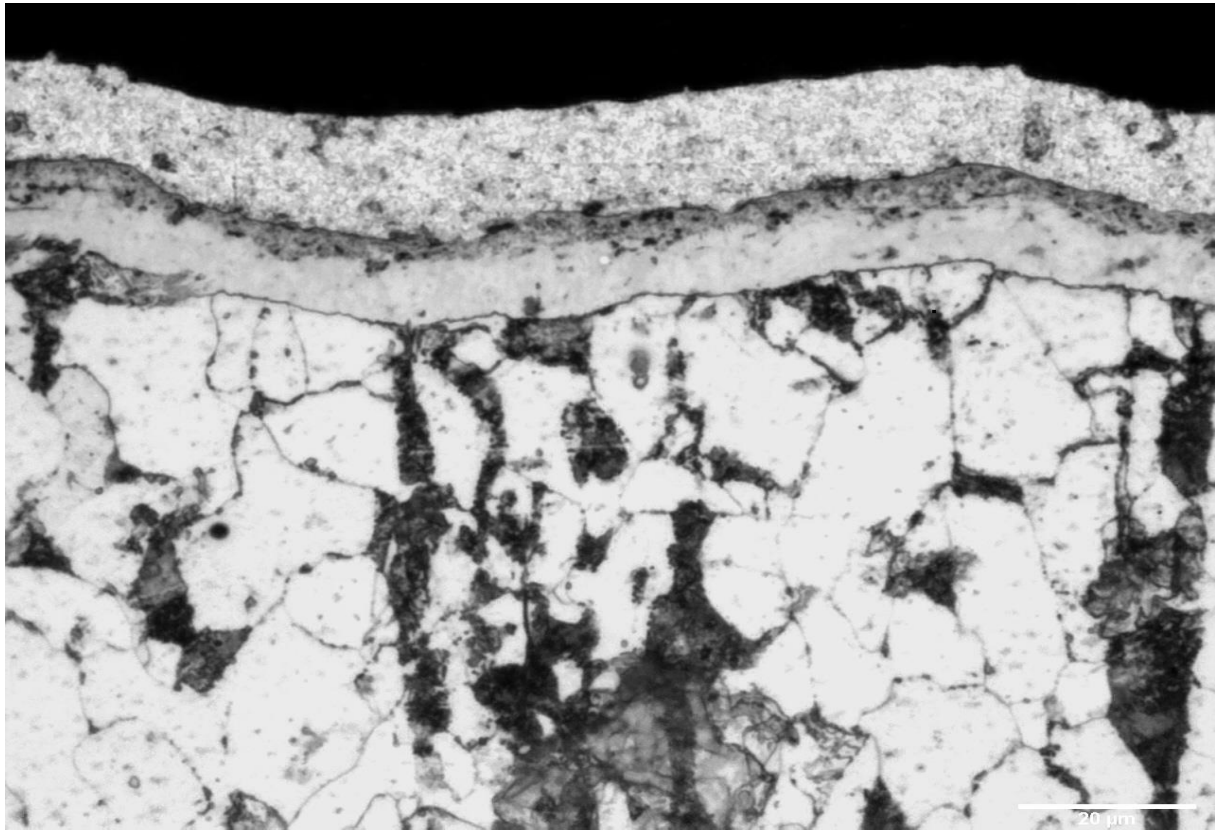




CHALMERS
UNIVERSITY OF TECHNOLOGY



Nondestructive Evaluation of Compound Layer on Nitrocarburized Steel

Master of Science Thesis

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Nondestructive Evaluation of Compound Layer on Nitrocarburized Steel

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Cover:
Surface microscopy image of a heat-treated structural steel sample,
500x, etched in Nital

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Abstract

In industry, the evaluation of process quality is a crucial component for providing up-to-task final components. The heat treatment services over steel components are no exception to that rule. Therefore, companies tend to give close attention to this final assessment. In fact, such a step would determine the quality of the final component studied along that of the process. In order to achieve such an assessment, companies tend to use conventional metallographic methods which seem to give an accurate insight over the properties of the studied component. However, these destructive ways provoke long lead times, a lot of scrap and a respective waste of money. Therefore, the use of a nondestructive evaluation seems like an attractive idea, as it would allow reducing all the inconveniences mentioned before.

Initially, this report includes a theoretical study about both nitriding processes and eddy current as a nondestructive evaluation technique of choice. Moreover, the study aimed at understanding the working principle of an already-deployed eddy current instrument. Consequently, the nondestructive equipment was used on a carefully chosen test matrix of steel samples. The main goal was to determine the compound layer thickness formed on top of several nitrocarburized steel components under different conditions. Eventually, these nondestructive evaluation values were compared to those of a metallographic study. Finally, the amount of information acquired from the literature survey and the nondestructive measurements was deployed to find other eddy current equipment suitable for the aforementioned application.

The results obtained from the experimental procedures allowed a better understanding of the working principle of eddy current as a nondestructive thickness measurement device. Also, the impact of several parameters over the compound layer thickness and porosity levels was determined. For instance, one cannot use different steel grades to determine their compound layer thickness with the same eddy current calibration. On the other hand, the porosity levels affect directly the accuracy of eddy current readings. Finally, finding a perfectly suited new eddy current instrument for compound layer thickness measurement was discovered to be somewhat complicated; as the existence of a nondestructive equipment with such application was deemed scarce.

Keywords - Nitrocarburizing, Compound layer thickness, Nondestructive evaluation, Eddy current, Frequency, Measurement methodology, Metallographic method, Porosity level

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List of Abbreviations

Acronyms	Signification
NDE	Nondestructive Evaluation
RISE	Research Institutes of Sweden
AB	Aktiebolag (Joint-Stock Company)
R&D	Research and Development
GDP	Growth Domestic Product
PLC	Public Limited Company
HSLA	High-Strength Low-Alloy
Al	Aluminum
Ni	Niobium
C	Carbon
N	Nitrogen
HV	Vickers Hardness
NH ₃	Ammonia
STD	Standard
SEM	Scanning Electron Microscope
ASNT	American Society for Nondestructive Testing
EC	Eddy Current
AC	Alternating Current
BN	Barkhausen Noise
SS	Swedish Standard
Ltd.	Limited
Inc.	Incorporation
GmbH	Gesellschaft mit beschränkter Haftung (Company with Limited Liability)
NDT	Nondestructive Testing
VBC	VärmeBehandlingen Centrum

1 Introduction

This section describes the content, purpose and scope of this thesis project as well as the presentation of the institutions where the thesis work took place.

1.1 Context

From an industrial point of view, steel is still considered to this day an extremely important material. It is used heavily in several industries due to its various advantages such as the low production cost, the high degree of recycling along its good strength and toughness. Nevertheless, these excellent properties for manufacturing can yet be enhanced using different types of heat treatment. This variety of processes helps providing the most adequate material with gained advantages beneficial for case to case scenarios. In this paper, the focus will be more dedicated towards processes which provide the industry with hard surface heat-treated steel. This type of processes allows the material to ensure good friction properties; an important characteristic for various applications.^[1]

No need to say, the final product in these industries must meet certain criteria to be functional and satisfactory to its task. Therefore, some testing methods are used to ensure that any previously-made goals have been met. Till this day, manufacturers still heavily rely on the use of metallographic ways. Unfortunately, these methods imply damaging the component and its future usability. Therefore, many manufacturers and researchers expressed their wish to replace such methods with nondestructive ones. Naturally, this would allow the further use of the components in question along saving both money and time; crucial parameters in the world of industry.^[1]

For the case of this project, the focus will be entirely dedicated towards the use of nondestructive evaluation (NDE) to assess the thickness of the outermost hardened layer of two different steel grades. The heat treatment process investigated upon in this project is explained in more detail in the following sections.

1.2 Presentation of the Institutions

The project duration was spent at two different institutes namely Research Institutes of Sweden (RISE) IVF at Mölndal and Bodycote Heat Treatment at Angered, Gothenburg. Both of which are presented below.

- **RISE Aktiebolag (AB)** is a network of Research and Technology centers owned by the Swedish State. The holding company takes in charge performing industrial research and innovation projects. It was founded in 1997 and became wholly owned by the State in 2007. Nowadays, the Swedish network constitutes of four corporate groups (Innventia, SP Technical Research Institute of Sweden, Swedish ICT and Swerea) with sixteen different Research and Technology organizations. This project was supported by one of these Research and Technology organizations known as RISE IVF in Mölndal, which focuses on the segment of Materials and Production.

^[2]

- ***Bodycote Public Limited Company (PLC)*** is a British company known as a supplier of different industrial services such as heat treatment, metal joining and coatings. It was created in 1923 as a textile business. Nowadays, the company is globally spread over 180 locations in 23 countries. This project was mainly conducted at Bodycote Heat Treatment in Gothenburg.^[3]

1.3 Purpose and Objectives

The purpose of this project is to assess the use of nondestructive techniques as a replacement to the conventional metallographic methods in nitrocarburized applications. This would allow the decrease of both cost and lead times associated with all the steps and implications metallographic testing imposes.

To reach such a goal, some objectives had to be met along the way. These objectives can be summarized as shown below. For more details, please refer to the initial project idea represented in appendix 01.

- Understand fully the working principle of the nondestructive instrument at Bodycote Heat Treatment
- Develop a full measurement methodology which would allow assessing any variation caused by the instrument or the operator
- Suggest a replacement for the nondestructive instrument at Bodycote
- Conduct the planned test measurements using both nondestructive instruments
- Define clearly the nondestructive instruments respective accuracy and precision using a statistical software tool
- Conduct conventional metallographic inspection over chosen samples and compare the reference values to the measured ones
- Comment on the efficiency of the measurement methodology used in the project

1.4 Delimitations

The project had the short timeframe of five months and therefore the focus had to be exerted over the essential tasks only. Consequently, the following delimitations have been identified.

- The project did not include any lab-made oven and therefore there was not a big control over the process variables. Hence, samples had to be placed in actual ovens with other clients' steel components at times and alone during others.
- The environment where the nondestructive instrument was held was not very suitable; since the workshop had a lot of noise and vibration due to constant movement of the personnel along that of the components' charges.
- The lack of manufacturers of nondestructive instruments with that specific application restrained the capability of getting any in-depth information about the working principle.
- The lack of actual numbers of both conductive and magnetic properties differences between the outermost layer and the rest of the material was also of concern.

1.5 STEEPLE Analysis

STEEPLE Analysis is a decision model made of seven factors which are directly linked to the assessment of various fields. Through this model, the project is evaluated based on the impact it has over society, economy and environment. Such analysis allows understanding and assessing the value and idea of this project.

Social - The improvement of the techniques used at the industrial level is necessary to keep up providing the society with better and less expensive final products. This project aims at using a certain nondestructive technique that would further allow that.

Technological - The continuous improvement of techniques used in industry is crucial to keep up with the never-ending technological progress within the heat treatment domain. This project aims at demonstrating that heat treatment industry can benefit of nondestructive techniques as it may well become a necessity for future competition.

Economic - An improvement in a specific application is aimed to lower both the cost and lead times of the process. Such a project has the goal of lowering the expenses of the company during process quality control. This goal is set as a priority, yet the introduction of this nondestructive technique must answer to the same standards of process quality control as with conventional methods.

Environmental - This project is also dedicated to lower the amount of scrap from the quality evaluation process along reducing the necessity for recycling. As a matter of fact, the environmental aspect is at the very core interest of this thesis work.

Political - The benefits of such a project are supported by the industry as well as the national research centers due to its various advantages. As for the Swedish government, the ministry of Enterprise and Innovation openly defends the development of the national innovative capacity for the sake of keeping up with future innovation in general. ^[4]

Legal - As for the legal system in Sweden, a huge importance is given to research and development (R&D). In fact, the country spends around 3% of the country's Growth Domestic Product (GDP) over R&D. This number proves that laws are strongly dedicated to promoting the continuity of innovation within various sectors such as industry and naturally process quality evaluation as well. ^[4]

Ethical - As an engineer, it is necessary to always keep in mind that improving the techniques used by the society and industry is a priority. This project mainly focuses on doing so with a specific industrial method in such a way to preserve both the economic benefit and the environmental sustainability.

2 Literature Survey

In this part, a straight to the point theoretical background is given. It introduces the main information needed to back up the understanding of the following sections. In the first part, the focus will be dedicated to the chosen types of steel and the respective heat treatment process of interest in this project. On the other hand, the second part will present a general understanding of the process quality evaluation from the metallographic methods to the nondestructive ones.

2.1 Steel and Heat Treatment

2.1.1 Types of Steel

Overview - The properties of steel are to this day considered excellent for several industries. Nevertheless, these properties can always be enhanced to provide a final component designed for a specific application. Therefore, industries dealing with steel came up with a clear classification of different types of steel. This classification contains categories of steel in which materials with closely common properties are gathered. Based on the different applications, these steel categories can be expressed as the following. ^[1]

- Structural and Heat Treatment Steels
- Tool Steels
- Stainless Steels
- Steels for Electrical and Magnetic Purposes

Regarding the scope of this project, the focus will be solely dedicated to the structural and heat treatment steels category.

Steels of Interest - The structural and heat treatment steels are mainly used for construction, load bearing applications and machine parts. Naturally, the steel types within this category have to some degree common properties. Yet, some of these are prioritized in some cases depending on the intended area of use of each steel grade. Undoubtedly, this steel category is considered the largest as it has a broad application area. For instance, building constructions is considered the most suitable area for the use of structural steels. On the other hand, machine parts such as gears, rollers, springs along piston rings are more dominated by heat treatment steels. However, they have been put in the same category as they can be used interchangeably at some occasions. ^[1]

Based on the respective yield stress limit, chemical composition and application field, this category has been divided into several steel subcategories. Those of interest to the understanding of this project are shown in table 01 below.

