
• 3i2 -> 3i2.1 + 3i2.2

The pictures in Figure 5.2.4 depict the specimens of the third heat treatment, the assigned codes for the specimens were as follows: 2.1 corresponds to 3i2.1, and 2.2 corresponds to 3i2.2.
The final specimens that were utilized for the subsequent test methods were 3i2.1 and 3i2.2.

2.3 Specimen Mounting Method: Utilizing the Struers CitoPress-1 and Hot Mounting Process
In cases where specimens are small in size and require a flat and smooth surface for further examination, it is often necessary to mount them using epoxy or phenolic resins. These resins come in various types depending on the method and application. Hard resins are typically used when durability and hardness are required, while softer resins are more commonly used for general purposes. Additionally, the choice of resin may vary depending on the specific specimen and its intended application.
Mounting can be achieved through either cold mounting or hot mounting processes. Cold mounting involves using room temperature curing resins, while hot mounting utilizes resins that require heating and pressure to facilitate the curing process. The selection of the mounting process depends on factors such as the nature of the specimen and the desired properties of the final mounted specimen.

The mounting machine selected for this method was the Struers CitoPress-1 (Fig. 2.3.1.A), which utilized the hot mounting process. The setup of the process can be observed in the provided picture in Figure 2.3.1.B. Struers AntiStick powder (Fig. 2.3.1.C) was employed to facilitate the mounting process. Additionally, two types of resins, namely DuroFast and MultyFast by Struers (Fig. 2.3.1.D and 2.3.1.E), were used for mounting the specimens.
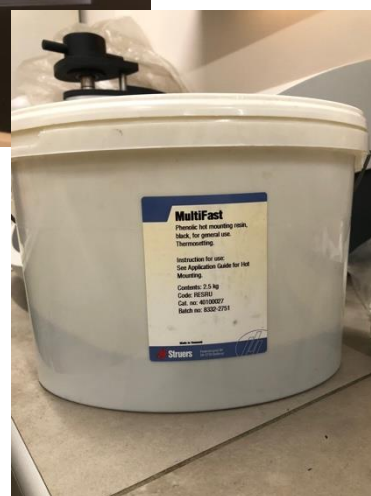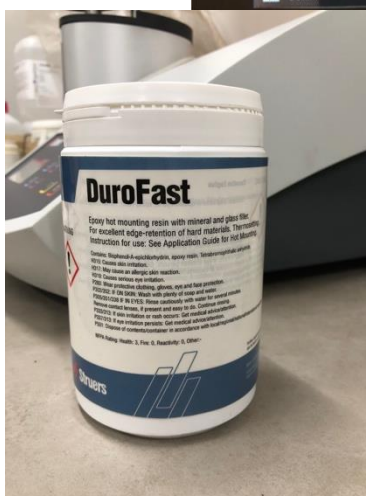
(C)   (D)   (E)
Figure 2.3.1: provides insight into the method, equipment, and materials involved in achieving effective specimen mounting using specified machinery and process : (A) Struers CitoPress-1 mounting

machine, (B) The process setup, (C) Struers AntiStick powder, (D) DuroFast resin by Struers, (E) MultyFast resin by Struers

The test was conducted to the Quality Control and Technology of Materials lab, University of West Attica [1] [2]. The experimental procedure followed for this test is outlined as follows:

1. Specimen Cleaning: The specimens were accurately cleaned using methanol (CH4) and later dried to remove any contaminants.
2. AntiStick Application: AntiStick was carefully applied to the rams of the mounting press to prevent sticking and ease smooth specimen removal.
3. Specimen Placement: The specimens were positioned on the rams of the mounting press, ensuring that the examining surface faced downwards, making contact with the powder.
4. Resin Application: A precise allocation of 10ml of DuroFast resin, followed by 20ml of MultiFast resin, was evenly applied to the specimens. Special attention was given to achieving a uniform distribution for optimal results.
5. Mounting Process: The device was securely locked, and the mounting process was initiated. The resins were subjected to controlled heating at a temperature of 180°C, accompanied by a pressure of 250 bar, for a duration of 3 minutes. Subsequently, the specimens were cooled under high-pressure water for 2 minutes.
6. Specimen Removal: Following the completion of the mounting process, the device was carefully unlocked, and the specimens were gently removed, ensuring their integrity and preservation for further analysis.

By following the mentioned procedural steps, the specimens undergo a cleaning process, ensuring the removal of any impurities or contaminants. The mounting process takes place under specific temperature and pressure conditions, precisely controlled to achieve optimal results. These steps are of utmost importance in preparing the specimens for further analysis and experimentation.

Results of the specimens after mounting:
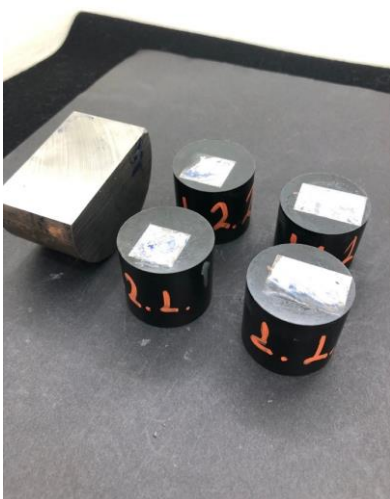
(A)

(B)

Figure 2.3.2: The results of the first heat treatment specimens after mounting : (A) Mounted Specimens 1.1.1.1, 1.1.1.2, 1.1.2.1., 1.1.2.2 and specimen 1.2 from an aerial perspective, (B) Mounted Specimens 1.1.1.1, 1.1.1.2, 1.1.2.1, 1.1.2.2 and specimen 1.2 from Top-Bottom perspective
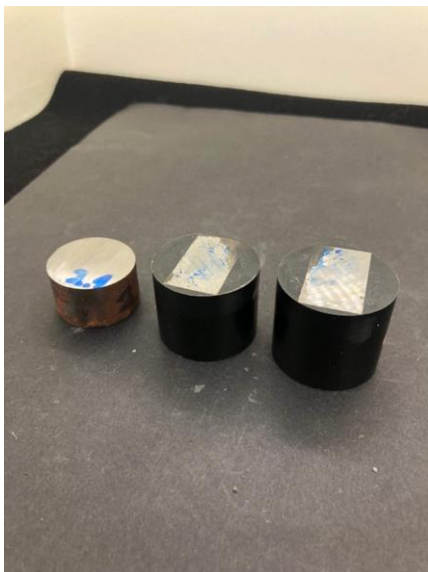
(A)

(B)

Figure 2.3.3: Results of the second heat treatment specimens after mounting : (A) Mounted Specimens 2.2.1, 2.2.2 and specimen 2.1 from an aerial perspective, (B) Mounted Specimens 2.2.1, 2.2.2 and specimen 2.1 from Top-Bottom perspective
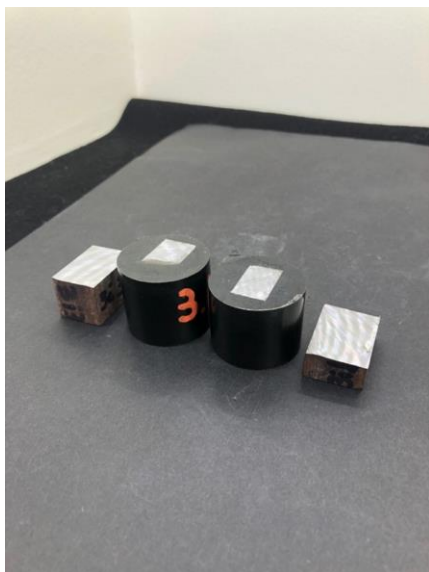
(A)

(B)

Figure 2.3.4 Results of the Third heat treatment specimens after mounting: (A) Mounted Specimens 3.i.2.1, 3.i.2.2 and specimens 3.i.1 and 3.i.3 from an aerial perspective, (B) Mounted Specimens 3.i.2.1, 3.i.2.2 and specimens 3.i.1 and 3.i.3 from Top-Bottom perspective

The mounting of the three specimens from different heat treatment stages produced positive results. The application of the designated mounting process, combined with the appropriate use of DuroFast and MultiFast resins, ensured secure and reliable fixation of the specimens. Upon visual inspection, all eight mounted specimens showed smooth and defect-free surfaces, indicating a successful mounting procedure.

These specimens are now ready for following testing and analysis, contributing to a comprehensive understanding of the heat treatment effects. The consistent and reliable mounting process employed across all three specimens enhances the validity of the research findings, providing a solid foundation for further investigations and conclusions within the study.

As per the experimental procedure, a 3D sketch was created using the Rhino or Rhinoceros 3D program to visually represent the surfaces that will undergo further testing using the mounting method. The sketch (Fig. 5.3.5) clearly highlights the selected surfaces in red colour, ensuring their easy identification. Each surface is also labelled with a unique code, namely p2.3.1, p2.3.2, and p2.3.3, for precise referencing.
Including this 3D sketch in the study provides a helpful visual reference to identify and work with the specific surfaces designated for testing. The clear labelling assist in accurate planning and completing of the next steps.

Figure 2.3.5: 3D visualisation of the surfaces that underwent further testing

(A) First heat treatment specimen in 3D figure
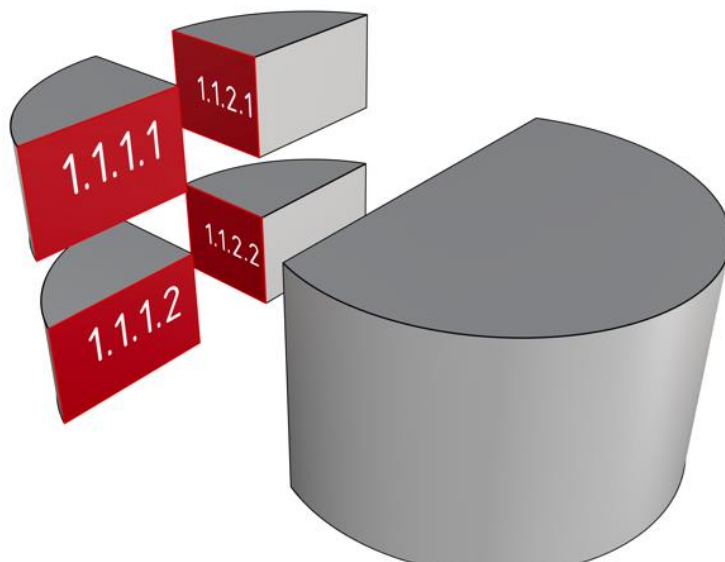
(B) Second heat treatment specimen in 3D figure
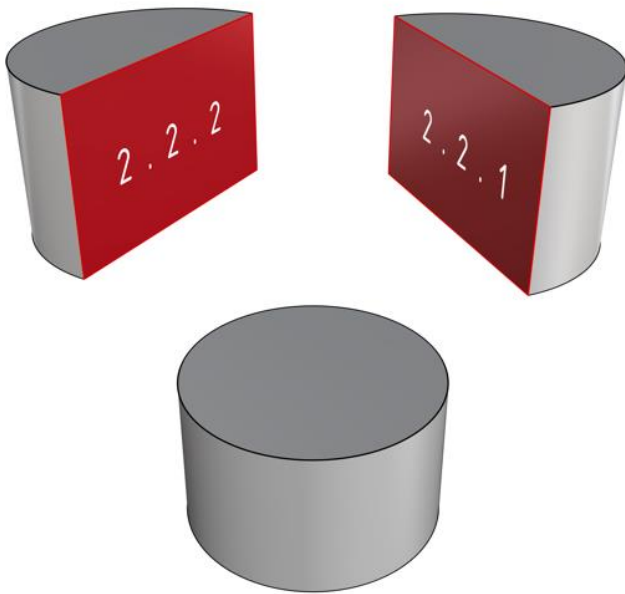
(C) Third heat treatment specimen in 3D figure
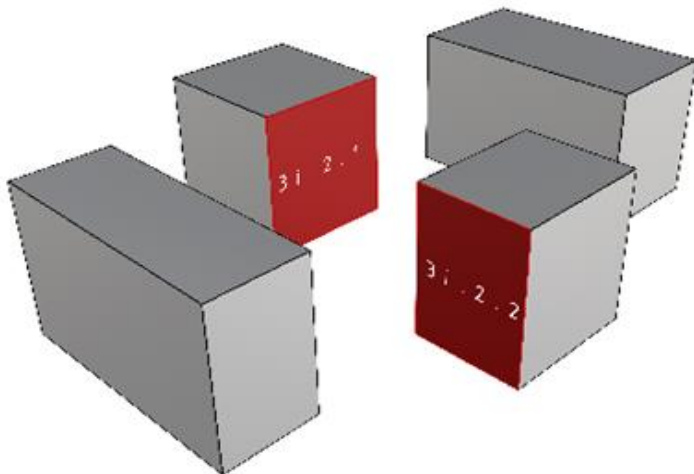
2.4: Griding and Polishing and chemical etching Procedure for Specimen Preparation