Table 01 - Classification of structural and heat treatment steels ^{[1][5]}

Designation	Description
Structural Steel	This type of steel is mainly used in the domain of construction as it can be shaped into a wide range of forms. As a matter of fact, it is well known for its good machinability and weldability. Therefore, it is used a lot in both commercial and industrial construction applications. Naturally, this subcategory of materials follows a well-regulated standard system which differs from a country to another. ^{[1][5]} The benefits of this low carbon steel are what explains its wide spread across construction applications. These advantages could be summarized in the following. First, it is undeniably easy to find as raw or pre-made material and relatively lightweight. Second, the high degree of recyclability makes it very attractive for eco-friendly constructions. Then, its high strength-to-weight ratio helps it withstand strong impacts. Finally, this type of steel is also known for a long service life which makes it economically viable in construction applications. ^{[1][5]} Many High-Strength Low-Alloy (HSLA) steels belong to this subcategory and are usually alloyed using Aluminum (Al) or Niobium (Ni). ^{[1][5]}
Quenched and Tempered Steel	This type of steel is known for being a medium carbon one which passes through hardening and subsequent tempering before service. This is done at very high temperatures which allow the final component to have good strength and toughness under a martensitic microstructure. ^[1] This subcategory includes both non-alloy and low-alloy steel grades. Moreover, this type of steel is frequently used in components dedicated for case or surface hardening. The reason behind that is due to the ease of austenitizing in relatively short periods. However, it is important to stress that the tempering temperature used after hardening is crucial in defining the hardness, strength and impact toughness of the final component. ^[1]
Case Hardening Steel	This type of steel has a low carbon content of a maximum of 0.25%. It is usually described as construction steel with steel grades differing in alloying elements. ^[1] This subcategory is designed to result in a high surface hardness and good wear resistance along a soft tough core capable of absorbing stresses due to high surface tension. With an accurate chemical composition and alloying elements, the exposure of steel from this subcategory to hardening using Carbon (C) or/and Nitrogen (N) should lead to the previously mentioned advantages along improved fatigue strength. ^[1]

Nitriding Steel	This type of steel gathers into consideration both the chemical composition and the intended process parameters to achieve the desired properties in the final component. These properties include strength, hardness along wear and corrosion resistance of the uppermost surface. ^[1] This type of steel is dedicated specifically for nitriding and nitrocarburizing processes. As a matter of fact, any steel type can be nitrided in principle. Yet, for a great deal of accuracy some industries prefer the use of this specific subcategory. Steels from this subcategory tend to be relatively expensive and therefore are not produced in large numbers. This explains the reason behind their scarcity as they represent only 5% of the volume of nitrided steel worldwide. ^[1]
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2.1.2 Heat Treatment Processes

Overview - Heat treatment processes involve changing the properties of materials such as metals using heat. It can be used for either hardening or softening and eventually changing the material properties of the component in question. Both the heating temperature used and the quenching one play a crucial role in determining the type of transformation. Moreover, the tempering temperature is considered a vital component in determining the final output properties as well. ^{[1][6]} Nowadays, several steels in the industry sector are being heat treated to enhance the performance of the final output along increasing the quality of the end-line product. These heat treatment processes can be divided into two categories; thermal processes and thermo-chemical ones. As the name suggests, thermal processes include basically the series of heat treatment which enhance the properties of steel through subjecting the component to a set of temperature change sequence only. For instance, through hardening, induction hardening, and flame hardening are all considered thermal processes. On the other hand, the thermo-chemical processes include thermal change along a chemical reaction on the uppermost surface of the component. In most of cases, the chemical reaction occurs between the surface of the component and an external environment. This causes a change in the chemical composition on the surface of the steel. The thermo-chemical processes include for example carburizing, carbonitriding and nitriding. ^[1]

Nitriding Processes - Regarding the scope of this project, the focus will be specifically dedicated to a thermo-chemical process known as nitriding. During nitriding processes, N or/and C is/are transferred to the surface of the steel through a bearing medium. The process medium adopted can be gas, salt or plasma. This type of heat treatment is used to increase the load bearing capacity and fatigue strength while causing negligible distortion on the base material. Such a process results in changes on the surface of the steel component in such way that the very top layer becomes way harder than the rest of the component. This upper layer is known as the “compound layer” and is comprising of both γ' and ϵ phases. These phases have respectively the following formulas; Fe_4N and $\text{Fe}_{2-3}(\text{N}, \text{C})$. This upper layer is also known as the “white layer” due to the excess of the ϵ -phase which appears to be white during microscopy

observation. Beneath the compound layer, a diffusion region forms and decreases in hardness as it gets deeper into the material. This layer is best known as the “diffusion zone” where the diffused N remains further down as a solid solution, interstitial one or in the form of nitrides. ^[1] In figure 01 below, a microscopy image of the surface of a specific type of steel after being subjected to one of the nitriding processes is presented for visual clarification.

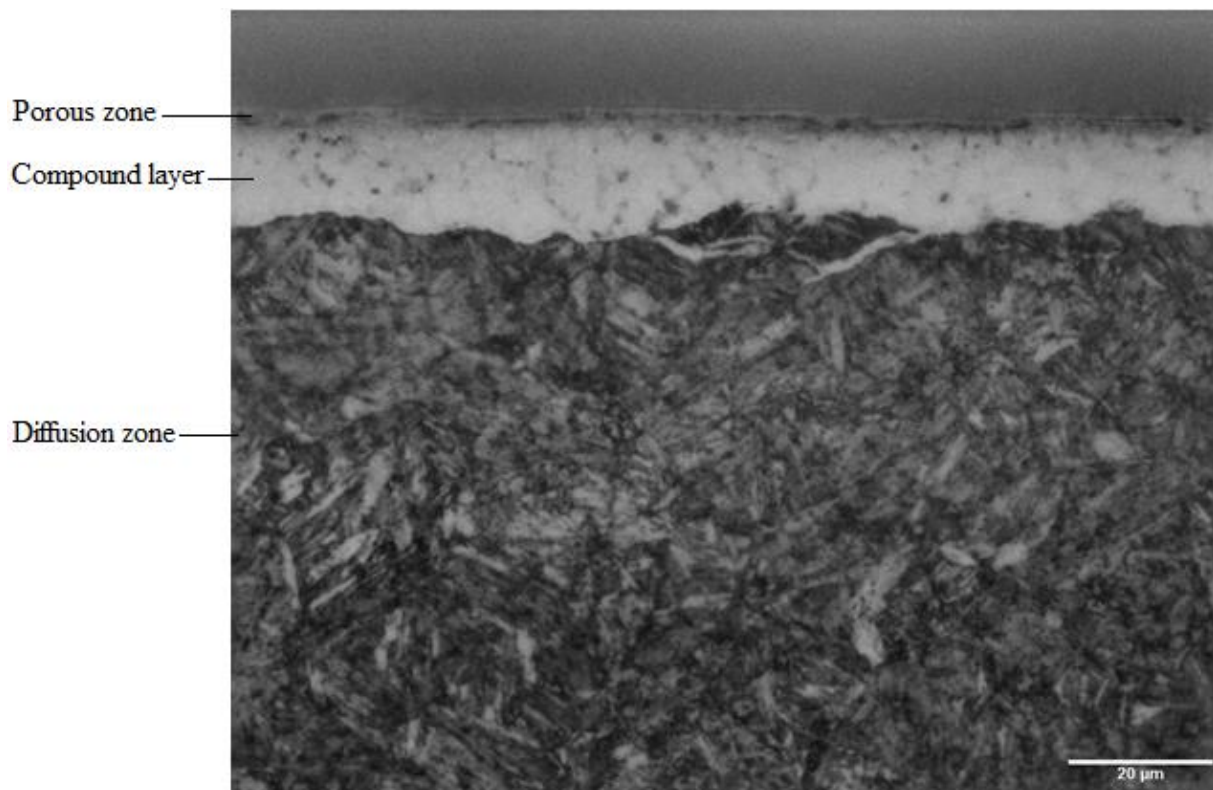


Figure 01 - Surface microscopy image of a heat-treated quenched and tempered steel sample, 500x, etched in Nital

General Principles - Each of the compound layer and the diffusion zone contributes differently in improving the properties of the steel in question. For instance, the compound layer allows an improvement of tribological and corrosion properties such as friction and resistance to wear. On the other hand, the presence of a diffusion zone helps improve fatigue strength and dimensional stability. Moreover, nitriding processes are known for causing minimum distortion in the final product, since they are classified as being low temperature procedures. As a matter of fact, nitriding processes have a heat treatment temperature below that of austenite transformation which implies the absence of any major changes in the base material. Consequently, even the production cycle becomes shorter than in high temperature processes as there will be no need for any dimensional correction measures. Low temperature processes such as nitriding also allows clean final parts, low gas consumption, limited emission and therefore can be judged as more eco-friendly processes than high temperature ones. ^[1]

Physical Principles - During nitriding processes, the transfer of C or/and N from the process medium to the component surface occurs in two main steps; diffusion through the compound layer and into the diffusion zone. On one hand, the formation of the compound layer follows several steps. First, a set of grain nucleations (γ') begins on the surface of the component. Then,

the grains continue growing further down till the ϵ -phase start to form on the surface. By the end, the result will ideally be an uppermost layer with ϵ -phase close to the surface and γ' -phase below it. Both these layers are what form the compound layer.^[1] However, it is necessary for process quality to put into consideration the porosity levels of this uppermost layer as it is deemed essential to assess the suitability of the final component. This porous layer appears mainly at the upper part of the compound layer. It results from the high N content after nitriding which after being introduced to the steel surface starts joining N atoms and forming N_2 gas. The pressure caused by this phenomenon ends up forming channels for the N_2 gas to exit the surface of the steel which results in leaving pores behind. It is also important to point that the porosity level present in the compound layer is linked primarily to the furnace temperature and chemical composition of the steel used.^[1] For a detailed visual description of what was mentioned above, please refer to figure 02 below where the formation stages of both compound layer and porous zone is shown.

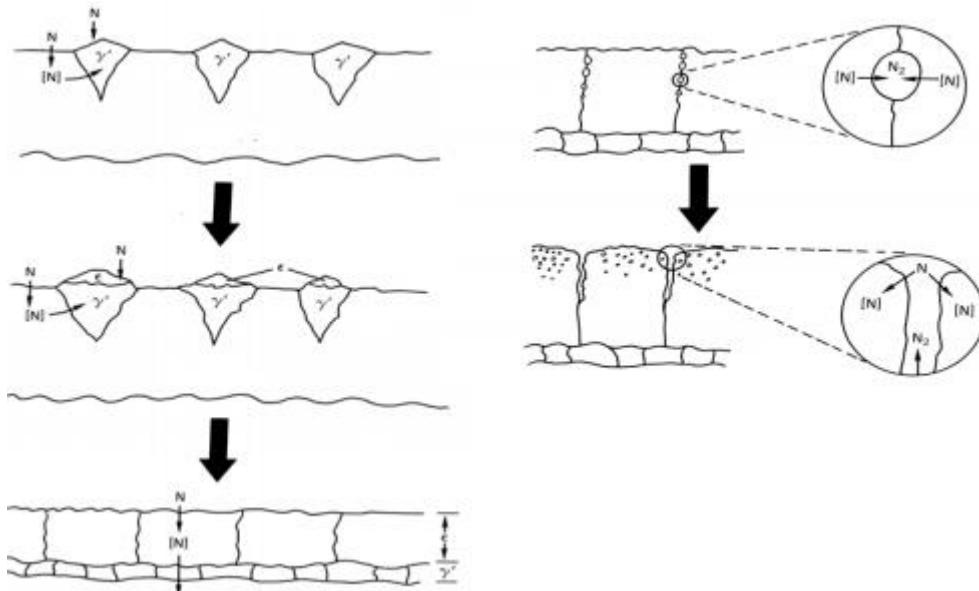


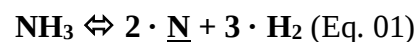
Figure 02 - Formation stages of compound layer (left) and pores (right)^[7]

Regarding the diffusion zone formation, the depth depends on the capacity of the N to infiltrate into the steel component. This is dependent on the process temperature and duration along the steel grade used. Concerning non-alloy steels, a rise in the furnace temperature leads to an increase of the N content penetrating the steel surface. Consequently, a fast cooling rate allows the N to stay in solid solution, while a slow one would cause the N to precipitate as nitrides of the alloying elements in the base material. This hardening mechanism is known as solution hardening. On the other hand, alloy steels are dominated by precipitation hardening. During nitriding processes, precipitates form at process temperature, but they are not affected a lot by the cooling rate. These nitrided precipitates of the alloying elements lead to an increase in the hardness of the diffusion zone. However, it is important to clarify that an increase in holding temperature does not necessarily lead to a greater hardness in the diffusion zone. It is rather related to multiple other factors such as the quantity and size of nitrides that form while subjected to that specific temperature.^[1]

General Evaluation Guidelines - Regarding the transformation that occurs on the surface of the nitrided steel, three major aspects decide the suitability of the final component. First, one must consider the thickness of the compound layer and the diffusion zone. Basically, the intended thickness varies based on the process parameters such as heating temperature and quenching medium along the steel grade used. It is also proved that the carbon content and alloying elements have respectively direct and inverse impact over the final compound layer thickness. As a matter of fact, this aspect is considered vital in determining the usability of the final component for its specific application. Moreover, it is reported that a porous zone starts forming at the top of the compound layer and is necessary in order to determine to assess the suitability of the final component. Finally, the hardness of both the compound layer and the diffusion zone is also considered crucial to determine. In fact, these values give an insight on the component ability to withstand the friction demands and contact fatigue of the intended application.^[1] Regarding the scope of this project, the interest is entirely dedicated to the compound layer; both thickness and porosity level.

Separate Processes - In literature, nitriding processes generally include both nitriding and nitrocarburizing heat treatment procedures. These two processes contain some differences which are discussed in more details in the following paragraphs.^[1]

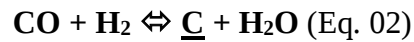
Nitriding - A thermo-chemical process which entails the use of N to diffuse on the surface of the steel in question. The process has a holding temperature of 500-550°C which results in a compound layer ranging from 0-20 µm and a diffusion zone of 0.5-0.8 mm thickness. On the other hand, the surface hardness of the final component can range between 350 and 1300 Vickers Hardness (HV). Due to the slow diffusion rate of N, this process can take from 5 to 100 hours depending on the intended surface hardening depth. Moreover, nitriding allows several medium choices such as gas, salt bath or plasma. For the scope of this project, the focus is solely dedicated to the gas process. Basically, this type of process is conducted in a closed and rich ammonia (NH₃) atmosphere. The NH₃ is used as an N-source and considered to be the best bearing medium. In the case of gas nitriding, the NH₃ reacts with the surface of the steel component and results in a chemical breakdown as shown in the equilibrium equation 01 below.^[1]



Nevertheless, this gas is considered toxic and hazardous therefore the nitriding furnaces must be extremely well sealed. Moreover, NH₃ becomes explosive if ever mixed with Oxygen (O) and therefore close attention must be dedicated to remove all the O present in the furnace.

Nitrocarburizing - A thermo-chemical process which entails the use of N and C to diffuse on the surface of the steel. This process has somewhat similar properties to the nitriding one and implies the transfer of N in a similar manner. However, this process entails -in addition to N- the transfer of C through a bearing medium during a much lower holding time period. This medium can either be Carbon monoxide (CO) or Carbon dioxide (CO₂). In most cases, companies prefer using CO₂ as it is more economically

viable. During nitrocarburizing, the CO or CO₂ reacts with the surface of the steel component which results in a chemical breakdown as shown respectively in the equilibrium equations 02 and 03 below. ^[1]



The nitrocarburizing process is largely like nitriding one, yet there are some differences in the process parameters. These parameters have been summarized in table 02 below. ^[1]

Table 02 - Usual process parameters of nitrocarburizing ^[1]

Process	Holding Temperature (°C)	Process Time (Hours)	Compound Layer (µm)	Diffusion Zone (mm)	Surface Hardness (HV)
Nitrocarburizing	550 - 580	0.5 - 5	0 - 30	0.5 - 0.8	350 - 1300

Quenching - One of the most important factors in the nitriding processes is the quenching medium as it has a huge impact over the microstructure, residual stresses and strength of the final component. Nowadays, there exists a variety of quenching mediums used in industry such as salt solutions, water, oil, gas, air or even water-based polymer solutions. The use of these different quenching mediums can result in major differences in the cleanliness of the final component surface, the environmental impact, along the compound layer thickness. This is mainly due to the difference in the quenching capacities of the available quenchants, the uniformity of the cooling rates along their respective stickiness and viscosity. ^[1] In this project, the quenching processes of interest are the ones carried out by oil or gas. These quenching types are discussed separately in more detail below.

Quenching by Oil - This type of quenching uses mostly mineral oils. Nevertheless, an ongoing investigation is taking place to replace that with vegetable oils for environmental purposes. This quenching medium can be classified into three categories shown below along their respective applied temperatures. ^[1]

- Rapid quenching oils applied at 60°C
- General quenching oils applied between 60 and 130°C
- Hot quenching oils applied between 120 and 200°C

In some cases, the mineral oils used for quenching are refined first. They are usually given additives to improve the wetting capacity, cooling one and washing properties. This allows ensuring better oxidation stability and therefore a longer service life. ^[1]

Quenching by Gas - The cooling capacity of this type of quenching differs based on the choice of gas. As a matter of fact, this quenching type can use a variety of gases such as

Argon (Ar), N, Helium (He) or H. Naturally, the properties of the final component differ from the use of one gas to the other. Nevertheless, this type of quenching in general results in better surface cleanliness than others. Also, it would be important to point out that a slow quenching using gas method is deemed beneficial through the martensite transformation range. Moreover, gas quenching allows a greater control over the cooling sequence since it is possible to control both pressure and flow rate if necessary. ^[1]

2.2 Evaluation of Process Quality

This part is dedicated to clarifying the different methods used to correctly investigate the quality of a nitrocarburized steel specimens. It considers describing the assessment of the following; the compound layer thickness and the porosity levels formed at its upper part. This can be done conventionally using metallographic methods or/and nondestructive ways through NDE. For clarification please refer to the following sections.

2.2.1 Metallographic Methods

Overview - Metallography is usually described as the study of the properties of a certain metallic component. As a matter of fact, both the microstructure and the appearance are of concern when it comes to metallography. Nowadays, metallographic evaluation is heavily used in industry for quality assurance either for validation or verification. The most conventional way to assess a metal component is through microscopy, which naturally requires the preparation of the samples to be assessed. The choice of the microscope type and magnifying capacity relies primarily on the nature of the intended assessment. ^[1] For the sake of this project, the focus is solely directed towards optical and scanning electron microscopy.

Sample Preparation - To prepare a sample for metallographic study, it is necessary to follow several steps. These steps must imply following the correct order along some precision for adequate component assessment by the end. First, a small cross section piece is cut away from the main component. In the case of heat-treated surface, the piece is wrapped in Al foil. This would constrain the loss of any layer on the surface of the piece due to subsequent hot mounting. Then, the small part is placed in an automatic mounting press along polymer materials such as Bakelite or epoxy resins. Consequently, the piece along the polymer material is heated and cooled under a certain pressure. The resulting mounted specimen is then rinsed with water, grinded and eventually polished using alcohol. For better polishing, one can put the mounted sample in an ultrasonic cleaner. As a matter of fact, polishing is considered a crucial step for metallographic analysis and therefore must be given a great deal of attention. The result is ideally a very shiny sample which is difficult to examine using microscopy. Therefore, etching comes at play to enhance the reflection and reveal microstructural features. As for the etchant, the most commonly used liquid for steel process quality check is 1-25% nitric acid in ethanol or simply Nital. It helps revealing steel cracks, segregations and even porosities. After all these steps, one can check the mounted sample using microscopy and get satisfying results. ^[1] For an example of mounted specimens, please refer to figure 03 below.



Figure 03 - Image of two hot mounted specimens

Optical Microscopy - After sample preparation, the mounted, polished and etched specimen can finally be examined using microscopy. Using optical microscopy, the mounted sample cross section can be magnified between 50 and 1000 times. One must pick the suitable magnification for each respective application and bear in mind that the higher the magnification the lower the resolution. In principle, optical microscopes use reflected light to picture the prepared sample. In short, a light source is directed towards the sample using a mirror and reflected to either the user's eye or into a camera. This simple physical principle is what allows obtaining an insight over the characteristics of a certain component. ^[1]

Scanning Electron Microscopy - A microscopy method used to characterize metals with a much higher possibility of image magnification (over 100000 times) than the optical one. The resolution depth of a Scanning Electron Microscope (SEM) is high and uneven surfaces can be studied as well. In most cases, SEM is used for the detailed imaging of microstructures but can also be supported by a chemical characterization. Regarding the physical principle, SEM allows a high magnification imaging through an electron beam scanning the surface of the sample. During that scanning, different electrons are detected and interpreted into a final image. ^[1]

Evaluation of Nitriding Results - To assess the results of a nitriding application over steel, two main aspects must be focused on in general. These two aspects represent basically the compound layer and the diffusion zone. Regarding this “white” layer, one must take into consideration two properties which are its thickness along the porosity level that forms on the top of it. Such properties can be well studied using metallographic methods. As a matter of fact, heat treatment companies usually adopt these ways as they have been around for a long time and are considered conventional. Nowadays, heat treatment industries usually put a test piece with similar properties to steel components within a charge dedicated for nitriding. After heat treatment, the small test piece is extracted and assessed within a laboratory to determine whether it meets intended requirements along that of the rest of the charge in which it was present. To check the compound layer, the test piece is examined through cross-sectioning and is subsequently prepared for optical microscopy. This allows the determination of the thickness of the compound layer; either by measuring the thickness at different locations and averaging the values or through linear measurements if the layer has a straight upper and lower boundary. Accessing such information allows validating whether the layer has the intended thickness. Eventually, one can therefore assess the suitability of the components on the charge for their future use. Also, such information can help assess the efficiency of the process itself and

whether the heat-treating machines along the parameters used are still up to the task. On the other hand, microscopy also helps determining the thickness of the porous zone on the upper part of the compound layer. This porosity is given as a percentage of the thickness of the compound layer. ^{[1][8]} In figure 04 below, a zoom-in on the surface of nitrocarburized steel shows the presence of a darker spotted area at the upper part of the compound layer below the Al foil. That area is what represents porosity in real life.

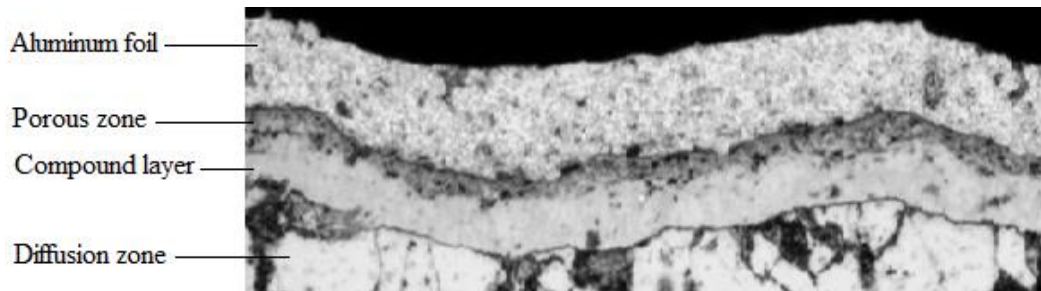


Figure 04 - A closer microscopy image of porosity (grey area) on the upper part of a compound layer

Some standards assess the porosity phenomenon within nitriding processes through the values of the porosity levels within a compound layer. For instance, STD 399-0001 from Volvo Group is specifically dedicated for nitrocarburizing. It entails all the requirements that the finished part must meet notably the compound layer thickness and the porosity level at its uppermost part. It is frequently referred to when porosity levels are to be assessed within a certain steel grade. The STD 399-0001 from Volvo Group is extensively used to classify the different porosity levels (in percentage) and eventually assess the quality of the final component. In general, this classification describes a porosity level of 10 - 20% within the compound layer as low. On the other hand, a porosity level between 20 - 30% is considered moderate, while the one above 50% is considered high and in some applications unacceptable. ^[8]

2.2.2 Nondestructive Evaluation

Overview - Metallographic methods are undeniably efficient when it comes to assessing the quality of both a component and the process which it has undergone. However, these methods come with a set of disadvantages which pushes different industrial contributors and research platforms to seek for other quality assessment methods. The main disadvantages of the conventional metallographic ways can be summarized in the following. First, the amount of time spent during sample preparation can be long, requires constant attention and presence. Second, the amount of steel deployed for quality process costs money and therefore would be preferable to avoid. Finally, the amount of metal scrap and hot mounted polymer is high and environmentally speaking harmful. Indeed, the recycling of this scrap material is always an option yet avoiding it would be a more suitable choice. Therefore, many researchers started lately dedicating their focus to the use of nondestructive techniques instead. Such methods aim at assessing the quality of the elements in question without the need for any destructive measures or sample preparation. These techniques can benefit the industry as they would help reduce lead times and expenses along preserving the environment by reducing the amount of scrap. ^[1]

Based on the definition of the American Society for Nondestructive Testing (ASNT), the examination of an object with technology that does not affect its future usefulness is considered nondestructive evaluation. The applications of NDE methods are in fact numerous due to their nondestructive nature; especially in industry. Some of the most frequent applications would be flaw detection, microstructure characterization or even dimensional measurements. This set of applications allows the possibility to control manufacturing processes along verifying their suitability. To choose the appropriate NDE method and instrument, five basic pieces of information must be cleared out first. This set of information is described as the following.^[9]

- Have knowledge about the material property to be inspected
- Have a clear view of the different characteristics and possibilities offered by respective NDE methods
- Understand both the potential and the limitations of available NDE methods
- Understand the interaction properties between the probe and the test piece
- Consider economic and environmental factors

The aim of these different levels would be to find an appropriate NDE method that would successfully interact with -or get influenced by- the test piece in question without altering its function through a specific physical phenomenon.^[9] For this project, Eddy Current (EC) was chosen as the most appropriate NDE method to use for reasons explained in more detail in the experimental procedures section.

Eddy Current - EC is an NDE method which is used solely on conductive materials. It is very praised within various industry sectors nowadays as a strongly understood method especially in automotive and aircraft domains. As a matter of fact, EC became indispensable in the process control of such manufacturing industries. The two main characteristics that EC is responsive to when subjected to a conductive specimen are electrical conductivity and magnetic permeability. This capability allows the study of various material conditions such as the material type, its defects, porosity and cracks along the thickness of thin layers. Such measurements are made possible since an EC method is generally governed by the response of the material in question to the electromagnetic fields which range from few kHz to some MHz in fact, this wide range of frequencies can come handy in handling the variety of applications that exist out there.^[9]

General Principles - Basically, EC technique manages to inspect conductive materials through electromagnetic induction principle following a set of general steps. These steps can be summarized in the following.^[9]

- Use of an excitation coil
- Interaction with a conductive material
- Use of a second coil to pick up the signal
- Display of the final signal

First, the use of an excitation coil which adopts an Alternating Current (AC) signal creates a magnetic field. Moreover, the excitation coil is connected to a pickup coil linked to a voltmeter. If the current is constant, and the coil locations are fixed, the voltage reading remains stable.

Nevertheless, once a conductive material (ferromagnetic or not) gets closer to the probe, the magnetic field is perturbed, and so does the voltage read. In general, bringing a conductive material into a magnetic field produces a set of currents which travel in circular closed path. These currents are best known as eddy currents which produce a second magnetic field opposing the first one. Through both magnetic permeability and electrical conductivity properties, a great deal of information can be deduced without the need of cracking open the specimen and visually inspect it. Finally, the EC signals can be displayed in different formats. However, all these formats represent the change in resistance of an electric component to AC signal; the impedance. This change is detected by the pickup coil, while the response signals are displayed on the normalized impedance plane with resistance on the x-axis and inductive reactance on the y-axis. ^[9] Below in figure 05, is a simple illustration of the EC working principle with an excitation coil only.

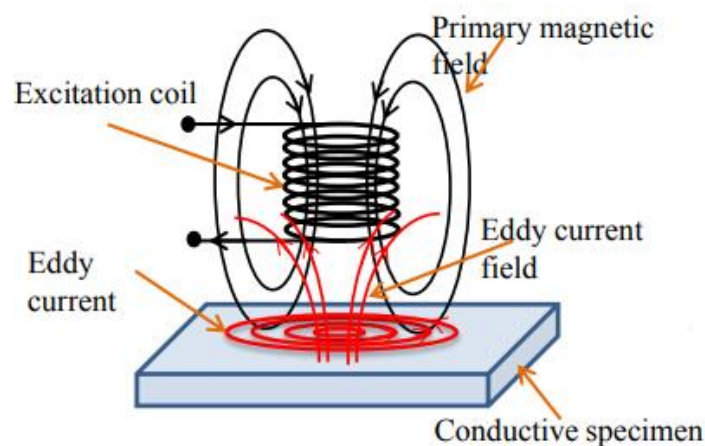


Figure 05 - Eddy current working principle illustration ^[10]

As mentioned previously, one of the most important levels towards determining the most suitable NDE method would be to understand the actual interaction properties between the probe and the test piece. Many companies made the coverage of all EC applications a priority and offer a variety of probe types. As a matter of fact, many companies can custom design the probe to be used based on the application it is supposed to fulfill. However, some standardized probes can be found on the market much easily. To classify a probe, one should consider both the operation mode and the configuration. On one hand, a probe usually belongs -based on its operation mode- to one of the four main categories described below. ^[11]

- Absolute probes => Probes with a single test coil to generate EC and to sense the presence of any fluctuations. Absolute coils can be used for flaw detection, thickness measurements and conductivity measurements.
- Differential probes => Probes with two coils in opposition. This probe detects defects only when one coil is over the defect and the other is over good material. This creates a differential signal, hence the name of the probe.
- Reflection probes => Probes with two coils where the first excites the eddy currents and the other senses the changes in the material.
- Hybrid probes => Probes with mixed characteristics usually designed for a specific application.

On the other hand, a probe configuration usually refers to the way the coil has been packaged and can be classified as the following. ^[11]

- Surface probes => Probes designed to react when in contact with the material surface. They generally consist of a coil with very fine wire inside a protective housing. These probes are good in detecting surface discontinuities oriented perpendicular to the test surface. However, defects parallel to the plane will go unnoticed.
- Pencil probes => Probes with a small surface coil encased in a long thin housing for inspection in difficult-to-reach areas.
- Bolt Hole probes => Probes which are like surface probes but used specifically to inspect bolt holes.
- Inside Diameter probes => Probes used with hollow products to inspect from inside out. The housing of this probe keeps it centered for the coil orientation to remain constant.
- Outside Diameter probes => Probes which are like Inside Diameter probes. Nevertheless, they are used to inspect the material from outside in.

Physical Principles - Basically, an EC working principle is that of an electromagnetic transformer. The relationship between three basic parameters provides the basis of every EC instrument. These parameters are respectively the current, the magnetic field and the voltage. In fact, any type of current flowing in a conductive wire will start a magnetic field, while the presence of a magnetic field in a conductive wire will induce a voltage. Naturally, if the conductive wire in question is forming a closed loop, the current will be flowing across. This crossed relationship between these three parameters is best known as magnetic induction; basic electromagnetic principle which partly explains EC working principle. On the other hand, one can understand the EC technique by simply looking at the impedance plane. As a matter of fact, an EC measurement is nothing but changes in the impedance plane which naturally implies resistance and inductive reactance. Moreover, EC capacity to operate is generally bound to three main parameters namely frequency, magnetic permeability and electrical conductivity. In fact, any variation in these can change the capacity of depth penetration which is defined as the depth at which the EC density has decreased to 1/e or 36,788%. To be more precise, all three parameters have an inverse relationship to the depth of penetration. Therefore, any increase in any of the three parameters will result in a shallower EC penetration to the material surface. Usually, the specimen studied comes with a fixed magnetic permeability and electrical conductivity. Therefore, the only parameter left for the user to adjust for his/her application is the frequency. Some new EC instruments can allow the user to adjust that property for a wider range of applications. This relationship is shown in equation 04 below. ^[9]

$$\delta = \frac{1}{(\pi \cdot f \cdot \mu \cdot \sigma)^{1/2}} \text{ (Equation 04)}$$

δ : Depth of penetration

f : Frequency

μ : Magnetic permeability

σ : Conductivity

Also, one must always keep in mind that alongside electromagnetic factors the geometric ones are of great importance. In fact, this type of factors such as the coil parameters, sample geometry and probe proximity to the sample edges have an impact over the magnetic field. Consequently, even the current flow is impacted and so is the final voltage signal. ^[9]

Capabilities and Limitations - As any other NDE technique, EC has a wide range of capabilities that make it an attractive method to consider. However, this method has also some disadvantages which limit its application fields. A number of these advantages and limitations can be found in table 03 below. ^[9]

Table 03 - Advantages and limitations of eddy current as nondestructive testing technique ^[9]

Advantages	Limitations
=> Usually light and portable instruments => No hazardous impact on operator health => No hazardous impact over the environment => Well understood technology => Relatively low cost => Rapid inspection guaranteed => Used for both ferromagnetic and non-ferromagnetic components	=> Only valid for conductive materials => Only valid for surface and subsurface inspection => Sensitive to geometrical factors => Sensitive to liftoffs => Interpretation of results needs some degree of expertise