Having reached the final phase of specimen preparation, the surfaces of the specimens underwent griding and polishing procedures. Through griding, small layers of material were gradually removed from the specimen surfaces to achieve a smoother texture. Subsequently, polishing was performed using different silicon carbide paper grit size to reach an even higher level of surface smoothness. Chemical corrosion is an essential procedure in order to proceed to the following test, the observation of the microstructure on the metallographic microscope.

For the grinding process, a Presi Mecapol P262 (Fig. 5.4.1) griding machine was utilized. Struers griding papers of various grit sizes (P160, P320, P600, P800, P1000, P1200) were employed. Each griding pass was repeated consistently for 20 seconds, ensuring repeatability across all specimens and sandpapers. The SiC papers rotated in a clockwise direction at a fixed 240 RPM for each stage.

(A)   (B)

Figure 2.4.1: (A), (B) : Presi model: Mecapol P262 grading machine
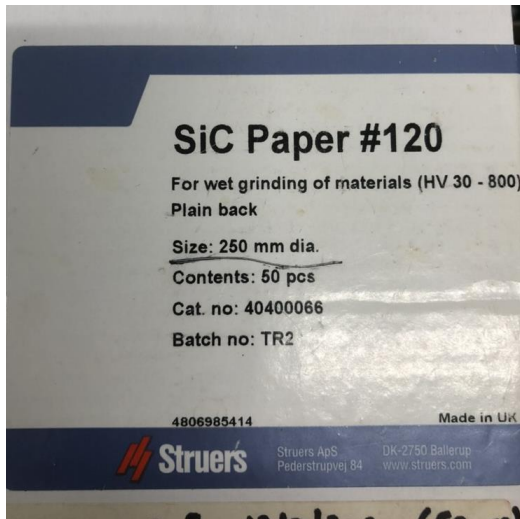
Figure 2.4.2: Samples derived from the utilized silicon carbide papers: (A) 120 grid paper by Struers, (B) 120 grid paper by Struers, (C)800 grid paper by Struers, (D) 2400 grid paper by Struers

The polishing process involves a series of four sequential steps to enhance the specimens. Each step necessitates a distinct cloth and a specific type of paste or liquid. The stages responsible for achieving a mirror-like surface include 6μm, 3μm, 1μm, and 0.3μm grain size.

The initial two steps will be carried out employing the Struers polishing machine (Figure 2.4.3.A and 2.4.3.B), with [sin: 1445569] (Figure 2.4.3.C). For both step one and step two, Struers polishing cloths will be utilized (Figures 2.4.4.A and 2.4.4.B). The consumables for these steps consist of Metkon diamond pastes of 6μm and 3μm (Figures 2.4.4.C) in conjunction with Metkon diamond lubricant water (Figure 2.4.4.D) for the first step (6μm). Furthermore, for step two (3μm), water-based diamond suspension and DP Lubricant blue by Struers will be employed (Figures 2.4.4.E and 2.4.4.F). Each specimen will undergo a single pass of 90 sec on the polishing machine at 250 rpm. Subsequently, the surface will be cleansed using a jet of water, followed by a jet of methanol, and dried with warm air and a cotton cloth.

The subsequent two steps will be executed utilizing the Buehler Metaserv polishing machine (Figure 2.4.5A), with [sin: 398824161]. These final two steps will follow the same procedure. The polishing cloths (Figure 2.4.5.B and 2.4.5.C) will be combined with alumina polishing suspension of 1μm and 0.3μm (Figures 2.4.6.E and 2.4.6.F). Distilled water will serve as the lubricant for these last two steps. The duration of each pass will remain consistent at 90 seconds on the polishing machine at 100 rpm. Similarly, the surface will be subjected to a jet of water, a jet of methanol, and dried using warm air and a cotton cloth.
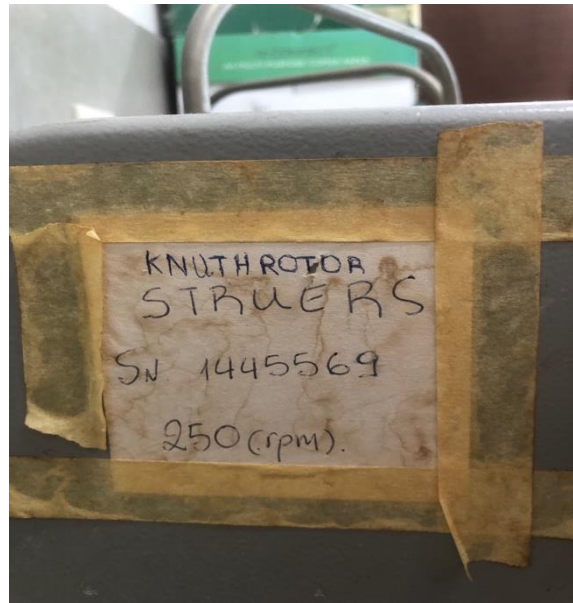
(A)   (B)

Figure 2.4.3: polishing machine used for 6μm and 3μm polishing stage : (A) Struers polishing machine, (B)serial number of Struers polishing machine and rpm
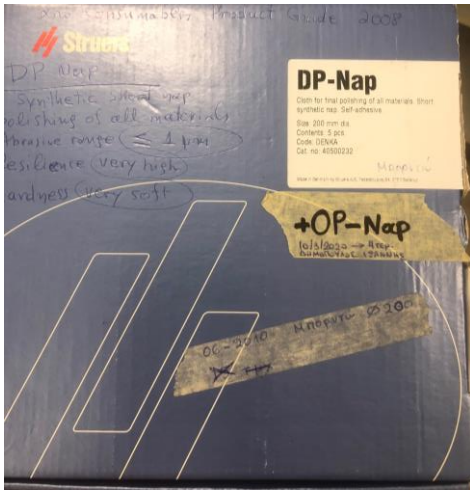
(A)
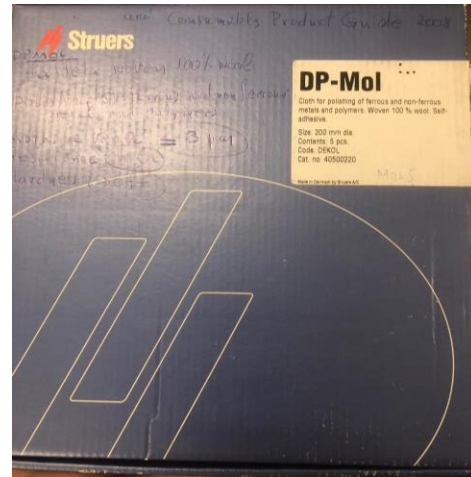
(B)
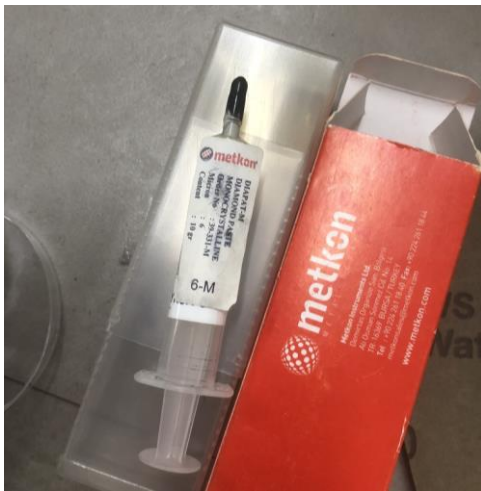
(C)
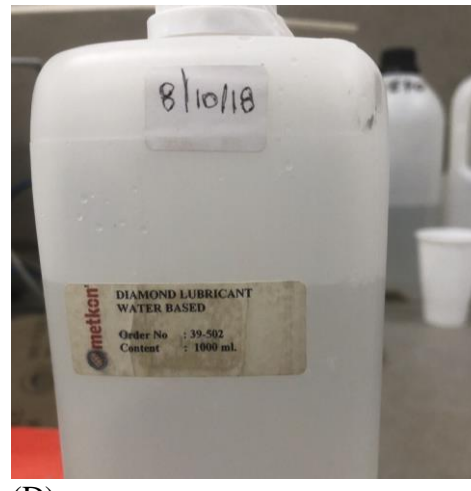
(D)
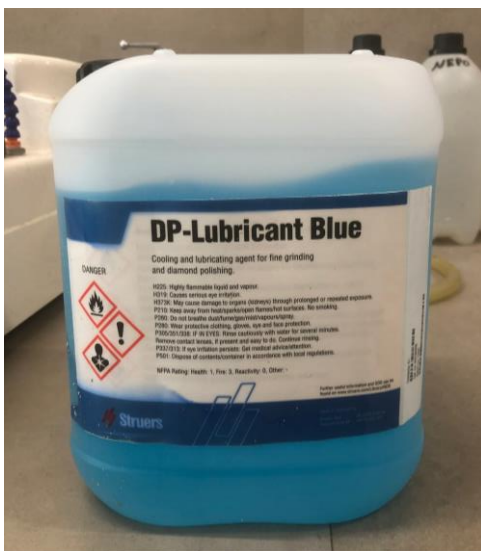
(E)

(F)

Figure 2.4.4: polishing cloths, and consumables for 6μm and 3μm polishing stages: (A) Polishing cloth used on 6μm, (B) Polishing cloth used on 3μm, (C) Diamond paste used on 6μm, (D) Diamond Lubricant water used on 6μm, (E) DP- Lubricant blue used on 3μm, (F) DiaDuo-2 Lubricant used on 3μm