Eddy Current Calibration - It is important to mention that almost every EC instrument requires comparing the signal obtained on the impedance plane to a well calibrated reference signal. Consequently, unknown references can be approximated with a great deal of accuracy using a regression analysis model. Saying that, one can ascertain that calibration is a vital component of EC technique as a thickness measurement application and therefore a very close attention to the reference test pieces is required. These calibration samples must meet the expected requirements based on the intended testing purpose. Moreover, these reference test pieces must be prepared with a big deal of attention as the quality of the test depends ultimately on that of the calibration process. ^[9]

Applications - As mentioned previously, EC as an NDE technique can cover several tasks from which defect detection and subsequent material characterization are the most widely spread within industry. Nowadays, this type of EC application is heavily used in aerospace applications. In fact, this type of industry requires a constant checkup of the different components of the aircraft frames for example. Nevertheless, it would be economically unviable to disassemble the airframe or the wings components each time an inspection is required. Therefore, it was necessary to find a way to detect the presence of any flaw without dismantling the whole aircraft. Fortunately, the presence of EC nondestructive technique within this field helped the preservation of quality and safety standards under economically viable circumstances. On the other hand, EC can also come handy in other applications such as material sorting or thickness measurement of thin layers. For instance, the evaluation of the

thickness of a non-conductive layer on top of a conductive specimen becomes possible if a certain EC frequency level was chosen. This allows quick quality assessment of deposited coatings on conductive material in surface treatment industries.^[9] Moreover, there are several applications which seem to be less frequent. For instance, the thickness measurement of a conductive layer on top of a conductive substrate with a difference in conductivity and magnetic permeability levels between both. Such an application is considered till this day very rare within industry as it requires the assembly of an NDE EC instrument with a narrowed down application range to that previously mentioned purpose solely. Generally, this specific EC technique can be beneficial in the heat treatment domain. In fact, the measurement of the compound layer thickness formed on nitrided/nitrocarburized steel seems like a promising field where this EC method can be widely adopted in the future.^[11]

3 Experimental Procedures

In this section, the focus is dedicated to the experimental procedures adopted in the project. The steps followed during the experiments are described in detail along the different instruments which made it possible. Also, the reasons for including such tests in the thesis work are being explained along a clarification of the sample and test matrix constituents. During the first month of the project, a presentation was performed at a VärmeBehandlingen Centrum meeting in order to introduce early findings. For more details about that presentation along the initial steps of this thesis work, please refer to appendix 02.

3.1 Choice of Nondestructive Technique

As expressed in the literature review, this project considers the use of a nondestructive technique for the evaluation of the compound layer thickness. Therefore, a choice had to be made when it comes to the nature of the most suitable nondestructive technique to use. Several factors had to be addressed such as the material to be inspected, the possible NDE methods to be used along the economic and environmental factors. For more details about the theory behind these factors, please refer to section 2.2.2.

Regarding the type of material used in this project, round samples of steel were intended to constitute the sample matrix. Consequently, it was clear that the parameters which could lead to a nondestructive inspection of the different layers are primarily electrical conductivity and magnetic permeability as the material may be ferromagnetic. Furthermore, these test pieces were subjected to nitrocarburizing and therefore the resulting compound layer thickness values were expected to range from 5 μm to a maximum of 32 μm . For more details about the process parameters followed please refer to section 3.3. Moreover, the characteristics, potential and limitations of the NDE method chosen had to be evaluated as well to decide. In fact, a choice had to be made at this stage between the use of either EC equipment or a Barkhausen noise (BN) one. However, due to a limited understanding of BN processes, scarcity of marketed equipment and difficulty of interpretation of results, the EC choice seemed more appealing in this case.^[13] As a matter of fact, it was proven that with a variation in conductivity and/or magnetic permeability, EC nondestructive equipment can determine the thickness of the uppermost layer and output it as a numerical result if assisted with suitable software.^[9] This property made the deployment of the old EC equipment at Bodycote very interesting for the scope of this project. For more details about the equipment which allowed that statement, please refer to section 3.5.1. Moreover, EC method penetration capacity is very low; usually referred to as a limitation.^[9] Nevertheless, in the case of the very small compound layer thickness range this would not cause a problem at all. On the other hand, EC nondestructive technique can be used with all conductive materials which is an advantage compared to BN which can only be used with ferromagnetic specimens.^{[9] [11]} Through a clear understanding of all what was previously mentioned, one can fully assess EC signals resulting from an interaction between the EC probe and the specimen in question. Finally, NDE equipment can be expensive in some cases, yet EC ones are known for a relatively affordable cost and with null implications on the operator health or the environment.^{[9] [11]} Consequently, the perfect choice for the project was indisputably the EC technique. However, BN is still a valid choice for this type of projects.

3.2 Sample Matrix

In this project, a sample matrix with all its constituents had to be determined at the very start of the thesis. In that way, one could understand the steps to follow along the studies to be conducted. All the samples were grinded from one side at the beginning of the project as to allow better microscopy study in the future. This sample matrix was composed of 160 steel samples divided into two different steel grades and dedicated to two different quenching mediums. Consequently, the steel samples have been divided in a total of four main groups where each forty samples belonged to a certain steel grade and a specific quenching medium. A summary of the general repartition of the sample matrix can be expressed as shown in table 04 below. Each group of these shown in table 04 contains four different subgroups with different compound layer thicknesses. The distinction in the expected depth ranges was basically a result of differences in process parameters. For more details about these process parameters, please refer to section 3.3 and appendix 03. Finally, each of these sixteen subgroups contained a total of ten samples; a demi-batch. In fact, a batch constitutes of twenty samples with half of them from steel grade 01 and the other half from steel grade 02. In fact, these samples were numbered from 01 to 10 to assess the upcoming measurements easily.

Table 04 - General repartition of the sample matrix

	Steel grade 01	Steel grade 02
Quenching medium 01	40 samples	40 samples
Quenching medium 02	40 samples	40 samples

Regarding the different constituents of the sample matrix, one must consider the following properties to get a clear idea about the matrix. First, this project entails the use of two different steel grades which are respectively SS 2172 and SS 2541. These two steel grades have been chosen as they are widely used by Bodycote and contain different chemical properties. This allowed the measurements to be diversified and eventually a comparison between these two steel grades was believed as interesting to investigate. The first steel grade is known under the Swedish standard as SS 2172. It is a structural steel with different denominations from a country to another, yet it is mostly known as S355JR. It is known as low carbon steel and is basically used for structural constructions.^[14] On the other hand, the SS 2541 is quenched and tempered steel with different denominations around the globe as well. In fact, it is mostly designated as 34CrNiMo6. It is known to be a medium carbon steel and is used heavily with surface and case hardening applications.^[15] For the purpose of this project, eighty samples from each of these two steel grades were deployed as shown in table 04 above. As a matter of fact, the use of a set of samples from different steel grades was intentional since the beginning. The idea was to prove experimentally the actual relationship between the number of alloying elements and the final compound layer thickness formation. The actual number of alloying elements in each of the respective steel grades (SS 2172 and SS 2541) can be found in table 05

below. ^{[14][15]} On the other hand, the use of different quenching mediums aimed at determining the difference that would result in the compound layer thicknesses and porosity levels from the respective use of oil and gas. More importantly, the accuracy and precision resulting from the EC equipment readings vis-à-vis the use of these two different quenching mediums was to be evaluated and compared to each other.

Table 05 - Percentages of alloying elements in both steel grades ^{[14][15]}

	Steel Grade	
Alloying Elements	SS 2172	SS 2541
Cr	0,06%	1,42%
Mo	0,019%	0,17%
V	-	0,01%
Mn	1,31%	0,75%
Ni	0,1%	1,37%

The process designations are composed of seven numbers; the couple first ones represent the year, the second two are dedicated to the month of process, while the last three represent the process number at Bodycote during that month. Such a designation makes it easier to extract information about that batch whenever required. For general information about the denomination of batches to which the resulting specimens belonged, please refer to table 06 below where the batch numbers are divided between oil and gas quenched processes. Also, they have been put in an ascending order regarding their respective compound layer thicknesses.

Table 06 - Process designations

Oil-quenched Batch Numbers	Gas-quenched Batch Numbers
1902 - 964	1901 - 184
1902 - 270	1902 - 092
1901 - 716	1901 - 223
1902 - 521	1902 - 289

3.3 Process Steps and Parameters

After some rigorous planning, the sample matrix expressed above was gathered at Bodycote. The 160 untreated samples were provided for the sake of the project; with 80 samples from each steel grade of interest.

The nitrocarburizing process steps and parameters adopted can be summarized in the following. First, the samples have been charged into different sets based on the parameters that were intended for each batch. It is important to point out as well that some of these batches were loaded with other components dedicated for Bodycote's customers. This was the case when dealing with process parameters used by the heat treatment service provider company. Nevertheless, it was necessary to go beyond that to achieve a wider range of compound layer thicknesses to serve the purpose of the thesis project. A set of samples were simply put in charges with dummy components rather than actual ones planned to be sent to customers. This fact can affect the results as the continuous use of dummies can cause the components in question to form the compound layer at different rates than when put amongst actual untreated specimens. Therefore, a close attention had to be dedicated for this distinctive matter. Each component within the batch would go similar heating, quenching and tempering.

Once the components had been charged, the respective batches had to undergo pre-washing to clean them from any grease, dust particles or other substances. This is a critical step which helps avoid any influence over the product properties or surface appearance. This step was done at Bodycote using a certain type of alcohol for cleaning purposes. Furthermore, a pre-heating step is necessary to reach a homogeneous temperature all over the charge. In the case of this project, the heating temperature aimed for was 400°C in a primary furnace with the gaseous environment composition of 5% CO₂, 60% N₂ and 35% NH₃. Then, the charge head for the hardening phase where the different batches were subjected to 580°C in the main furnace. In fact, the difference in compound layer thicknesses was basically a result of the use of a variety of holding times dedicated to differentiating between the processes results. Later on, a decisive step was conducted which is none else than quenching. For this project, two types of quenching were applied and required different quenching times and temperatures. The first quenching medium used was oil with a quenching time of 20 minutes and a temperature of 60°C for the entirety of the eighty samples dedicated for this quenching type. On the other hand, the other eighty samples had to undergo quenching by gas with a quenching time of 105 minutes for three batches but with 150 minutes for the remaining one. The use of such different quenching times was intended to spot the impact that this parameter has over the final compound layer thickness, while the quenching temperature was held at 100°C for all these samples. Finally, these samples were sent for post-washing to remove any substances remaining on the samples surface using alcohol mainly; as to avoid any negative impact over the surface of the final component. For a more detailed description of the process properties, please refer to Appendix 03.

Finally, it is important to mention that the resulting nitrocarburized samples had different degrees of cleanliness when quenched using oil or gas. Such a parameter will be discussed further in the following sections as an essential aspect to investigate.

3.4 Nondestructive Test Matrix

After a clear determination of the sample matrix to use along subjecting all of it to the respective nitrocarburizing parameters, it was big time to start planning for the type of tests to be conducted. Basically, these tests were nondestructive using EC as an NDE technique. The instrument to use for that was an already-deployed EC device used at Bodycote. However, one of the goals of the project was to find a replacement for this EC instrument in the market. Consequently, a similar measurement methodology was planned to be applied on both instruments to get a final comparative assessment of both machines. Eventually, these measurements were to be compared with the actual compound layer thicknesses of the different nitrocarburized batches. The nondestructive measurement methodology constitutes of four main parts to cover a respective set of information. These test matrix constituents along the information it tends to cover can be found in table 07 below.

Table 07 - Nondestructive test matrix

Measurement Designation	Measurement Description	Measurement Aims
Full test	Extended EC measurements over the entire set of samples except for the first ones from each demi-batch. Each respective sample is measured five times at different spots on its grinded surface. These measurements were made using two types of calibration.	- Get an overall idea of the device readings - Compare the EC instrument results to the actual metallographic ones - Compare the EC instrument results following the use of two different calibration types
First samples	Measurements of the EC instrument over the first sample of each demi-batch. Each respective sample is measured ten times on the same spot. Two set of measurements were repeatedly applied on the same spot. The first set of measurements was done continuously, while the second one required retrieving the sample after each time and placing it again. Also, the measurements were made using two types of calibration.	- Compare all these EC measurements with the metallographic study - Get an idea over the instrument-only (repetition) readings and the instrument-operator (replica) ones. - Compare the EC instrument results following the use of the different calibration types

Stability over time	Measurements of the first samples of two chosen batches from both quenching mediums and steel grades. Each respective sample is measured five times on the same spot. These measurements were conducted over the period of a month every Monday, Wednesday and Friday.	- Determine the instrument measurements accuracy and drift over a period - The aim of such measurements is to expect how often recalibration is needed
Measurement System Analysis	Measurements of the first samples of every demi-batch. These measurements are conducted ten times over each of the studied samples and are conducted by three operators respectively. Each operator repeats the entire set of measurements twice.	- Get an idea over the dominating source of variation from the measurements conducted - Get percentages of the repeatability, reproducibility and part-to-part contribution in variation - Determine the EC instrument capacity in outputting accurate values and define the appropriate working conditions for that

3.5 Nondestructive Equipment

The nondestructive measurement plan was then applied using the already-deployed nondestructive EC instrument. In this section, this EC instrument will be introduced, and the experimental steps conducted using it will be described as well. Moreover, the set of steps followed to determine all the characteristics of this nondestructive equipment will be determined. For further information about this first nondestructive equipment, please refer to the self-translated manual in Appendix 04. On the other hand, the quest to find another replacement will be mentioned along the different equipment directly and indirectly investigated.

3.5.1 First Nondestructive Equipment

Overview - The primary instrument used for this thesis project was an already-deployed EC device at Bodycote Heat Treatment in Gothenburg. The instrument is best known as NORTEST 2060 and was built by Dr. Albert Ott in Wiesbaden, Germany. It is specifically dedicated for the measurement of compound layer thicknesses of nitrided layers. The ultimate advantage of this machine is that it comes with software assistance and basically manages to output the result

(compound layer thickness in μm) as an actual numerical value. For a descriptive image of the output on a computer's screen, please refer to figure 06 below.



Figure 06 - Output screen from NORTEST 2060

To cover all the pieces of information regarding NORTEST 2060, one had to start from the operation manual of the device. Unfortunately, and as it could be guessed from figure 06, the manual was in German and therefore had to be translated into English. It took a while before getting to the final translated version which could be found in Appendix 04. This step was important to go through as it allowed the understanding of a great deal of the working and measuring principles of the instrument.

NORTEST Principles - Regarding the working and measuring principles, NORTEST 2060 is an eddy current equipment used for the determination of the compound layer thickness on nitrided steel. One of the most important steps towards that would be a well-performed calibration. Such a calibration works in the following manner. First, the user must calibrate the equipment through a specific calibration number using samples from the same steel grade as the intended sample to be measured. It is worth mentioning that the instrument offers the possibility of multiple calibrations stored as calibration numbers. The user needs only to change the calibration number to work with any of the already calibrated sets. Calibrating NORTEST 2060 is considered easy as the user only needs to place the probe over the surface of the test piece and press the enter button on the keyboard. This would then generate a time-varying eddy current in the specimen which will provide eventually a representative signal of the measured location. These signals are then treated using already built-in software assistance. These values are then correlated to actual known compound layer thickness values inputted by the user. Based on the principle of linear regression, a calibration function is constructed from the x-y values which respectively represent resistance and inductive reactance values from the eddy current device. After finalizing all these steps, one can measure the compound layer thicknesses of components which belong to the same steel grade with which the calibration was conducted.

Furthermore, the user can store up to eighty different calibrations within the instrument and can freely use any of them. However, one must pay close attention to the fact that these calibrations experience some sort of drift over time and may result in erroneous outputted numerical values after a while of use. Therefore, a constant verification -using the samples with which the calibration was set in the first place- must be conducted every now and then. By the end, the user can simply change to the calibration number of interest and conduct as much nondestructive measurements as wanted. In this project, two types of calibrations were used. The first calibration required the separation of the set of samples based on both quenching medium and steel grade, while the second calibration relied solely in a separation based on steel grades. Although the samples used for calibration were presented by Bodycote, it seemed necessary to add a couple of samples from own specimens. The reason behind that was mainly the inappropriate surface condition of some of the calibrating Bodycote samples due to ageing.

Regarding the actual measurement principle, NORTEST 2060 serves as a quality assurance for the nitrided layers in general. Through the eddy current method, one can measure the thickness of the compound layer in a much easier manner than the conventional metallographic ways. In this project, a spring-mounted test setup was used during the entire set of measurements. Regarding the working procedure of NORTEST 2060, the test piece is ought to be put over a flat stand at a distance close enough for the probe to reach the sample's surface. Then, the probe is brought down gently using a lever until it touches the component. Consequently, the enter button is pressed on the keyboard to initiate the measurement. For more control over the test setup, a damper was added to reduce the measurements variation due to the instrument. Basically, the probe makes the measurement possible through the change of the impedance. This change in impedance is interpreted by a voltmeter which in its turn allows the signal to be digitally converted. Hence, the user can get a numerical value of the compound layer thickness shown on the screen in a couple of seconds. For a clear image of the test setup alongside that of the probe tip, please refer to figure 07. For more information about the components of NORTEST 2060, its connections, functions and options, please refer to the translated operation manual in Appendix 04, while images of the instrument can be found in Appendix 05.



Figure 07 - NORTEST 2060 test setup at Bodycote Heat Treatment in Gothenburg

Probe - Naturally, the most critical component of the NORTEST 2060 setup is the probe. Therefore, the measurements conducted had to deal very carefully with it as sliding it over the test piece surface will be disastrous for future use. Moreover, it was made sure that the probe pole gets cleaned using a soft alcohol-wetted cloth between each now and then.

The NORTEST 2060 operation manual offered quite extensive set of information about the instrument working principle and usage. However, the manual did not give enough details about the probe used. Therefore, it was necessary to get an in-depth idea about the inner components of this part. Luckily, the project work got access to an already-made tomography study of a supposedly identical probe. It was shown that the probe has one coil only with some sort of iron core. As it is seen in figure 08 below, the coil is made parallel to the surface to get a measurement at the end of the probe (top of the picture). Therefore, it is believed that the project is probably dealing with some sort of absolute probe. For more details about the characteristics of such a probe, please refer to section 2.2.2. For other images from the tomography study with a focus over the tip of the probe, please refer to Appendix 06.

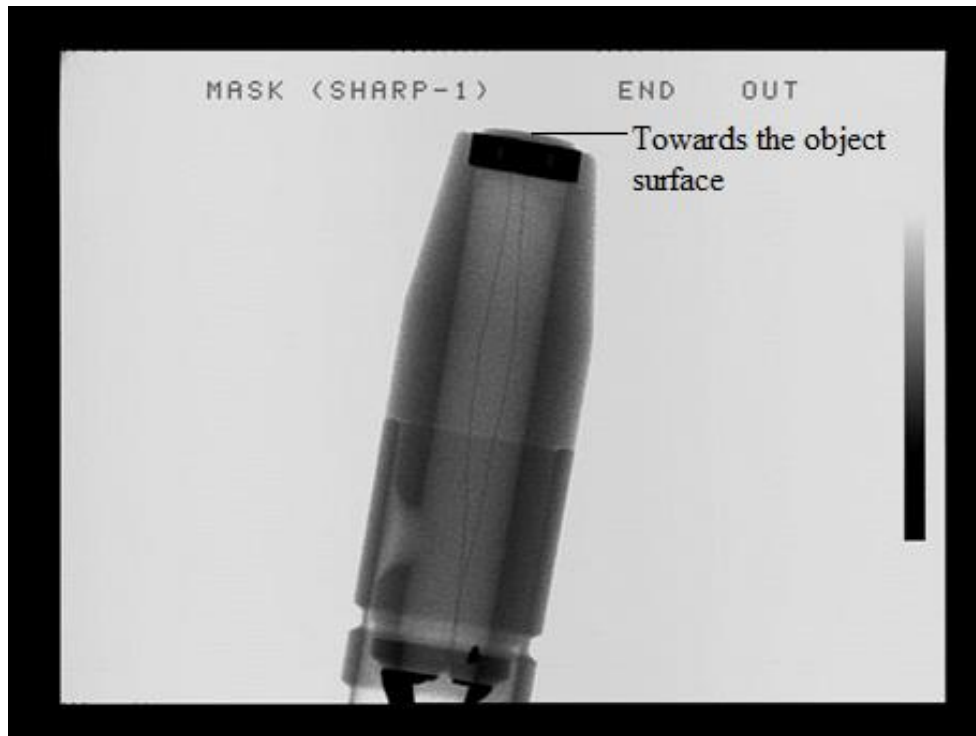


Figure 08 - Tomography picture of a similar probe (Courtesy: Jonas Holmberg, RISE IVF)

Frequency - During the measurement weeks, it was shown that a final important piece of information was missing from the operation manual and the NORTEST website; the frequency of the instrument. It was very important to determine such a parameter as it would help in the quest of finding a suitable replacement for NORTEST 2060 in the future. A clear idea about the instrument frequency could assess whether the new equipment proposed has the appropriate capacity for an application with such a need for small depth of penetration (from 5 μm to 32 μm). For that reason, an oscilloscope was deployed to extract the frequency value alongside its type. The first guess was a one-variable wave frequency with several MHz in value. For the image of the USB oscilloscope used, please refer to figure 09 below. The oscilloscope in question was connected to a portable computer using PicoScope06 software. For any frequency measurements, the oscilloscope probe is linked to its hook and forms a circle through which the NORTEST probe goes to assess its frequency value and type. However, there were some doubts about the oscilloscope device to catch satisfying results. Therefore, a self-made number of cables turns over a plastic piece was linked to the probe from one side and the hook of the oscilloscope from the other. The intention was to allow more visibility of the frequency signals for better interpretation under the AC mode and a voltage of $\pm 50\text{ mV}$.



Figure 09 - Oscilloscope from Pico Technology (Courtesy: Anders Rosell, GKN Aerospace)

3.5.2 Quest for a New Eddy Current Equipment

Overview - One of the main goals of this thesis project was to find a new EC equipment which would successfully replace NORTEST 2060. Therefore, several EC instruments were either directly or indirectly investigated to fill up the purpose of the NORTEST 2060. At the beginning, this was thought to be a simple matter of search. However, as the project was going on it has been found to be a tougher task than expected; as an EC instrument with properties like NORTEST 2060 is quite scarce.

The main properties that were being looked for in the future EC instrument are the following. First, the presumably new EC instrument had to be dedicated for thickness measurement. As a matter of fact, most of the EC instruments in the market are defectoscopy ones due to its use across several industries such as aerospace and automotive. Therefore, one must be careful in picking the right EC instrument application to replace NORTEST 2060 tasks. Second, one had to investigate the frequency provided by the equipment as well. For instance, a small frequency would result in a higher depth of penetration than needed which would result in getting little information from the compound layer thickness or none. Therefore, it was a necessity to focus on finding a future NDE instrument to replace NORTEST 2060 with a suitable frequency value. Furthermore, the EC equipment setup was to be taken into consideration as it would allow assessing its usability at Bodycote workshop or laboratory. Finally, it was also necessary to look at the price of the new EC instrument candidate and provide quotations for Bodycote. Taking into consideration all these aspects, it was possible to narrow the project's search field and make it easier to find a suitable replacement.

Instruments - Through the quest towards suitable EC equipment to replace NORTEST 2060 at Bodycote, a set of equipment were respectively investigated, described and even tested either directly or indirectly. The search was done by contacting several NDE companies/ institutions, heat treatment service providers and different contributors within the field. The descriptions of the strongest EC equipment candidates along their respective companies can be found in table 08 below.

Table 08 - Replacement candidates for NORTEST 2060

Company Name	Instrument Name	Description
Elcometer Ltd.	Elcometer 456	Coating thickness gauge with a measurement uncertainty of $\pm 1\%$ ^[16]
Fischer Inc.	Dualscope - MPOR	Coating thickness measurement pocket instrument used virtually with all metals ^[17]
Foerster GmbH	MAGNATEST D 3.623	Instrument used for the magnetic induction testing of metallic objects ^[18]
Fraunhofer IZFP	3MA Testing Unit	Multi-parametric instrument used for analysis of residual stress and microstructural one near the surface area ^[19]
Rohmann GmbH & CNS GmbH	QUTEC.DELTA. S	Instrument specifically dedicated for the determination of the compound layer thickness of nitrocarburized components with an uncertainty of approximately $\pm 1 \mu\text{m}$ ^[20] The instrument itself is from Rohmann GmbH but the software assistance allowing the monitor to display the final numerical result is from CNS GmbH

*Fraunhofer IZFP - Fraunhofer Institute for Nondestructive Testing

*3MA - **M**icro-magnetic **M**ulti-parameter **M**icrostructure and stress **A**alyzer

*CNS - **C**ustomized **N**ondestructive Testing **S**olutions

MAGNATEST Investigation - The suitability of the MAGNATEST D 3.623 EC device was personally investigated at KmK Instrument at Västerås. Samples 02 from each demi-batch were deployed, while the calibration was applied using the thinnest compound layer thickness and the thicker one within the same batch. The calibration used is the one where a clear separation of both steel grades and quenching mediums is deployed. Consequently, four different graphs were obtained with four signals within each one of them. Each signal represents a sample within that group. For more details, please refer to section 4.2.2.

3.6 Metallographic Procedures

3.6.1 Optical Microscopy

Overview - To compare the EC measured values to actual compound layer thicknesses, it was necessary to use metallographic procedures as well as nondestructive ones. In that way, one could assess the measured values obtained from NORTEST 2060 and compare them to actual ones. This had allowed an actual study of the NDE equipment used when it comes to its measurement uncertainty, repeatability and stability over time. For more details about the type of measurements conducted on the EC equipment, please refer to section 3.4.

The optical microscopy procedures conducted during this thesis work can be divided into two phases. As a matter of fact, this metallographic study was applied under two different occasions. At the beginning, an introductory survey was planned as it allowed getting an overview about the actual depth ranges vis-à-vis the results from NORTEST 2060. For more details about these depth ranges, please refer to Appendix 03. On the other hand, another phase was conducted at the end of the NDE measurements to consolidate the results from the introductory survey. For more details regarding all the steps followed and the phases adopted, please refer to the paragraphs below.

Sample Preparation - Naturally, conducting metallographic studies required preparing the samples in question as to make them ready for optical microscopy. The steps followed are quite familiar to the reader as they were mentioned vaguely in section 2.2.1. However, in this part a clear description of the actual steps followed during this thesis work will be clarified.

The study was divided into two phases; an introductory survey and a consolidating one. At the beginning, sample 01 from each demi-batch was cross-section cut, covered in Al foil and hot mounted using Bakelite (a spoon of soft Bakelite and another of hard one). Then, the sample prepared specimens were grinded and polished automatically (using a Struers Tegrapol 31 grinder polisher machine) to remove any abrasives or dust stuck on them. For an illustration, please refer to figure 10 below. By the end, the hot mounted samples were cleaned thoroughly with alcohol then etched using Nital as to get more reliable images from optical microscopy. This initial phase allowed a clear determination of the actual compound layer thicknesses and respective porosity levels formed on the upper part of them. It is also worth mentioning that each hot mounted specimen contained two cross sections belonging to the two different steel grades used during this thesis work. Regarding the consolidating phase, it was added at the end of the measurements phase as to prove the homogeneity of the results across each demi-batch. In fact, samples 09 and 10 from each demi-batch were sample prepared for optical

measurements as well. The goal was to get a better overview regarding the actual compound layer thicknesses obtained after each nitrocarburizing. However, and due to a constraint in time, this phase did not include a porosity level study.



Figure 10 - TegraPol 31 grinder polisher station from Struers

Compound Layer Thickness - It was self-explanatory that a clear idea of the real compound layer thickness was necessary to compare with the measured values outputted from the NORTEST 2060 EC device. It was believed that three samples from each demi-batch (two samples with different steel grades from the same batch) would be enough to formally assess the homogeneity of the nitrocarburizing process. Moreover, it allowed a comparative study that would later be used to determine the reliability of the NORTEST 2060 device. When brought to the optical microscope, the polished and etched hot mounted samples were investigated and especially at the very surface of the cross section where the compound layer lies. A zoom-in by fifty times allowed getting the actual measurements of the compound layer thickness. Furthermore, these measurements were taken at five different spots for a well averaged value that can eventually be compared to the NORTEST 2060 one.

Porosity - As for the porosity levels, it was important to focus on the presence of such a phenomenon both theoretically and experimentally. The reason for such an interest is the necessity to understand the behavior that eddy currents have in the presence of a porosity field. It was interesting to make a conclusion over the impact of porosity over the impedance change and whether one had to take it into consideration or not. This specific type of study was conducted through an approximate determination of the darker dotted layer which exists on the upper part of the compound layer. In that way, one could assess the porosity level by simply

dividing the porosity thickness by the average compound layer thickness on that specific sample as shown in equation 05 below.

$$\textbf{Porosity Level} = \frac{\textbf{Porosity Thickness}}{\textbf{Compound Layer Thickness}} \text{ (Equation 05)}$$

3.6.2 Scanning Electron Microscopy

Overview - The use of the SEM was deemed as complementary for this thesis project. However, it was still interesting to use a higher magnification over the compound layer upper part to get a better view of the porosity formation. Such a study could help identify the channel formations discussed in the literature review section 2.1.2.

Sample Preparation - Regarding the SEM sample preparation, it is quite like the optical microscopy one. In fact, an already prepared hot mounted specimen was simply deployed. However, it is known that SEM uses an electron beam which would have caused the polymer material within the hot mounted specimen to illuminate. That would have led to an impossible investigation of the compound layer. Therefore, it was decided to cover the polymeric black area all around the sample surface with copper instead.

Porosity - The use of SEM was intended to approve what was mentioned in the literature review about the formation of channels on the compound layer surface. In this project, the investigation solely included porosity over samples from 1901 - 223 batch only. In fact, magnifications from 1000 up to 30000 times were used for that specific purpose. However, only the SEM images with a good resolution are the ones being introduced within the results section.

4 Evaluation of Results

In this part, all the different sets of results are presented in the order in which they have been conducted. It includes nondestructive results as well as microscopy ones; both numerical values and images. Bear in mind that this section presents the results with a simple preliminary evaluation only. In fact, a proper discussion will not follow until the next part.

4.1 Introductory Survey

4.1.1 Compound Layer Thickness

The measurements conducted using optical microscopy at the beginning of the thesis project gave a clear perspective over the actual compound layer thickness of each batch. These preliminary metallographic results are shown in detail in tables 09, 10, 11 and 12 below. Each table of these represents the first samples of each quenching medium and steel grade.