(A)
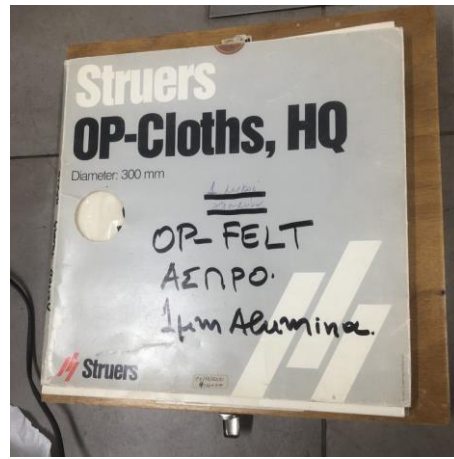
(B)
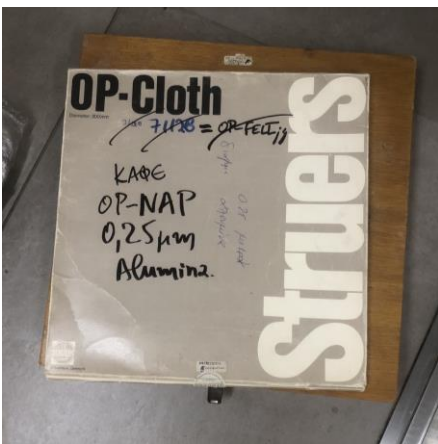
(C)

(D)

(E)

Figure 2.4.5: polishing cloths, and consumables for 1µm and 0.3µm polishing stages: (A) Buehler Metaserv polishing machine, (B) Polishing cloth used on 1µm, (C) Polishing cloth used on 0.3µm, (D) AP-A suspension used on 1µm, (E) AP-A suspension used on 1µm

Chemical etching is a process which the surface of the specimen (polished down to 0.3μm) is getting corrodes specific parts of the surface of the metal. After that process the pattern of the microstructure of the metal can be presented on the microscope. It's essential that the last stage of the polishing (0.3 μm) and the chemical etching of the specimens to occur consequently one after the other with minimum delay in order to avoid corrosion of the surface with the oxygen of the environment. The chemical agents used for the specimens are the following:

NITAL 5% (95ml methanol CH_3OH – 5ml HNO_3)
VILLELAS (100ml methanol CH_3OH – 5ml hydrochloric acid HCL – 30ml Picric Acid $C_6H_2(NO_2)_3OH$

For the NITAL, the specimens were exposed to it for 10 seconds, then cleaned with methanol and dried with hot air and cotton cloth. For the VILLELAS, the specimen was exposed to it for 20 seconds, then cleaned with methanol and dried with hot air and cotton cloth. All the agents are merged and stored Quality Control and Technology of Materials lab, University of West Attica.
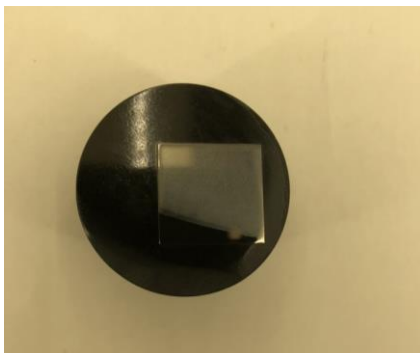
(A)
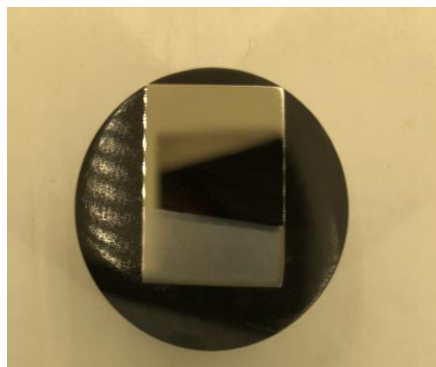
(B)
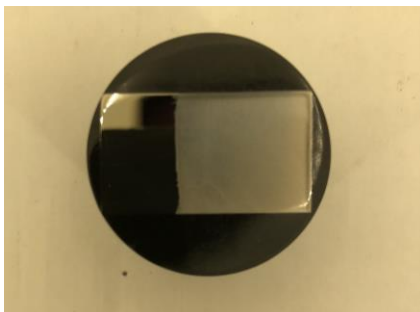
(C)

Figure 2.4.6: surfaces of the specimens after the chemical etching and macroscopic observation: (A) Specimen 1.1.2.1 after the chemical corrosion, (B) Specimen 2.2.2 after the chemical corrosion, (C) Specimen 3.i.2.1 after the chemical corrosion

2.5 Metallographic analysis of the microstructure of the specimens with optical metallographic microscope.

The metallographic analysis of the specimens will occur through the ability of observing the microstructure of the metal with great magnification of the surface. the microscope that is going to be utilized is: Zeiss Metalovert (fig 2.5.1) with capability of magnification (x50, x100, x200, x500, x1000) with [S/N : 3848001221]. All the pictures were taken in full bright field on the microscope. The pictures that follows are categorized through the specimen and the magnification.

(A)

(B)

Figure 2.5.0.1 (A), (B) Metallographic microscope Zeiss Metallovert inverted reflected light microscope.

Figure 2.5.1: microstructure of the first heat treatment specimens

(A1) Specimen 1.1.1.1, nital, x50, carburized zone, total impacted depth
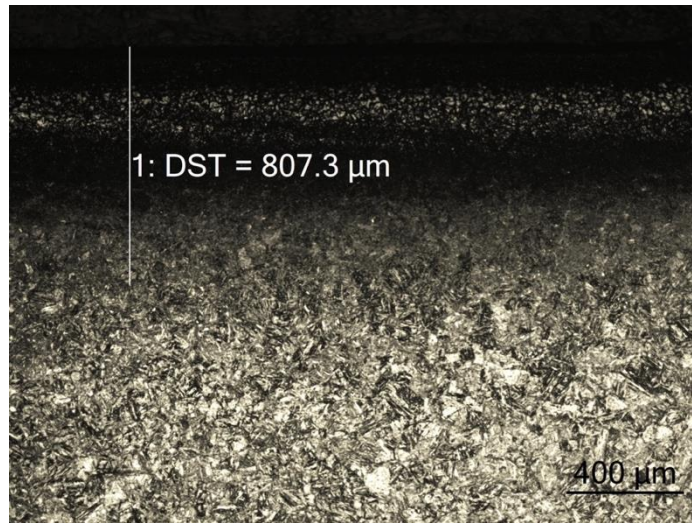
(A2) Specimen 1.1.1.1, nital, x100, carburized zone
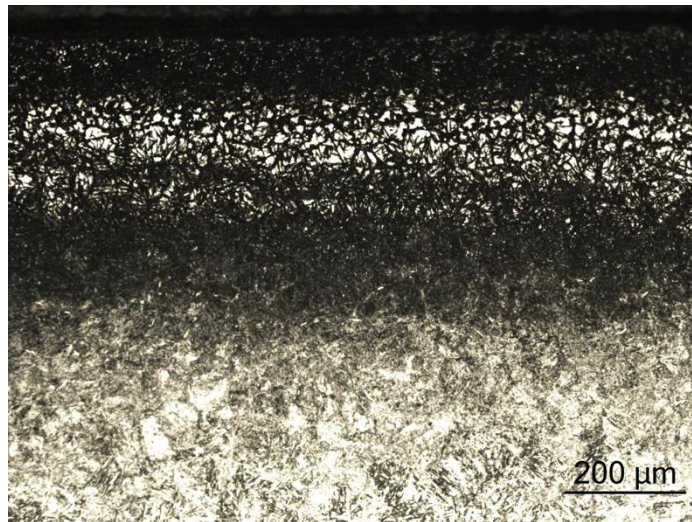