Table 09 - Actual compound layer thickness of first oil-quenched SS 2172 samples

Batch Number	Microscopy Results (μm)					Average Result
1902 - 964	15,96	15,53	15,32	15,11	13,83	15,15
1902 - 270	16,49	14,26	16,40	16,38	16,17	15,94
1901 - 716	16,39	18,09	16,28	16,60	15,11	16,49
1902 - 521	24,80	26,61	25	25,65	25,75	25,56

Table 10 - Actual compound layer thickness of first oil-quenched SS 2541 samples

Batch Number	Microscopy Results (μm)					Average Result
1902 - 964	13,40	13,41	13,19	13,30	13,41	13,34
1902 - 270	12,87	13,83	12,98	14,15	13,94	13,55
1901 - 716	14,37	12,24	12,98	14,90	14,79	13,86
1902 - 521	23,62	23,62	22,02	22,55	20,43	22,45

Table 11 - Actual compound layer thickness of first gas-quenched SS 2172 samples

Batch Number	Microscopy Results (μm)					Average Result
1901 - 184	8,51	9,58	10,43	9,89	10,01	9,68
1902 - 092	12,24	10,32	11,28	12,77	10,64	11,45
1901 - 223	19,57	20,96	21,49	20,64	19,89	20,51
1902 - 289	20,96	22,24	23,51	21,81	22,23	22,15

Table 12 - Actual compound layer thickness of first gas-quenched SS 2541 samples

Batch Number	Microscopy Results (μm)					Average Result
1901 - 184	10,85	9,58	7,98	8,09	10,43	9,39
1902 - 092	10,53	10,53	11,81	11,17	9,79	10,77
1901 - 223	15,85	15,96	17,34	17,77	17,88	16,96
1902 - 289	20,86	20,53	20,53	21,17	20,64	20,75

These results served as initial study to get an overview over the real compound layer thicknesses of each batch. From tables 09, 10, 11 and 12 a clear distinction between the oil and gas quenched strikes at first sight. It was indeed proven that the use of non-identical quenching mediums led to different compound layer thicknesses. In fact, the use of oil quenching allowed the compound layer thickness of the different samples to range from 13 μm to approximately 25 μm . However, the use of gas as a quenching medium permitted to obtain a compound layer thickness range from 9 μm to a maximum of 22 μm . Therefore, one can easily evaluate from the results in tables 09 to 12 that the quenching medium is an important factor in the composition and thickness of the final compound layer. Furthermore, a difference in final compound layer thicknesses between the SS 2172 samples and the SS 2541 was a striking fact as well. The results showed that SS 2172 samples were proved to get a thicker compound layer thickness than SS 2541 ones. This fact stands even if samples under the same batch number were put on the same charge which means they had undergone similar process parameters. Consequently, it is fair to declare that the different holding times used proved to result in different compound layer thicknesses.

For process properties, please refer to Appendix 03. As a matter of fact, a pattern could be proved since the higher the holding time the thicker the compound layer. These results can be expressed in the form of graphs and are valid for both SS 2172 and SS 2541 samples as shown in figure 11 below.

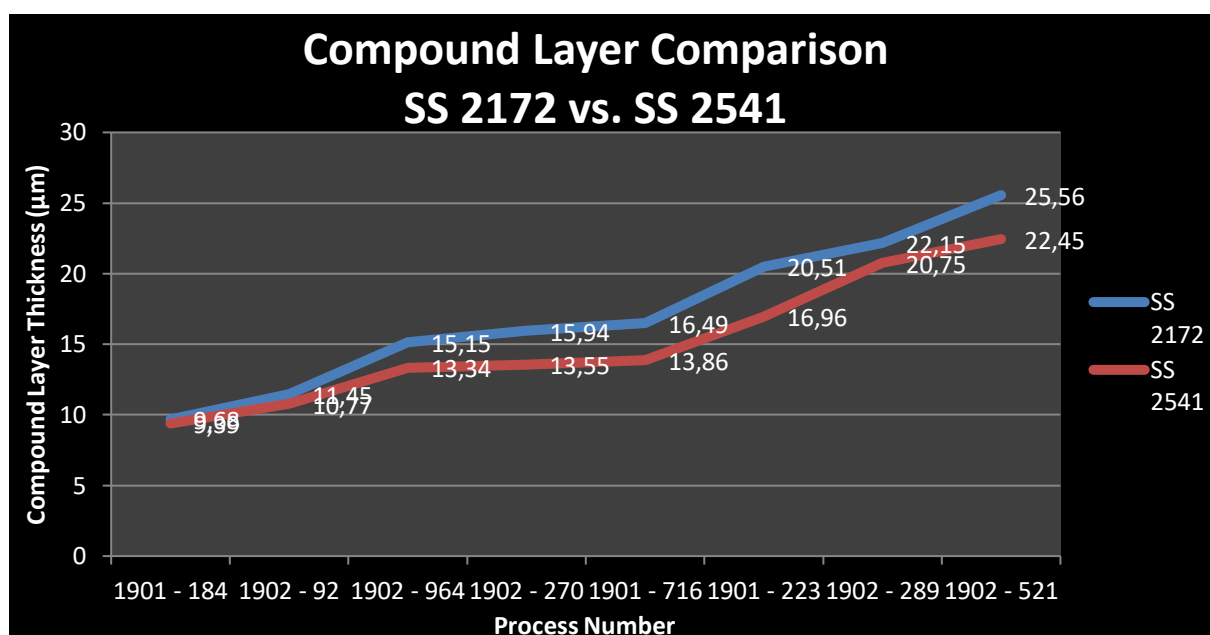


Figure 11 - Comparison between compound layer thickness of SS 2172 and SS 2541 samples

As expressed in the graphs and paragraph above, three main evaluations could be further discussed. First, the impact of the quenching medium over the compound layer thickness seems interesting to investigate. Second, an explanation behind the difference between compound layer thicknesses of SS 2172 samples and those of SS 2541 ones seems necessary as well. Finally, the impact of holding time is also important to look at.

4.1.2 Porosity Level

A study concerning porosity was deemed essential since the beginning of the thesis work. Therefore, it was included in the introductory survey as to evaluate the behavior it follows. The porosity results obtained from this specific metallographic study can be summarized in table 13 below. The porosity measurements were solely applied on the first samples of each batch and compared to the value of the average microscopy measurements.

Table 13 - Porosity levels of the first samples of each batch

Quenching Medium	Process Number	Steel Grade	Porosity Thickness (µm)	Microscopy Measurement (µm)	Porosity Level
OIL	1902 - 964	SS 2172	4,47	15,15	29,50%
		SS 2541	3,19	13,34	23,91%

OIL	1902 - 270	SS 2172	4,89	15,94	30,68%
		SS 2541	3,94	13,55	29,07%
	1901 - 716	SS 2172	5,43	16,49	32,92%
		SS 2541	5	13,86	36,09%
	1902 - 521	SS 2172	11,06	25,56	43,27%
		SS 2541	8,4	22,45	37,42%
GAS	1901 - 184	SS 2172	2,66	9,68	27,47%
		SS 2541	1,91	9,39	20,35%
	1902 - 092	SS 2172	3,51	11,45	30,66%
		SS 2541	2,77	10,77	25,73%
	1901 - 223	SS 2172	7,77	20,51	37,88%
		SS 2541	6,38	16,96	37,62%
	1902 - 289	SS 2172	7,13	22,15	32,19%
		SS 2541	5,74	20,75	27,67%

From the results on table 13, the porosity values generally increase with an increase in the compound layer thickness (or the holding time). Therefore, porosity thicknesses seem to be bigger on SS 2172 samples than on SS 2541 ones. Such a growth in porosity appears to be to some degree parabolic except for the samples from batch 1901 - 223. An evaluation of these porosity values is important as it would allow a comparative study along a better understanding of the nondestructive results from NORTEST 2060. For a clear representation of the porosity

behavior within the compound layer of the different samples, please refer to figure 12 below. Also, for the entire set of introductory microscopy images from which these values (both compound layer and porosity) were extracted, please refer to appendix 07.

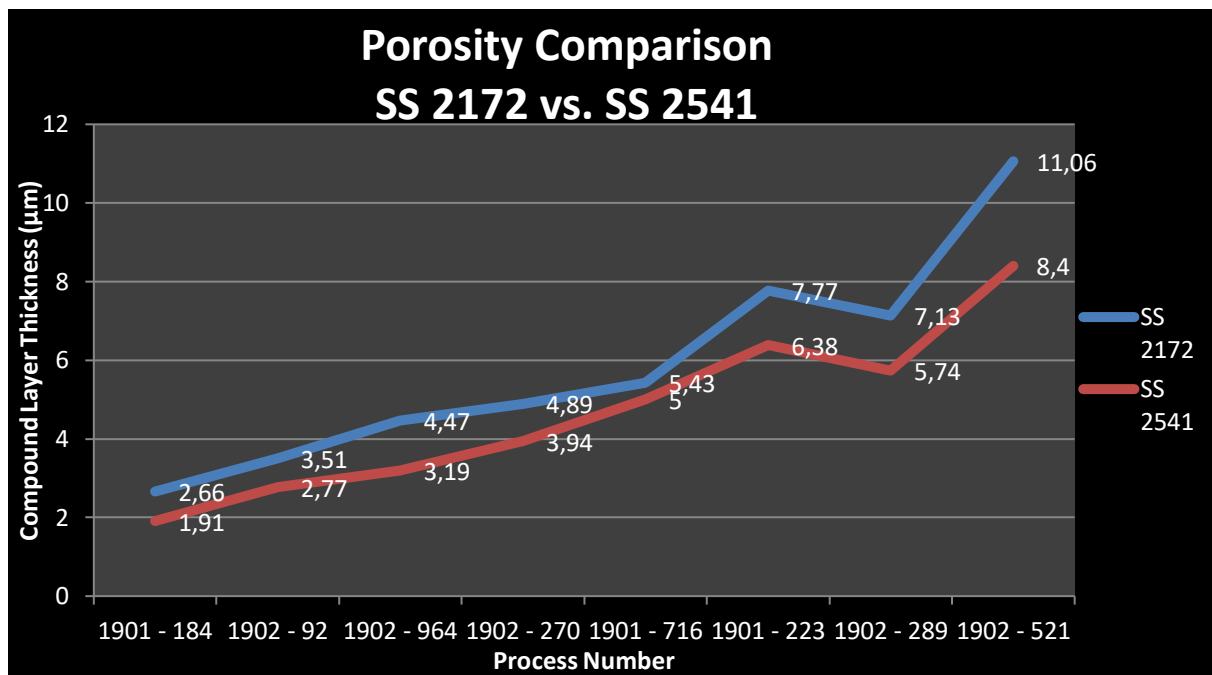


Figure 12 - Comparison between porosity thickness of SS 2172 and SS 2541 samples

Based on the STD 399-0001 from Volvo Group, all these porosity levels seem to have acceptable percentages as they all have values below 50%. Therefore, the outcomes of all nitrocarburizing processes applied during this project are deemed as acceptable.

4.2 Nondestructive Results

After the introductory survey, it was big time to start the core measurements of this project. These measurements were supposedly divided into two parts. The first part would be conducted by NORTEST 2060, while the second one through a similar EC device of choice from the market. The first step was conducted successfully using the EC nondestructive testing device at Bodycote. However, the second phase of the nondestructive study happened to be more demanding than expected. As a matter of fact, the market seems lacking EC devices like NORTEST 2060 in application. Therefore, a quest was started to get as close as possible to a device which could be improved later to reach the level at which NORTEST 2060 stands, or so was the idea.

4.2.1 First Nondestructive Equipment

Frequency - The final missing piece of information regarding NORTEST 2060 is -as expressed previously- the frequency exerted by the probe. Therefore, the use of an USB oscilloscope was decided, and the results obtained from that can be found in figure 13 below.

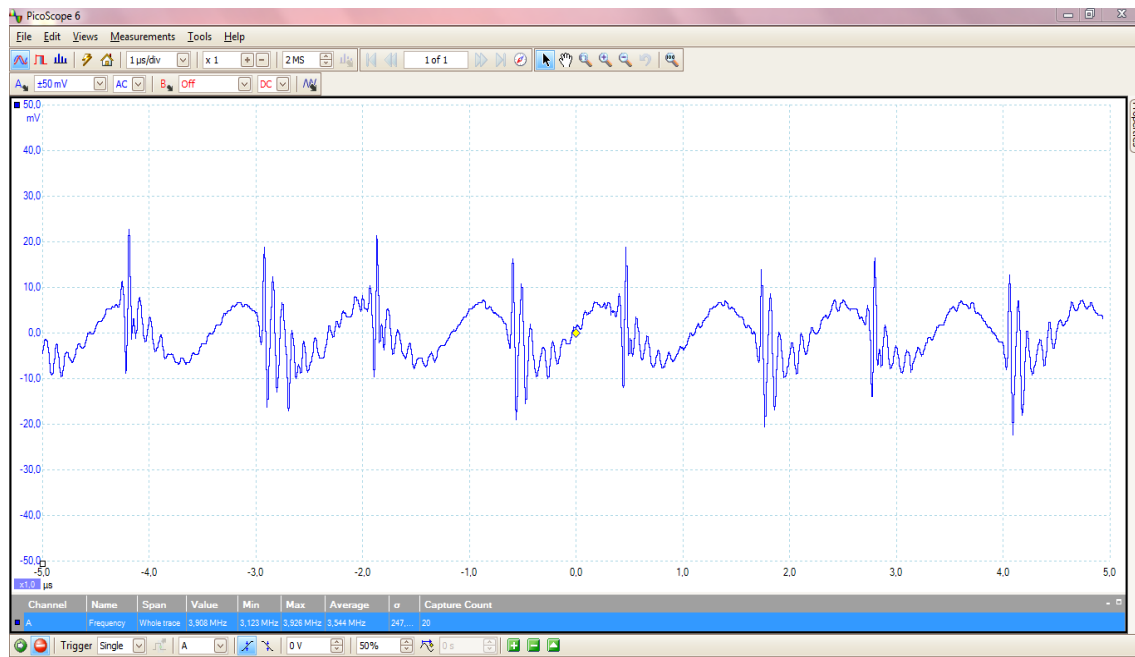


Figure 13 - Frequency wave of the NORTEST 2060 probe tip

From figure 13, the Pico Scope 6 software shows that the frequency used by NORTEST 2060 is a one variable frequency of 1 MHz on one side. This shape and value of the frequency was expected at the beginning of the thesis project. However, another superposing signal is noticed with a higher frequency and smaller bandwidth than the first one with a decaying sinusoidal wave (damped). Such a signal seems to show up on a regular basis. However, a further discussion imposes itself as the source of such a signal can be debatable.

Measured Values - Afterwards, the measurements following the plan represented on table 07 were conducted respectively to obtain the required results. Only the average values of the measurements will be included in this section due to the vast number of nondestructive values outputted. The nondestructive results obtained from the various compound layer measurements conducted using NORTEST 2060 can be summarized as the following.

First, a **full test** was conducted over the remaining nine samples after the introductory survey for each demi-batch. As expressed in section 3.4, the results of such test are divided into two categories based on the type of calibration. The first type of calibration relies on the separation of both quenching medium and steel grade, while the second type is categorized based on the steel grade only. The average measurements of this full test representing both types of calibration can be found in table 14 below as NORTEST Measurement #01 and #02.

Table 14 - Full test measurements results

Quenching Medium	Process Number	Steel Grade	Microscopy Measurement (µm)	NORTEST Measurement #01 (µm)	NORTEST Measurement #02 (µm)
OIL	1902 - 964	SS 2172	15,15	14,67	16,67

OIL	1902 - 964	SS 2541	13,34	13,05	14,17
	1902 - 270	SS 2172	15,94	15,41	18,41
		SS 2541	13,55	13,25	14,73
	1901 - 716	SS 2172	16,49	16,66	18,97
		SS 2541	13,86	13,68	15,82
	1902 - 521	SS 2172	25,56	24,91	23,18
		SS 2541	22,45	21,68	24,57
	GAS	1901 - 184	SS 2172	9,68	10,05
SS 2541			9,39	9,54	10,50
1902 - 092		SS 2172	11,45	11,31	13,16
		SS 2541	10,77	10,41	11,60
1901 - 223		SS 2172	20,51	19,70	19,62
		SS 2541	16,96	17,44	19,08
1902 - 289		SS 2172	22,15	21,81	19,95
		SS 2541	20,75	21,10	21,05

The results from the full test showed a big difference in the measured compound layer thickness under the two types of calibration. As shown in figures 14 and 15 below, this difference varies from a batch to the other depending mostly on the calibration itself. However,

it is worthy to mention that a higher value is generally noticed regarding the second type of calibration regardless of the type of quenching medium used.

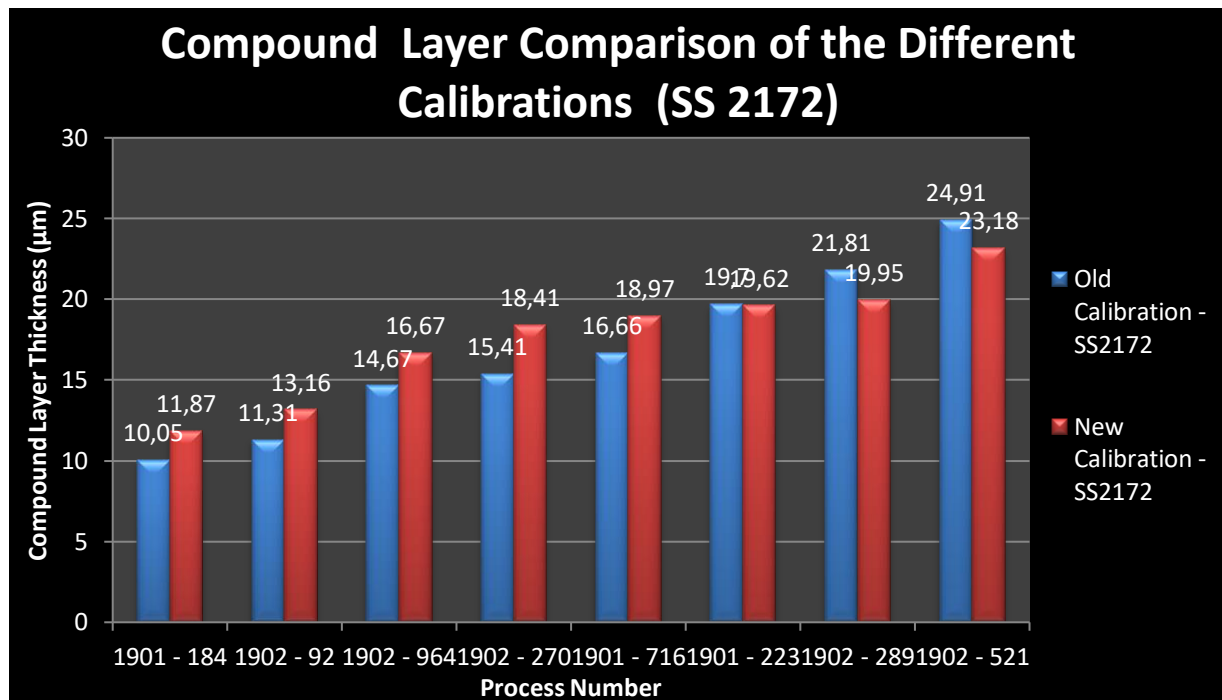


Figure 14 - Comparison of compound layer thicknesses from different calibrations for SS 2172 samples

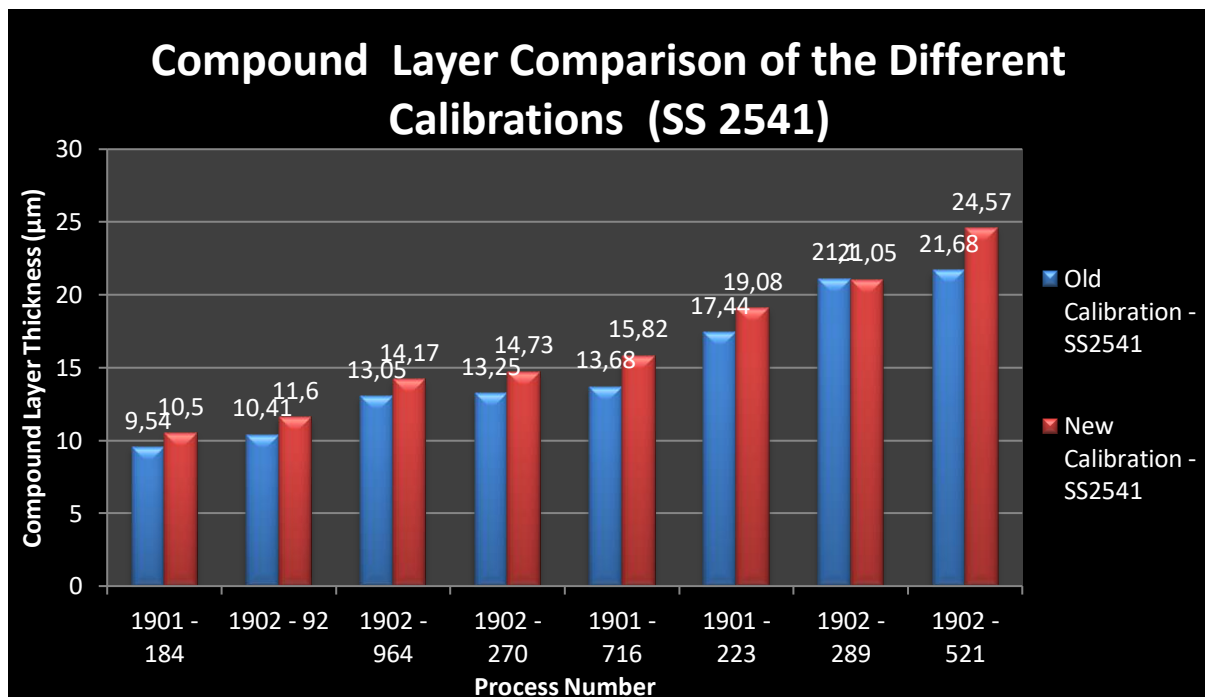


Figure 15 - Comparison of compound layer thicknesses from different calibrations for SS 2541 samples

These two sets of results were then compared to the actual microscopy values as shown in figures 16 and 17 below. It is clearly showed that the separation of both steel grade and

quenching medium factors during calibration is necessary for a closer measured output to the actual compound layer thickness value. A bigger uncertainty is expected if that separation was not fully applied. In fact, the curves in figures 16 and 17 show clearly that the old calibration compound layer thickness values are tightly correlated with the microscopy ones.

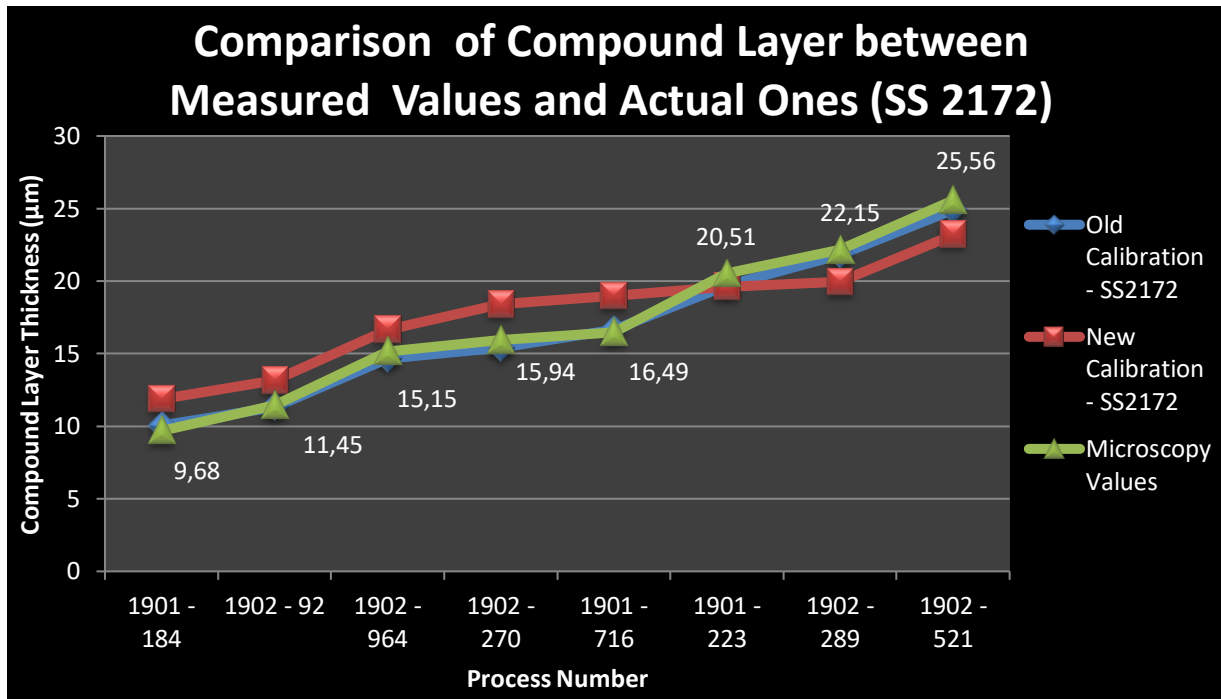


Figure 16 - Comparison of measured compound layer thicknesses to actual ones for SS 2172 samples

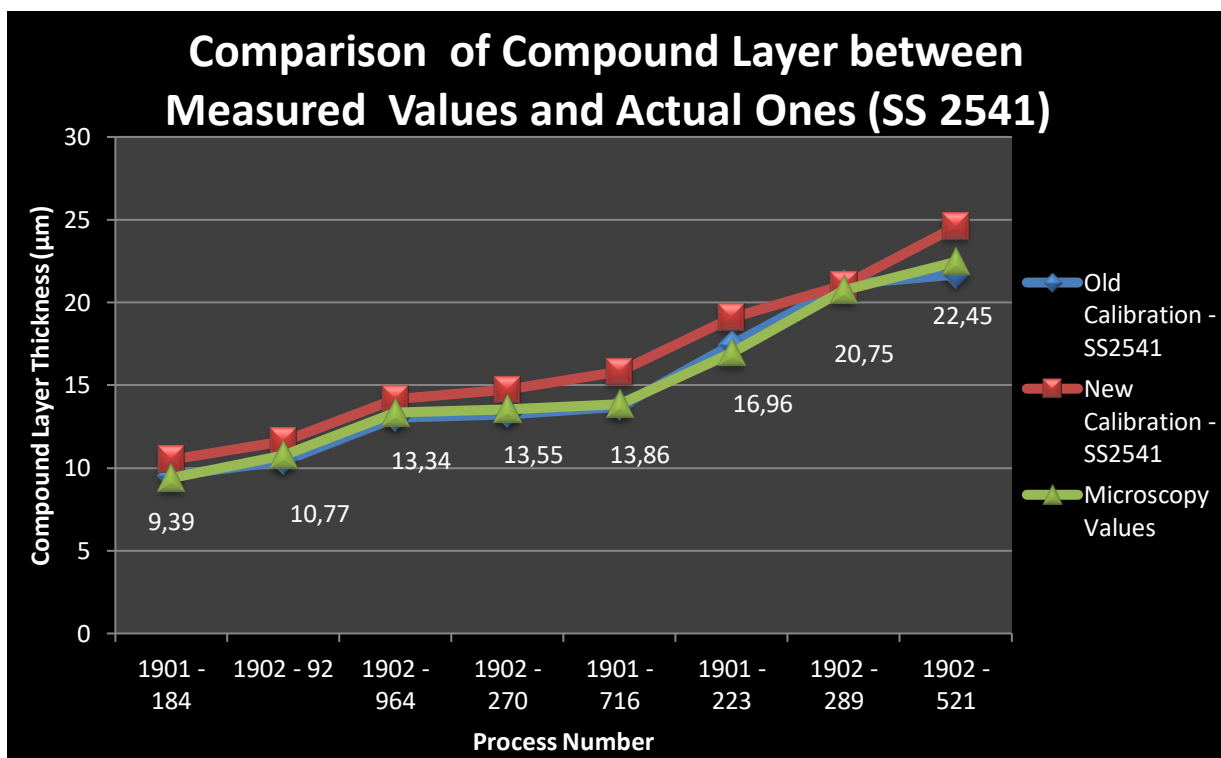


Figure 17 - Comparison of measured compound layer thicknesses to actual ones for SS 2541 samples

Second, measurements regarding the *first samples* have been introduced as well to get a better overview of both *repetition and replica* measurements offered by NORTEST 2060 EC device. The results from these tests can be found in tables 15 and 16 below. On one hand, the results show a clear correlation between the measured and actual values. On the other hand, it was noticed that repetition measurements and replica ones showed very close values under each respective type of calibration.

Table 15 - First samples repetition measurements results

Quenching Medium	Process Number	Steel Grade	Microscopy Measurement (µm)	NORTEST Measurement (Old Calibration)	NORTEST Measurement (New Calibration)
OIL	1902 - 964	SS 2172	15,15	14,92	16,47
		SS 2541	13,34	12,94	13,81
	1902 - 270	SS 2172	15,94	15	17,85
		SS 2541	13,55	13,09	14,82
	1901 - 716	SS 2172	16,49	16,92	18,93
		SS 2541	13,86	14,31	15,77
	1902 - 521	SS 2172	25,56	24,44	23,21
		SS 2541	22,45	22,75	24,55
GAS	1901 - 184	SS 2172	9,68	9,85	11,87
		SS 2541	9,39	9,41	10,72
	1902 - 092	SS 2172	11,45	11,67	13,14
		SS 2541	10,77	10,36	12,08

GAS	1901 - 223	SS 2172	20,51	19,63	19,58
		SS 2541	16,96	17,43	19,93
	1902 - 289	SS 2172	22,15	22,52	19,86
		SS 2541	20,75	20,48	21,45

Table 16 - First samples replica measurements results

Quenching Medium	Process Number	Steel Grade	Microscopy Measurement (µm)	NORTEST Measurement (Old Calibration)	NORTEST Measurement (New Calibration)
OIL	1902 - 964	SS 2172	15,15	14,58	16,37
		SS 2541	13,34	12,71	14,66
	1902 - 270	SS 2172	15,94	15,54	18,60
		SS 2541	13,55	13,29	15,10
	1901 - 716	SS 2172	16,49	16,72	18,95
		SS 2541	13,86	14,08	15,97
	1902 - 521	SS 2172	25,56	25,37	23,55
		SS 2541	22,45	22,36	24,46
GAS	1901 - 184	SS 2172	9,68	9,41	11,84
		SS 2541	9,39	9,25	10,54

GAS	1902 - 092	SS 2172	11,45	11,60	13,19
		SS 2541	10,77	10,58	11,75
	1901 - 223	SS 2172	20,51	20,11	19,50
		SS 2541	16,96	17,08	19,66
	1902 - 289	SS 2172	22,15	22,60	20,31
		SS 2541	20,75	20,42	21,57

Furthermore, it was important to work on *stability measurements over time*. The results from such measurements are presented in table 17 below. It is clear from these results that the values indeed fluctuate over time, yet a more in-depth study of the measurements is needed and cannot be applied unless through a thorough statistical study. On the other hand, it would be important to mention that this study did not take into consideration the operator flaws. Therefore, one must bear in mind that the results shown in the table below do not reflect such a parameter amongst others. The measurements represented in table 17 below belong to the first samples of two different batches which have compound layer thickness values in the middle of the set of measurements.