(A3) Specimen 1.1.1.1, nital, x100, subtractive microstructure

(A4) Specimen 1.1.1.1, nital, x200, intermediate zone 2-3
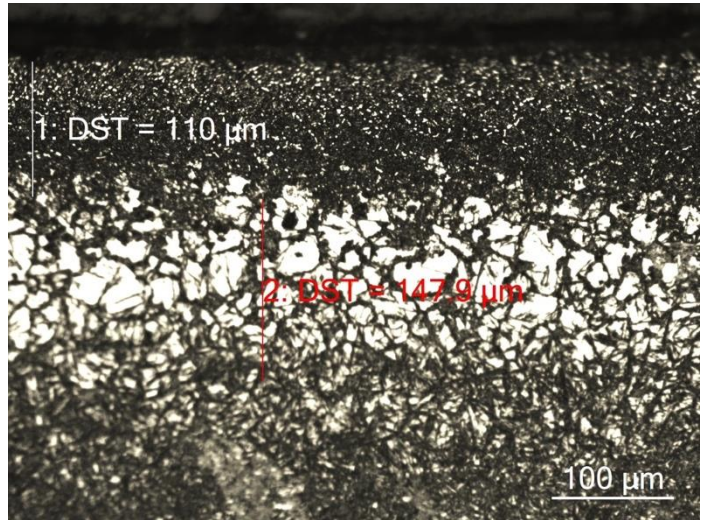
(A5) Specimen 1.1.1.1, nital, x200, intermediate zone 3-4
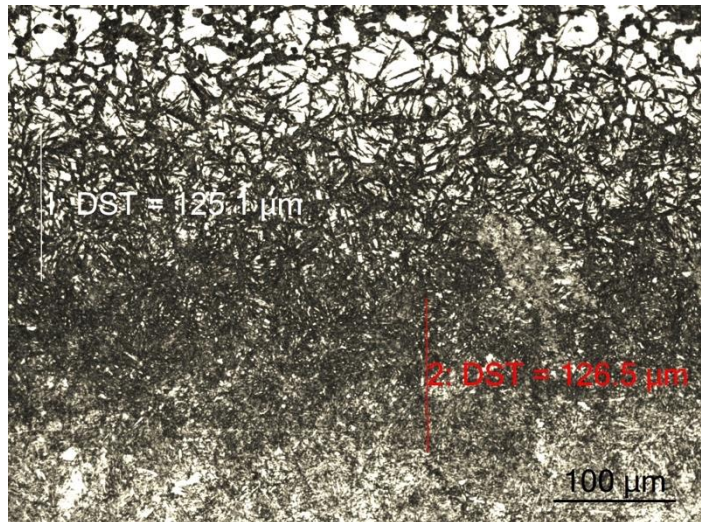
(A6) Specimen 1.1.1.1, nital, x200, intermediate zone 4- subtractive microstructure
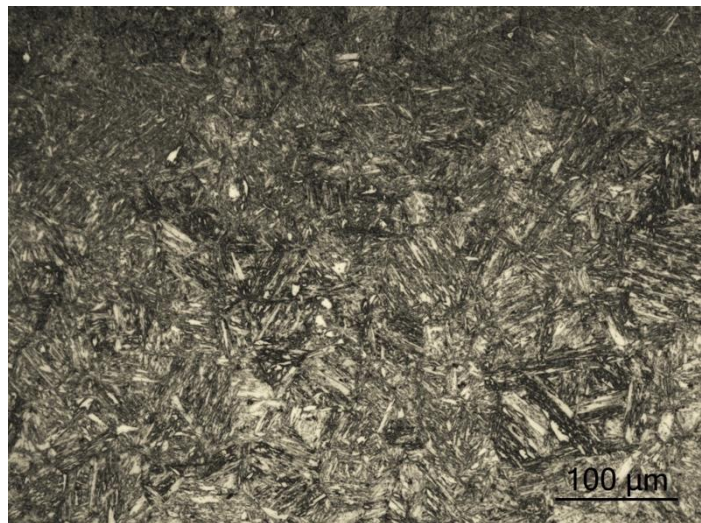
(A7) Specimen 1.1.1.1, nital, x500, intermediate zone 1
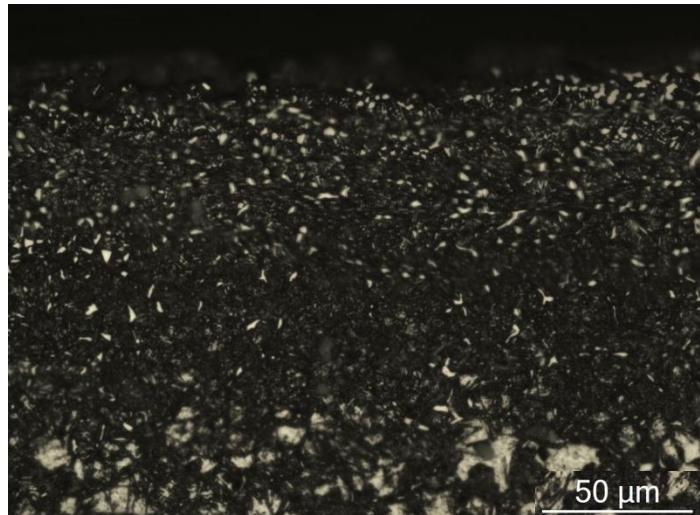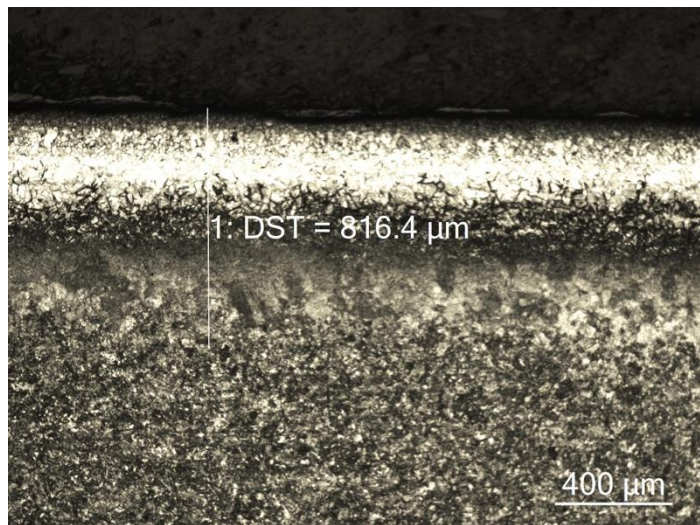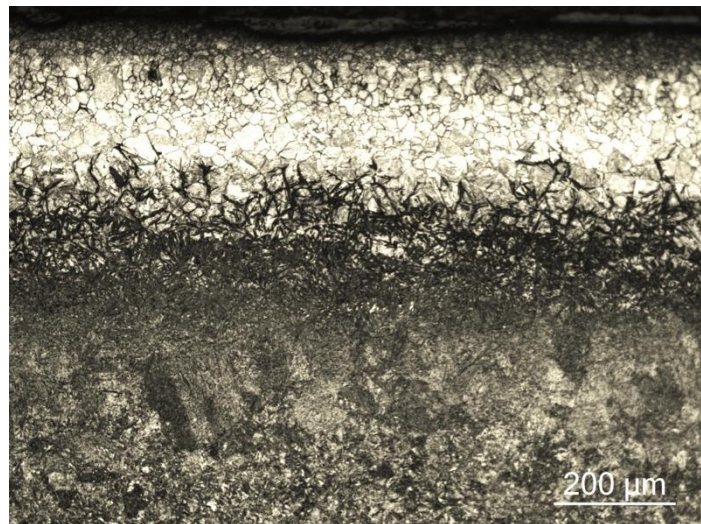

(A8) Specimen 1.1.1.1, nital, x500, intermediate zone 2
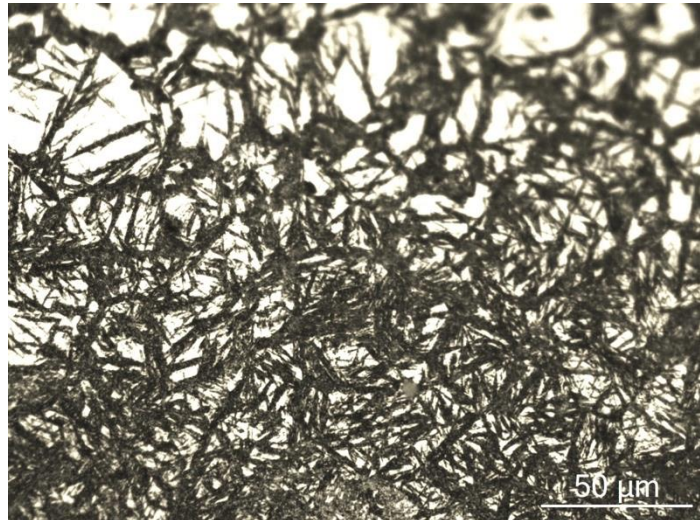
(A9) Specimen 1.1.1.1, nital, x500, intermediate zone 3
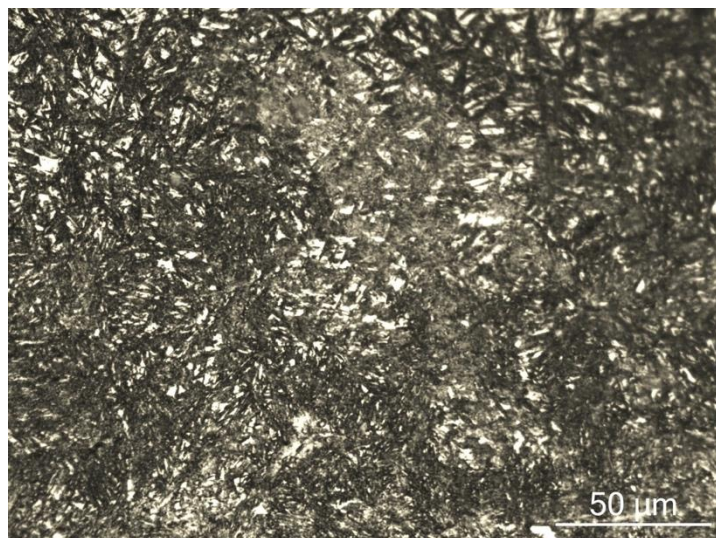
(A10) Specimen 1.1.1.1, nital, x500, intermediate zone 4
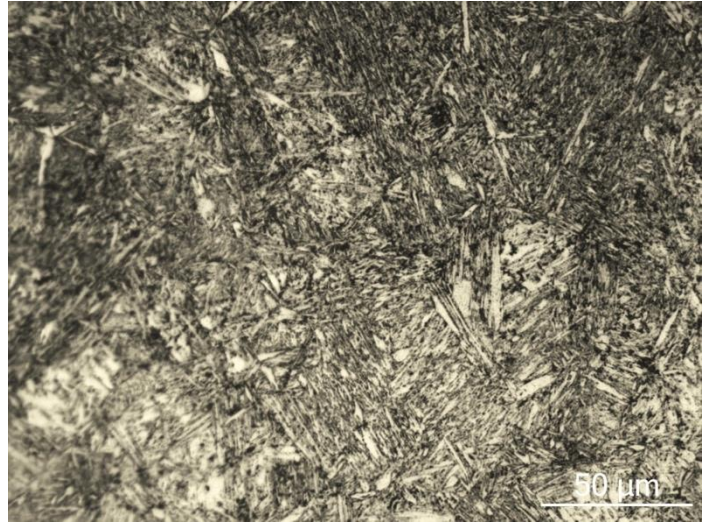
(A11) Specimen 1.1.1.1, nital, x1000, intermediate zone 3, bainite structure
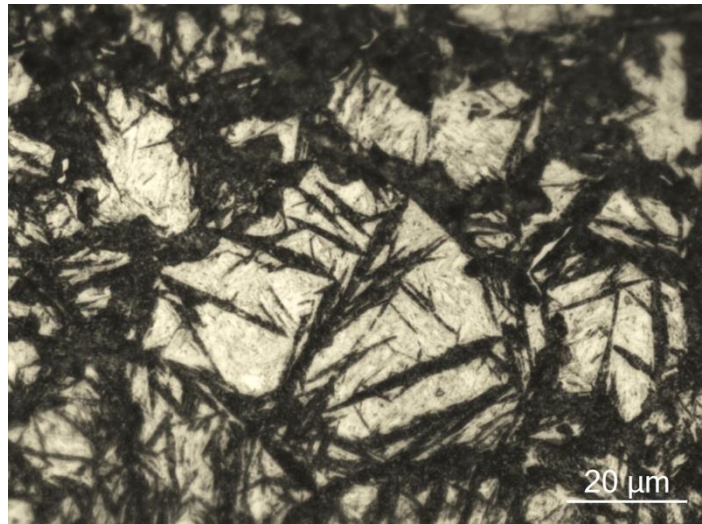
(A12) Specimen 1.1.2.1, villelas, x100, carburized zone, total affected depth

1: DST = 644.7 µm
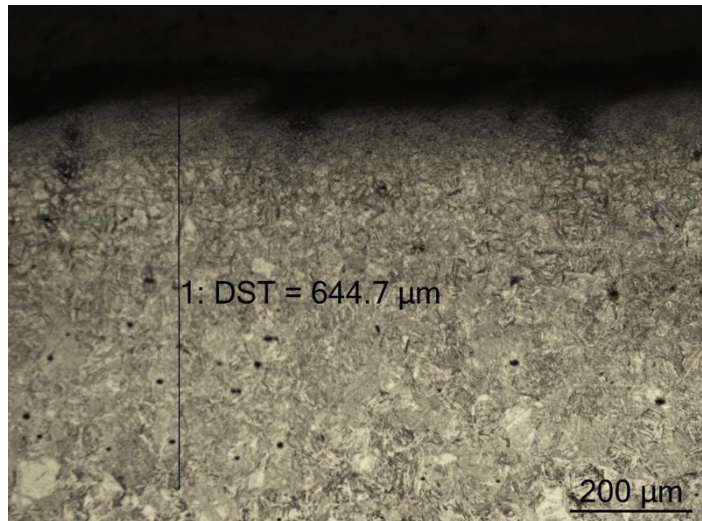
(A13) Specimen 1.1.2.1, villelas, x200, intermediate zone 1-2
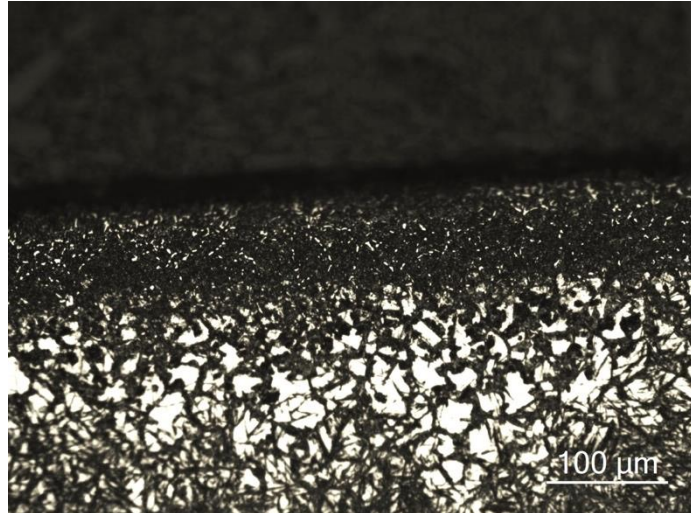
(A14) Specimen 1.1.2.1, villelas, x200, intermediate zone 2-3
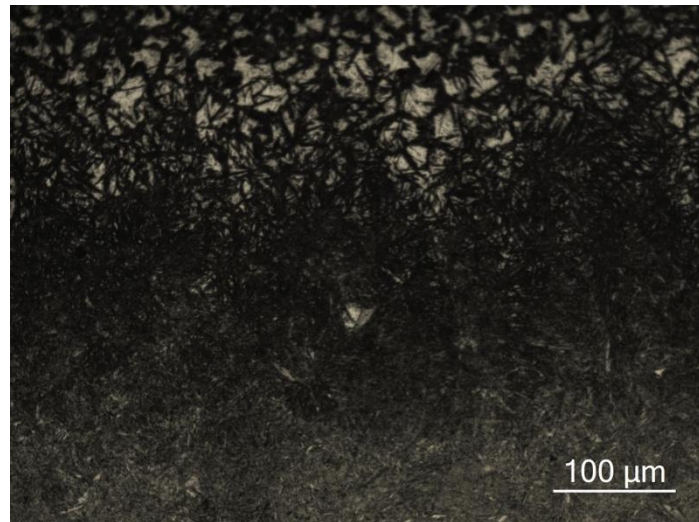
(A15) Specimen 1.1.2.1, villelas, x200, intermediate zone 3-4
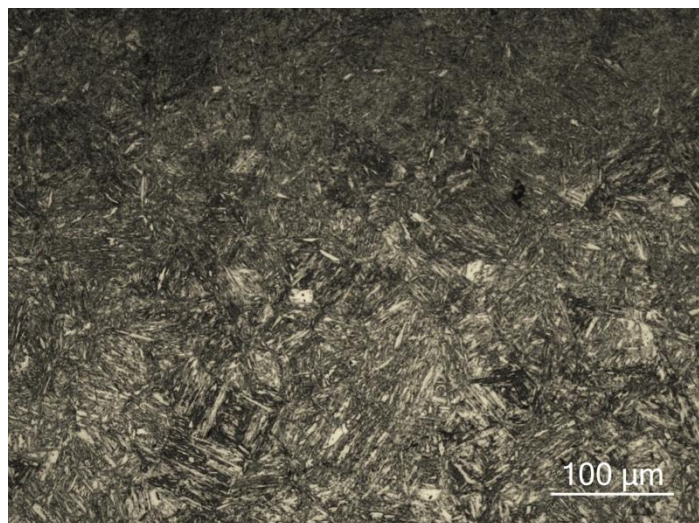
(A16) Specimen 1.1.2.1, villelas, x500, subtractive microstructure
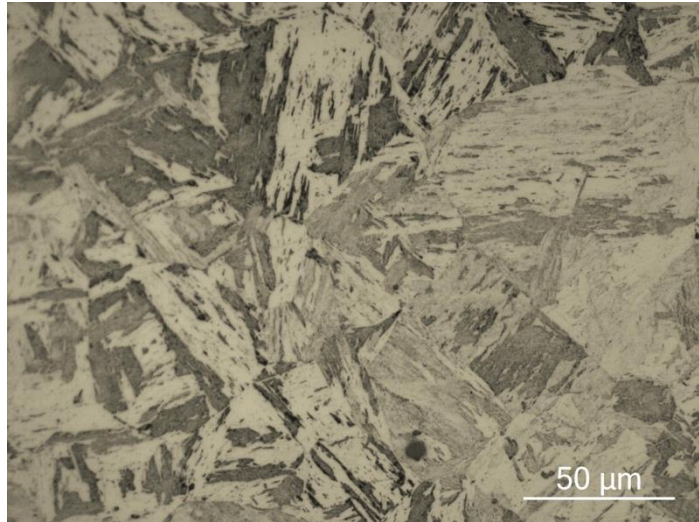
(A17) Specimen 1.1.2.1, villelas, x500, intermediate zone 3
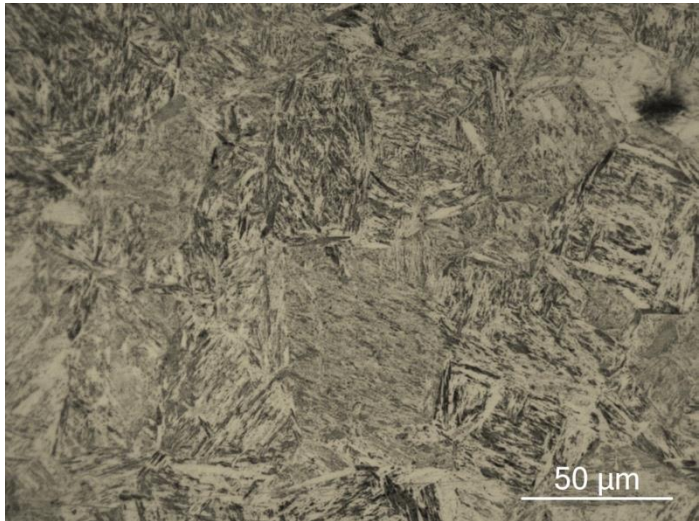
(A17) Specimen 1.1.2.1, villelas, x1000, intermediate zone 2
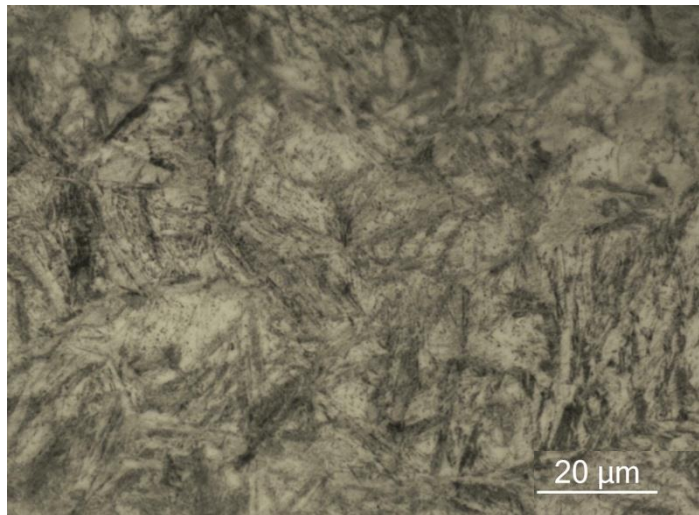
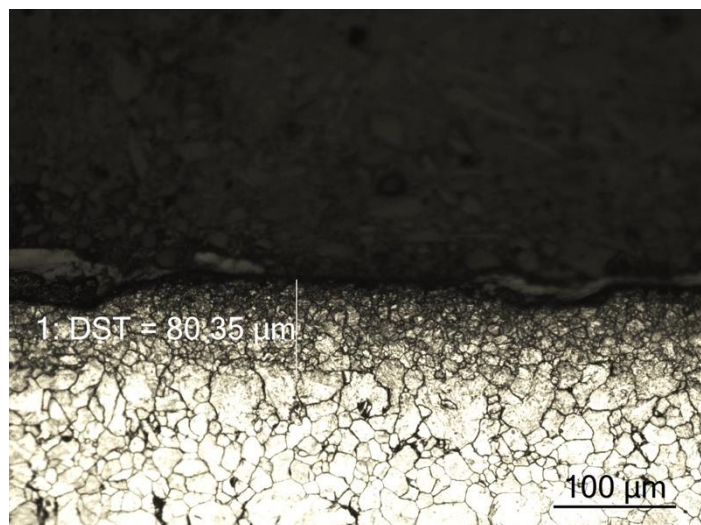**Figure 2.5.2: microstructure of the second heat treatment specimens**

(B1) Specimen 2.2.2, nital, x50,
carburized zone, total affected depth

(B2) Specimen 2.2.2, nital, x100,
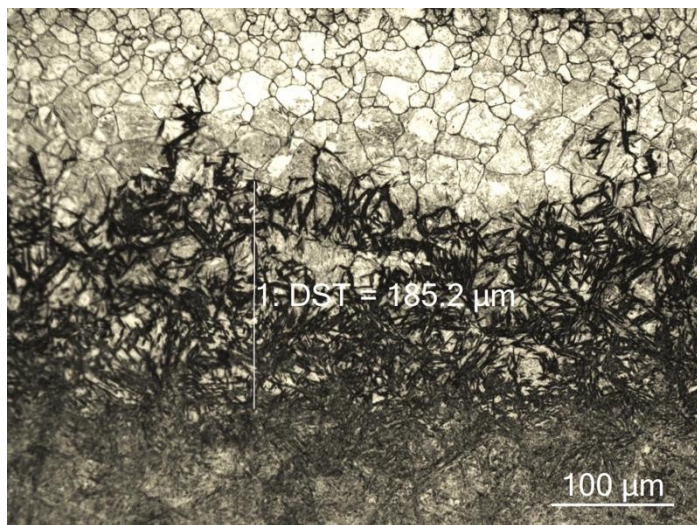carburized zone, total affected depth

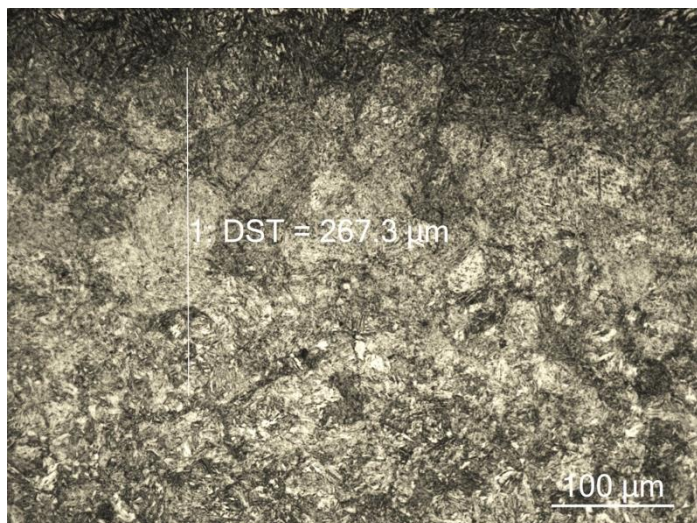(B3) Specimen 2.2.2, nital, x200,
intermediate zone 1-2-3

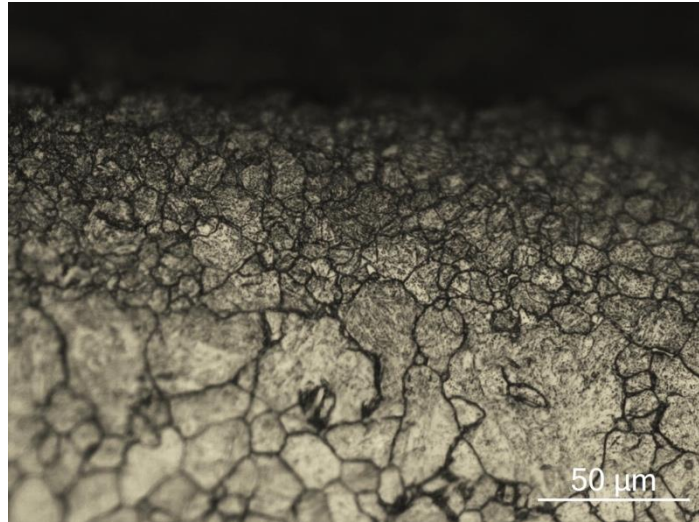(B4) Specimen 2.2.2, nital, x200, depth, intermediate zone 1-2
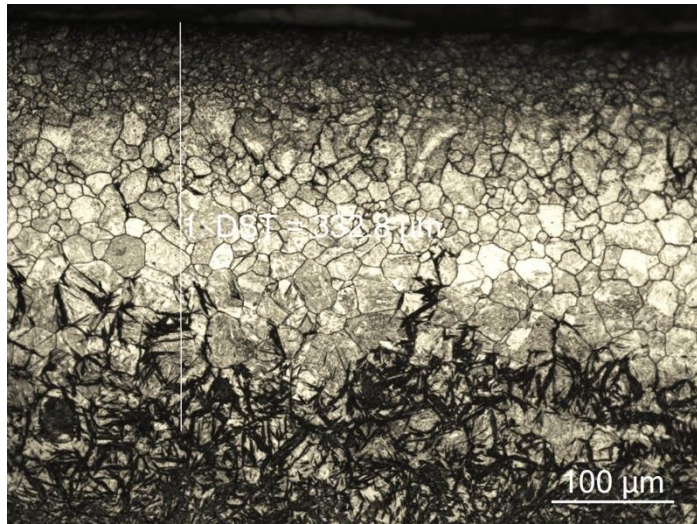
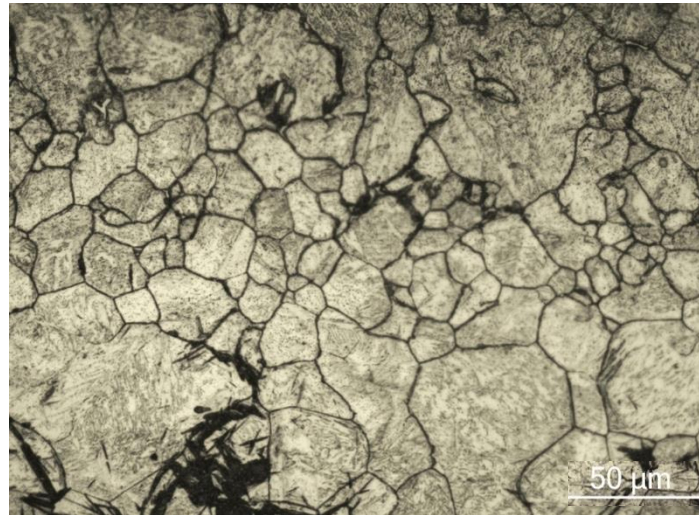(B5) Specimen 2.2.2, nital, x200, depth, intermediate zone 3

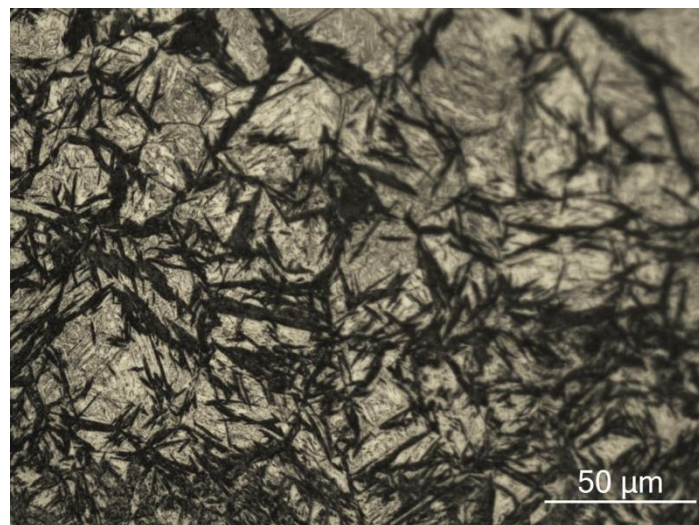(B6) Specimen 2.2.2, nital, x200, depth, intermediate zone 4

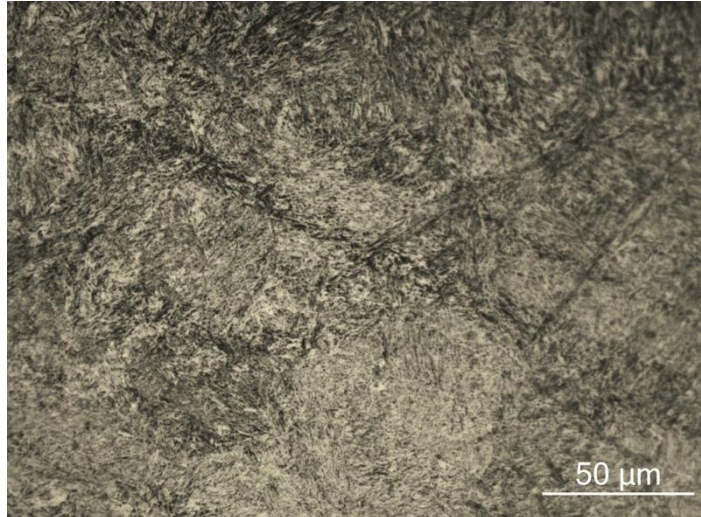(B7) Specimen 2.2.2, nital, x500,
intermediate zone 1-2

(B8) Specimen 2.2.2, nital, x500,
intermediate zone 2

(B9) Specimen 2.2.2, nital, x500,
intermediate zone 3

(B10) Specimen 2.2.2, nital, x500, intermediate zone 4

(B11) Specimen 2.2.2, nital, x1000, intermediate zone 1

(B12) Specimen 2.2.2, nital, x1000, intermediate zone 2

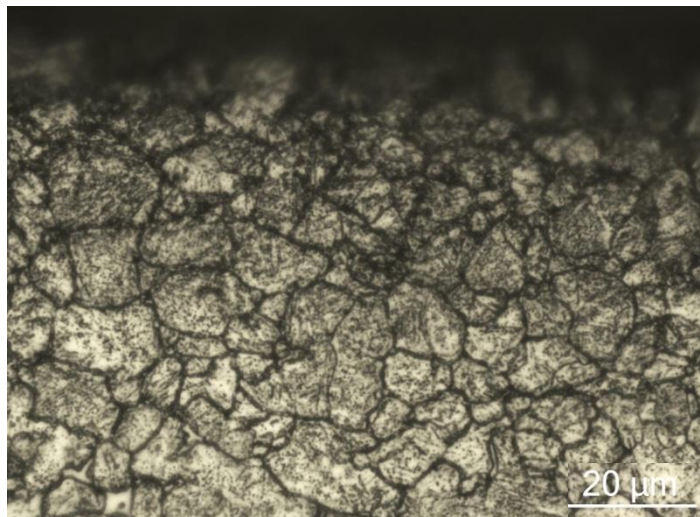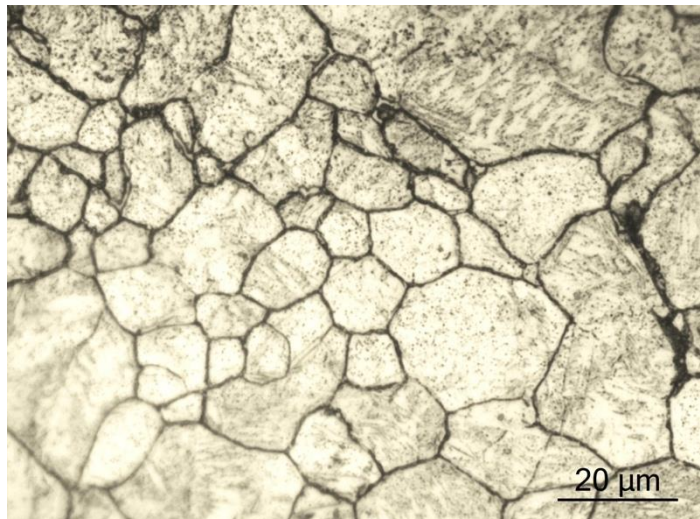(B13) Specimen 2.2.2, nital, x1000, intermediate zone 3
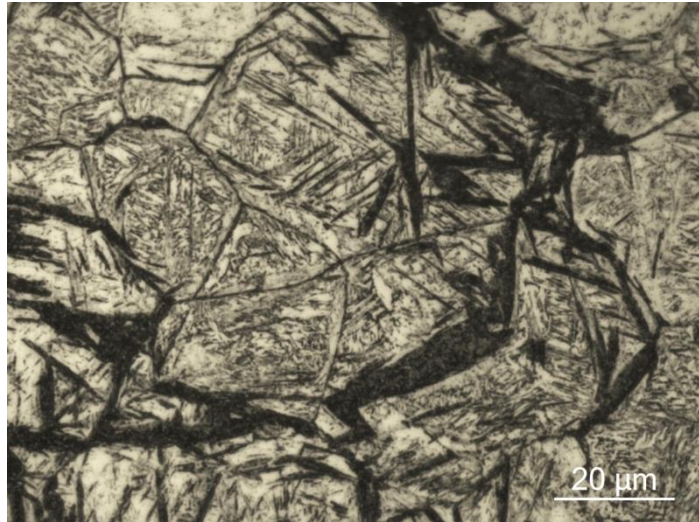
(B14) Specimen 2.2.2, nital, x1000, intermediate zone 4
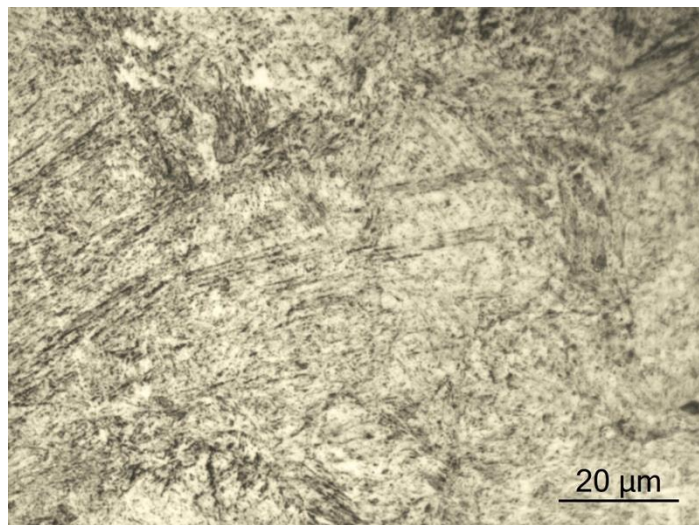
(B15) Specimen 2.2.2, nital, x1000, intermediate zone 1-2

Figure 2.5.3: microstructure of the third heat treatment specimens
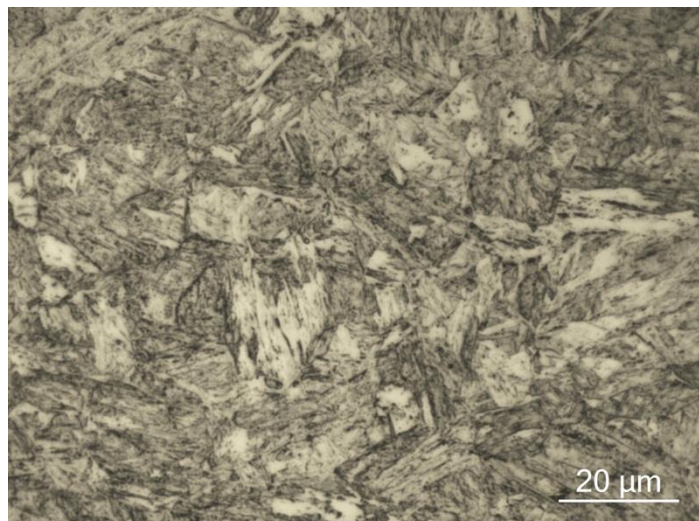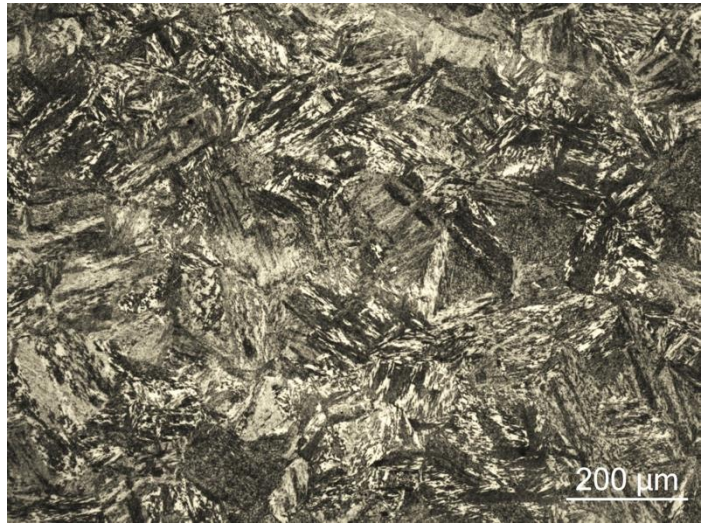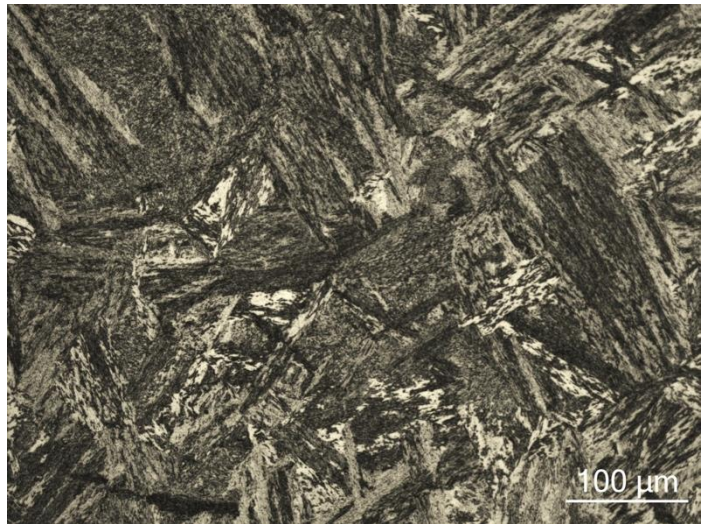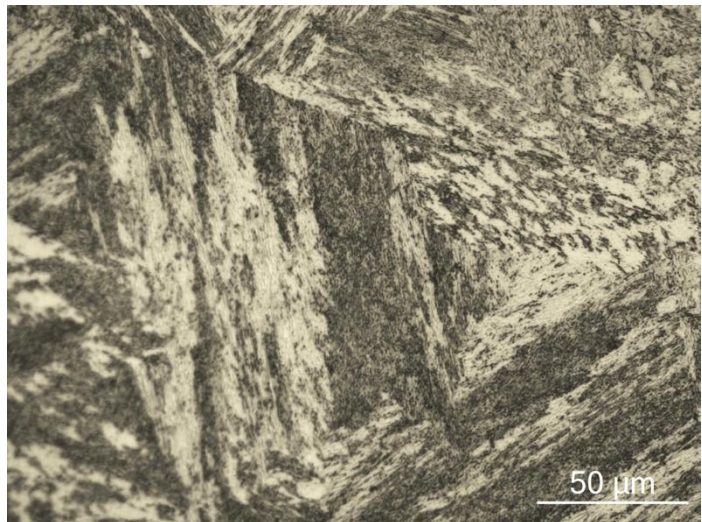
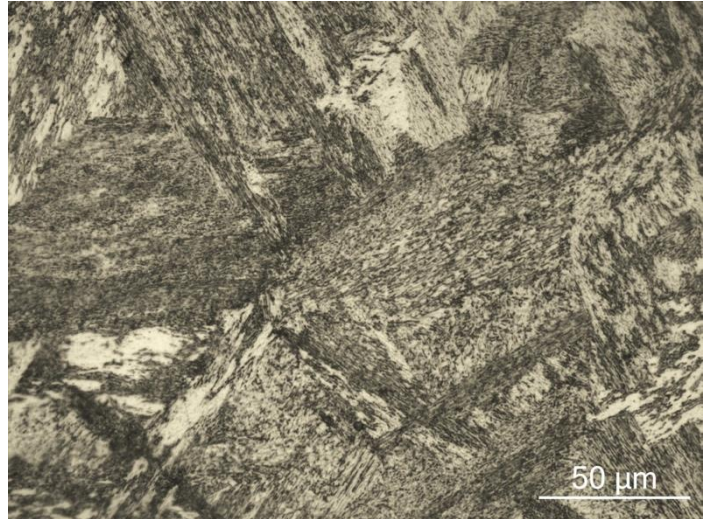(C1) Specimen 3.i.2.1, nital, x100

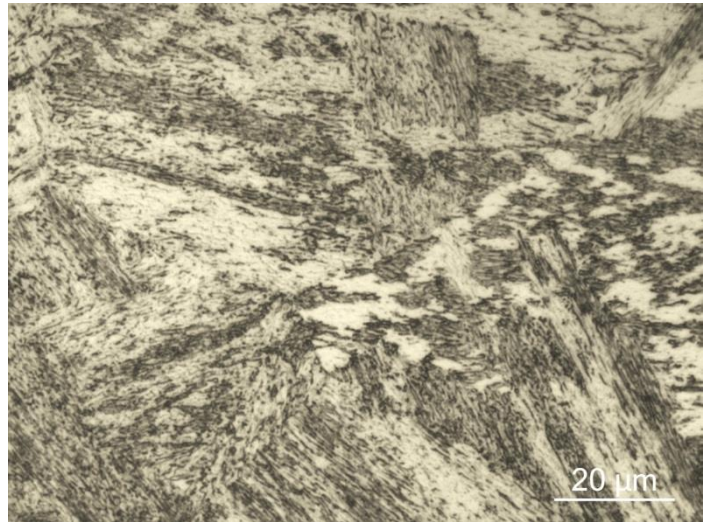
(C2) Specimen 3.i.2.1, nital, x200
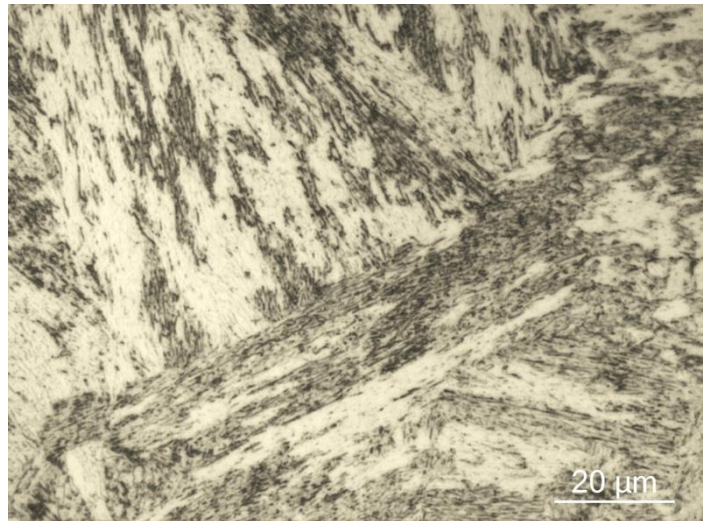

(C3) Specimen 3.i.2.1, nital, x500

(C4) Specimen 3.i.2.1, nital, x500

(C5) Specimen 3.i.2.1, nital, x1000

(C6) Specimen 3.i.2.1, nital, x1000

2.4. Results & Analysis

First heat treatment (Carburizing, quenched in water, cryotreatment, tempered)

Responsible for the remarkable increase in hardness in this steel is the martensitic transformation. After the tempering process, tempered martensite becomes the prevailing microstructure in the first layer close to the surface of the steel. However, in Figure 2.5.1 (A7), the fine grain structure and characteristic grain boundaries of the martensitic microstructure are not clearly observed. Instead, precipitation of carbides of Mo and Cr occurs at the grain boundaries, where the smaller ones are Mo carbides, and the larger ones are Cr carbides. The presence of these carbides is directly linked to the formation of the martensitic structure, which occurs due to rapid water cooling from 850°C to room temperature. The depth of the first subzone is approximately 110 μm. [35] Figure 2.5.1 (A4)

From the second subzone and below, a complicated grain structure is evident. The microstructure of the second subzone is lower bainite, characterized by even-sized grains without any signs of uneven grain size. The high temperature of 900°C for 180 minutes is responsible for determining the relatively coarse grain size in this subzone. The depth of the subzone is 148 μm. Figure 2.5.1 (A4)

The third subzone also comprises of bainite, but it differs from the second subzone in that it contains a higher percentage of cementite in the microstructure, rendering the third subzone more robust than the second with depth of 125μm. [35] Figure 2.5.1 (A5)

The fourth subzone consists of upper bainite, characterized by plates of ferrite and cementite with depth roughly of 126 μm. Figure 2.5.1 (A5)

The presence of martensite after heat treatment confers a significant advantage to this steel, allowing it to be used effectively following carburizing and quenching. This makes it suitable for applications involving mechanical parts with an equivalent diameter of 25mm and high demands on abrasion resistance. The presence of martensitic structure in this steel supports the assertion that carburizing and quenching in water is the reason for martensite formation, and the martensitic start temperature (ms) lies below room temperature. [35]

FIGURE 2.6.1 [38]

Fig.24.TTT diagram of Uddeholm Nimax made by JMat Pro modeling software.
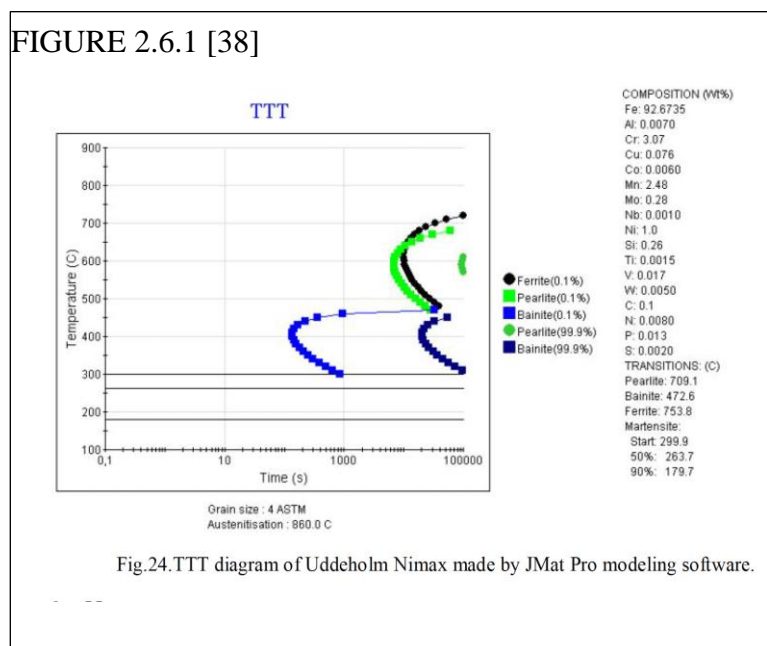
Second heat treatment (Normalizing and double quench hardening)

The second heat treatment also exhibits four distinct subzones where the microstructure has been altered. The total depth affected by the heat treatment measures approximately 816 μm. The microstructure of the metal is only bainite (lower and upper). [35]

In the first subzone, fine grains of lower bainite with 0.8% C are observed due to the 900°C for 180 mins. That had an effect of entering the austenitizing zone. That's why the first layer of the metal has fine grain size. The depth of the first subzone is approximately 80 μm (Figure 2.5.1 (B3)).

The second subzone displays an uneven grain size of lower bainite with 0.5 – 0.7% C, which can be attributed to the exposure 780°C for 30 mins. That has an effect of partial austenitizing so that's the reason for the uneven grain size on the second subzone. The depth of the second subzone is roughly 250μm Figure 2.5.1 (B3)). [35]

The third subzone is characterized by lower bainite and the grain size that is unaffected by the (Figure 2.6.1). The approximate depth of this subzone is 185 μm (Figure 2.5.2 (B5)).

The fourth subzone corresponds to upper bainite, and its depth measures approximately 267 μm (Figure 2.5.2 (B6)). [35]

It is important to note that the subtractive zone is no longer contains lower bainite in its microstructure.

During this heat treatment process, the grain size of the steel undergoes a significant reduction by three grades on the ASTM G scale ranging from 1 to 14. Initially delivered by Uddeholm at approximately grade 10, the steel's grain size is lowered to approximately 6.5 after the specific heat treatment. This downgrading has a notable impact on diminishing the fatigue durability of the steel, making it unsuitable for applications in molds and mechanical components.

The austenitizing process at 780°C results in partial recrystallization of the grains within the area of 0.5 – 0.7% C, at a depth ranging from approximately 80 – 280μm. This partial recrystallization negatively affects the fatigue durability of the steel, particularly near the surface, which is undesirable for molds and mechanical parts. Cooling the steel in water after austenitizing at 780°C poses a high risk of failure and breakage due to the uneven grain size distribution. Consequently, water cooling is strictly prohibited in this heat treatment.

Figure 2.6.2

Therefore, in order to proceed with the heat treatment, certain parameters must be modified as follows: Austenitizing at 860°C for 30 minutes, followed by isothermal cooling at 180°C for 20 minutes, and then air cooling. This adjusted heat treatment process can be safely applied for molds. Additionally, for smaller parts with an equivalent diameter of 20mm, the cooling process can also be performed using oil as the cooling medium.
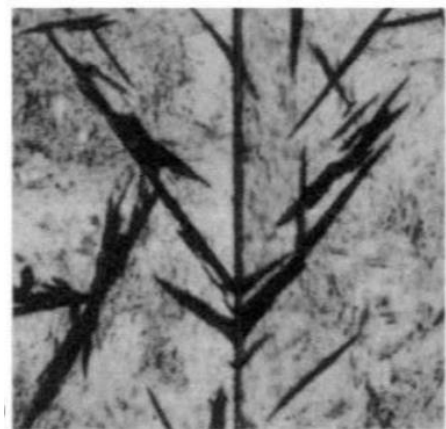
0.8C steel transformed at 300 ºC. Two surface composite micrograph, lower bainite structure x260

Third heat treatment (Normalizing)

The third heat treatment applied to the specimens primarily results in the formation of upper and lower bainite, composed of ferrite and cementite.(Figure 2.6.4) The white areas observed in the microstructure indicate the presence of ferrite. The lower bainite exhibits higher hardness compared to the upper bainite. Additionally, Figure (C1) reveals the presence of auto-tempered martensite with a small amount of austenite [36]. This particular type of martensite appears more rounded and softer than typical martensite due to the heat transfer from the specimen's center to its surface. As a result, the already formed martensite on the surface experiences prolonged exposure to heat, leading to its unique form. Scattered elliptical-shaped cementite particles can be observed in the images (C5, C6). [35]

In terms of microstructure, the heat treatment does not introduce significant differences compared to the original microstructure of the pre-hardened Nimax tool steel, except for the distribution of cementite within the bainite structure.

According to Uddeholm, the third heat treatment involves tempering at 700°C for 2 hours, which results in a reduction in hardness. Heating the steel to 850°C and then air cooling yields a bainitic structure with carbides. Slow cooling approaches achieve nearly equilibrium conditions, although the presence of small portions of martensite cannot be entirely ruled out. However, considering that this steel is produced in smaller quantities, the presence of martensite is minimal, or it could be in an auto-tempered state. This suggests that the martensitic finish temperature is significantly lower than 0°C, indicating a rapid transformation of austenite to other phases. [35]

Prior to the formation of eutectic ferrite, the cooling process transforms austenite into cementite. According to Uddeholm, heating the steel to 850°C and cooling it in circulating air constitutes a normalizing heat treatment.

Figure 2.6.4 [38]

'B1, low-carbon, acicular'   'B4, higher-carbon, massive'
'MA phase'
Carbide-free bainitic structures with phases of retained austenite and martensite ('MA phase')
(- - - - - prior austenite grain boundaries)

'B3', lower bainite   'B2', upper bainite
Angled intra-lath carbide
Inter-lath carbide
Low-alloy, FeC steels – the common forms of bainite with carbide distributions

Figure 2.6.3 [37]

a

b

Dominantly Bainite structure
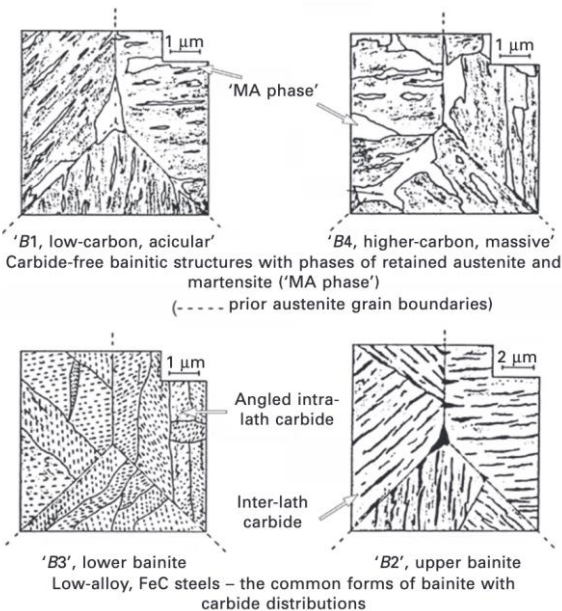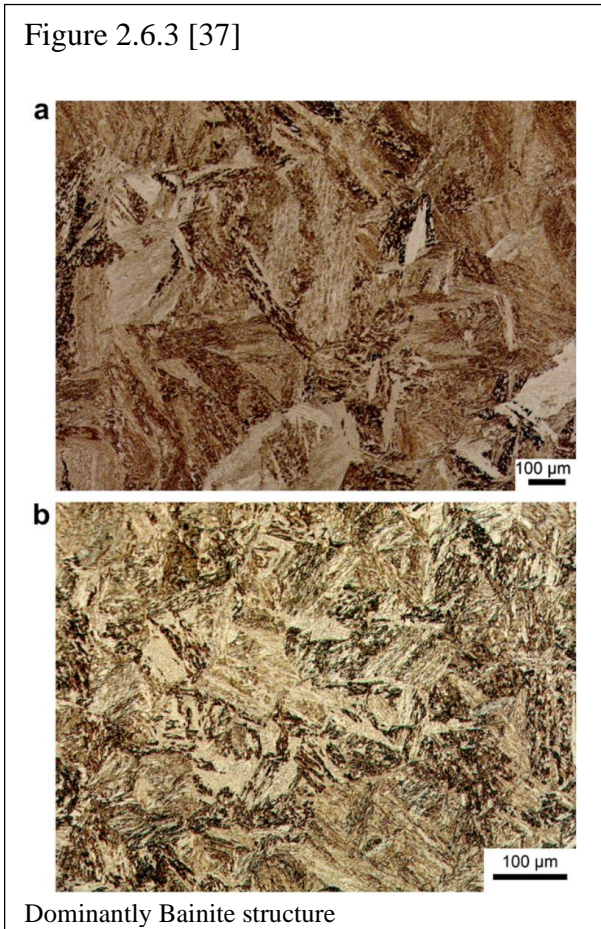
3. Conclusions

Plastic mold tool steel having the trade name Nimax, developed by Uddeholm, is an exceptional steel renowned for its unique ability to be hardened without compromising its other valuable properties. This remarkable feature is attributed to its well-balanced chemical composition and the meticulous manufacturing process.

Throughout its various heat treatments, Uddeholm Nimax has demonstrated outcomes that range from highly advantageous to potentially disastrous. Each heat treatment process unveils distinct effects on the steel's microstructure and mechanical properties, leading to outcomes that can either enhance its performance or adversely impact its suitability for specific applications.

Given the critical significance of this steel grade in industries relying on plastic mold tooling, it becomes imperative to thoroughly investigate and understand the consequences of different heat treatments. Such studies will pave the way for optimizing the heat treatment process and ensuring that the steel's exceptional properties are effectively preserved or enhanced while avoiding potential pitfalls that could lead to catastrophic results.

Conducting in-depth research and rigorous experimentation can unveil further insights into the intricate relationship between heat treatment, microstructure, and mechanical properties of Uddeholm Nimax tool steel. This knowledge is essential in unlocking the full potential of this special steel and in advancing its applications in various engineering fields. Thus, future endeavors should be devoted to investigating the effects of specific heat treatments to harness the maximum benefits of Nimax steel while mitigating any potential risks.

For future studies, it is recommended to conduct a comprehensive investigation into the detailed hardness distribution resulting from the two heat treatments. This would entail analyzing and documenting the hardness values across various depths and regions of the specimens to gain deeper insights into the effects of each heat treatment on the steel's mechanical properties.

Additionally, conducting standard tensile and bending tests after these heat treatments would be highly beneficial. These tests would provide valuable data on the steel's tensile strength, yield strength, and ductility, as well as its resistance to bending forces. By subjecting the specimens to these standard mechanical tests, a more comprehensive understanding of the steel's performance and behavior under different conditions can be obtained.

The combination of detailed hardness allocation and standard mechanical tests would enhance the overall understanding of how each heat treatment affects the steel's mechanical properties and would provide valuable information for optimizing the heat treatment process to meet specific application requirements. Such future studies could contribute significantly to the advancement of knowledge in this field and aid in the development of improved heat treatment strategies for enhancing the performance of the steel in various engineering applications.

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