Table 17 - Stability over time measurements results

Quenching Medium	OIL		GAS	
Batch Number	1901 - 716		1901 - 223	
Steel Grade	SS 2172	SS 2541	SS 2172	SS 2541
Measurements per Occasion (µm)	18,58	15,50	20,52	19,60
	19,10	16,06	20,14	19,56
	19,28	15,26	20,40	21,08

Measurements per Occasion (μm)	19,38	14,96	21,08	21,42
	18,60	15,50	21,00	19,42
	18,80	15,56	21,10	19,92
	19,08	16,26	21,50	18,72
	18,66	15,94	21,16	18,72
	19,32	16,02	20,28	19,38
	19,04	14,96	19,92	18,78

The fluctuations of the four different samples under stability measurements can be summarized in figures 18, 19, 20 and 21 below. No clear evaluations can be deduced from such graphs though; therefore, one must use a statistical tool for that. However, one can assess that the fluctuations are quite random from first sight.

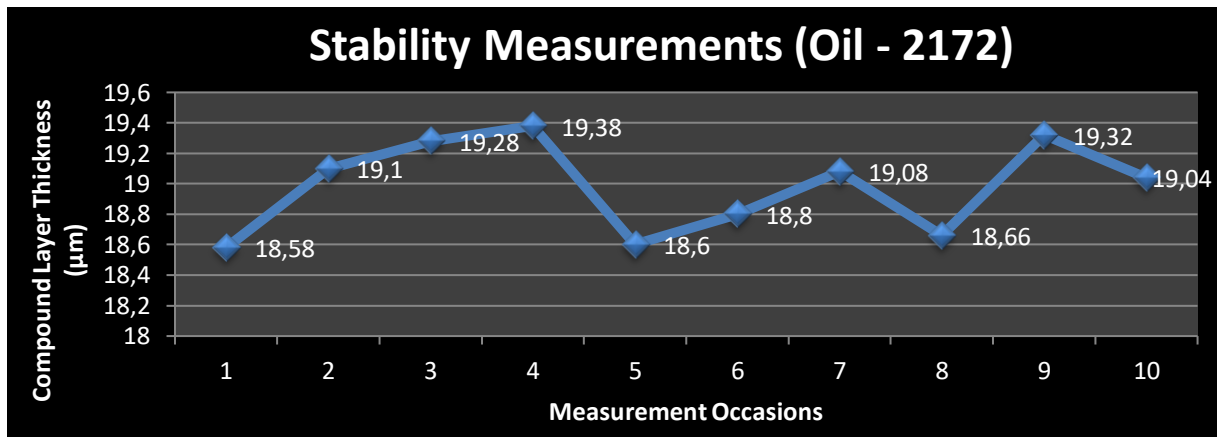


Figure 18 - Stability over time measurements (oil-quenched, SS 2172)

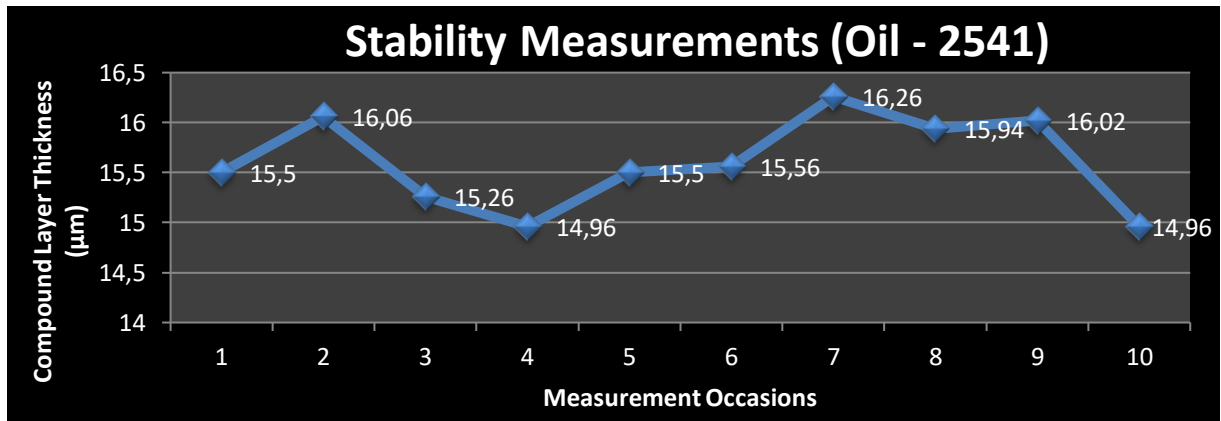


Figure 19 - Stability over time measurements (oil-quenched, SS 2541)

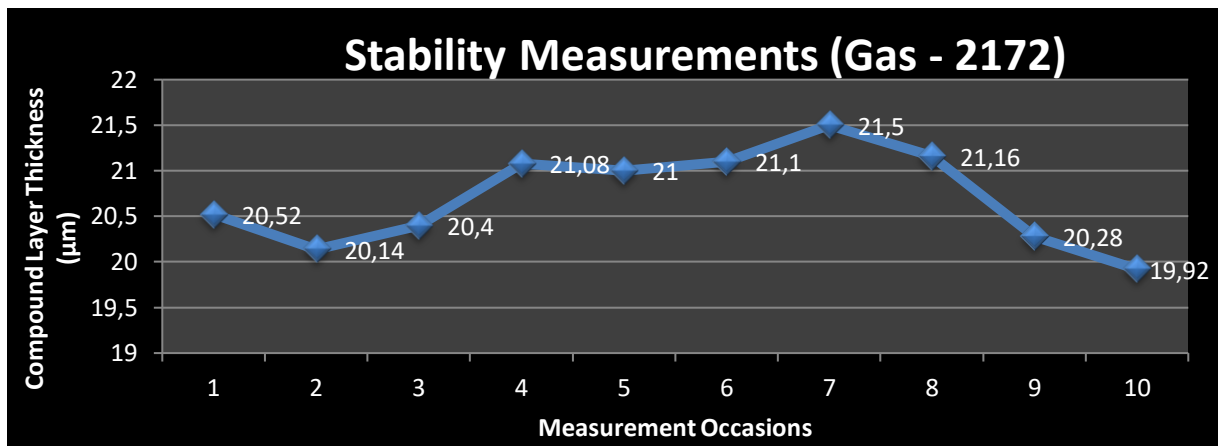


Figure 20 - Stability over time measurements (gas-quenched, SS 2172)

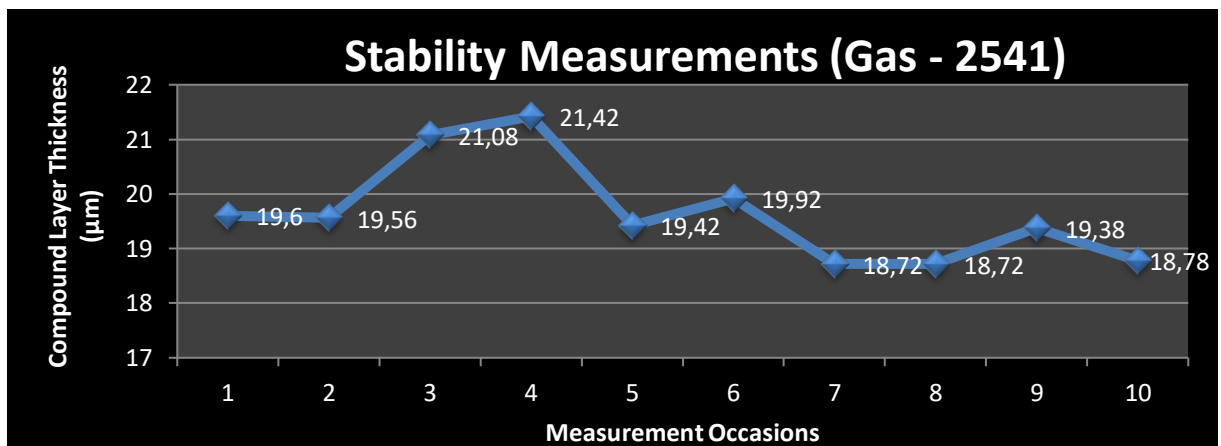


Figure 21 - Stability over time measurements (gas-quenched, SS 2541)

Finally, the last set of measurements using NORTEST 2060 tried to cover the **variation sources**. Such measurements were essential for the assessment of the EC instrument along that of the different operators. As shown in table 18, the operators had very close results for each respective sample. The idea here would be that the variation due to the operator may be lower than that of the instrument. However, one cannot assess the sources of variation by such a simple evaluation. Therefore, a more in-depth statistical study is presented in the discussion section.

Table 18 - Measurement system analysis measurements results

Quenching Medium	Process Number	Steel Grade	Operator 01 Measurements (µm)		Operator 02 Measurements (µm)		Operator 03 Measurements (µm)	
			First round	Second round	First round	Second round	First round	Second round
OIL	1902 - 964	SS 2172	16,42	16,56	16,81	16,64	16,47	16,37
		SS 2541	13,96	14,13	14,29	14,56	13,81	14,66
	1902 - 270	SS 2172	18,09	18,19	18,56	18,48	17,85	18,6
		SS 2541	14,71	15,12	15,16	14,98	14,82	15,10
	1901 - 716	SS 2172	18,72	18,83	19,26	19,02	18,93	18,95
		SS 2541	15,70	16,07	15,91	16,04	15,77	15,97
	1902 - 521	SS 2172	23,15	23,27	23,03	23,42	23,21	23,55
		SS 2541	24,55	24,54	24,59	24,49	24,55	24,46
	1901 - 184	SS 2172	11,84	12,13	11,84	12,09	11,87	11,84
		SS 2541	10,39	10,46	10,53	10,57	10,72	10,54
GAS	1902 - 092	SS 2172	13,15	13,3	13,29	13,45	13,14	13,19
		SS 2541	11,88	11,92	12,17	12	12,08	11,75
	1901 - 223	SS 2172	19,52	19,55	19,19	19,35	19,58	19,50
		SS 2541	19,05	19,26	19,51	20,02	19,93	19,66

GAS	1902 - 289	SS 2172	19,7	19,92	19,46	19,69	19,86	20,31
		SS 2541	21,22	21,40	21,35	21,78	21,45	21,57

4.2.2 Other Nondestructive Equipment

Overview - As expressed previously, one of the thesis goals was to find a suitable replacement for NORTEST 2060 EC device and conduct the same set of measurements as expressed in table 07. Unfortunately, such a device was found till the very end of the project and there was not enough time to conduct the intended measurements. However, a detailed description of the quest followed to find a replacement EC instrument was introduced instead from the very first investigated EC equipment till the suitable one found lately. These EC equipment suitability and investigation (direct or indirect) results can be found in table 19 below. For basic information about these EC devices, please refer to table 08 in section 3.5.2.

Table 19 - Investigation comments of replacement candidates for NORTEST 2060

Instrument Name	Investigation Comments
Elcometer 456	The equipment was not suitable for the application in question. In fact, the difference between the magnetic permeability of the compound layer and the rest of the material was deemed not big enough. Moreover, the tolerance values of this device are too high regarding smaller thicknesses.
Dualscope - MPOR	This instrument was decided off the list when it was proven that only a calibration of maximum two points is possible. After sending some samples to Fischer in Germany, it was concluded that a multiple point calibration feature is not available within the device settings.
MAGNATEST D 3.623	This instrument was directly investigated at KmK Instrument in Västerås. Indeed, the results gave signals within the normalized impedance plane which were quite readable and assessable. As shown in figures 22, 23, 24 and 25 below, a clear representation of the possible comparative studies is demonstrated. However, the low frequency used by this machine (4 kHz) compared to that of NORTEST 2060 (1 MHz) was of concern. By the end, the EC instrument was deemed as economically non-viable (≈ 350 000 SEK) for the application required at Bodycote.

3MA Testing Unit	Due to time constraint, this instrument could not be investigated. However, the latest updates from Fraunhofer IZFP claimed that this multi-parametric magnetic instrument can be used for the determination of the compound layer thickness over nitrided/nitrocarburized steel. When discussed at Bodycote, the instrument was deemed as overqualified as it holds other characteristics besides eddy current assessment such as Barkhausen analysis. Moreover, it was economically non-viable ($\approx$ 966 000 SEK) if used for the determination of compound layer thickness alone.
QUTEC.DELTA. S	This device was not particularly tested during this thesis project. However, both a contact at CNS and a descriptive report assessed that this EC equipment is specifically dedicated for the determination of compound layer thickness of nitrocarburized steel. Moreover, the accuracy of the measurements is set to $\pm 1\mu\text{m}$ alongside a direct numerical output of the compound layer thickness measurement. The equipment is claimed to be a mixture between an EC instrument with a similar working principle as MAGNATEST D 3.623 but with higher frequency value and assistance software from CNS under the name of DELTA.S. Such a solution was provided to another heat treatment company in Germany which had a similar development project as the one in this thesis work. The reason for taking such a long time before getting knowledge of such EC equipment is the exclusive use of German as the only language to introduce it. Clearly, the interest for such equipment seemed to be solely German previously.

MAGNATEST Results - The MAGNATEST D 3.623 EC instrument was personally investigated to determine its usefulness regarding the thesis project goal. As shown in figures 22, 23, 24 and 25, the domains A and B represent respectively the thinnest compound layer thickness and the thickest one. All the R signals between these two domains represent samples with a compound layer thickness outside the calibrated fields. Naturally; the more adjacent the signal to a domain means the closer the compound layer thickness to the value of that specific field. Such a representation could allow comparative studies as to define the compound layer thickness range to which a sample belongs. Nevertheless, an exception imposes itself in figure 25 where a sample with a lower compound layer thickness showed a higher signal than the one for domain B. The reason behind such an anomaly can be found in section 5.1.2. For the resistance (x-axis) and inductive reactance (y-axis) numerical values, please refer to appendix 08.

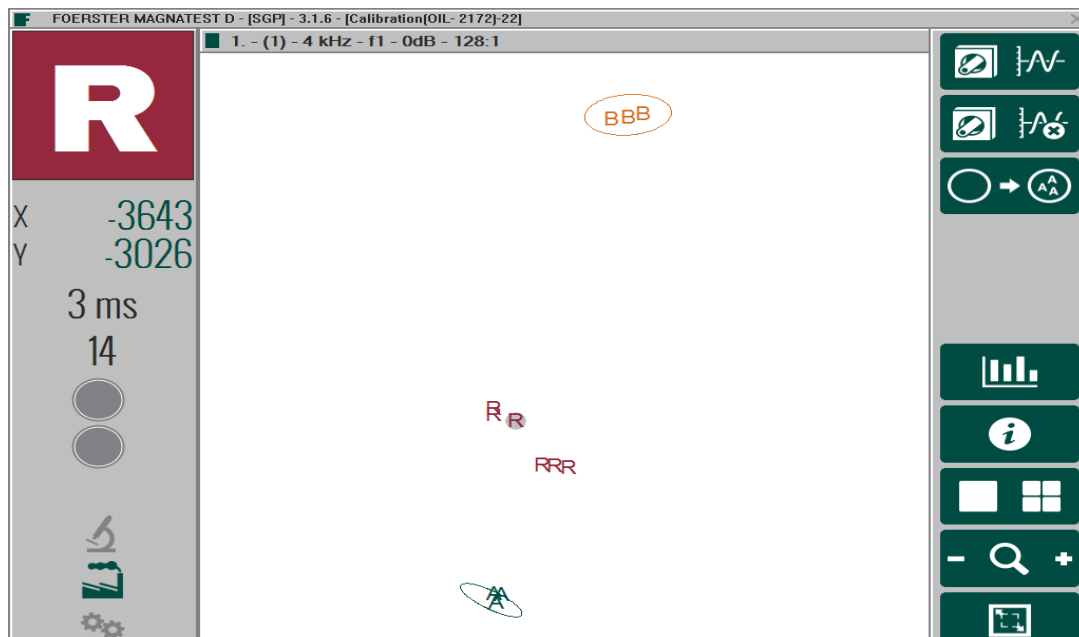


Figure 22 - MAGNATEST output screen for oil-quenched SS 2172 samples

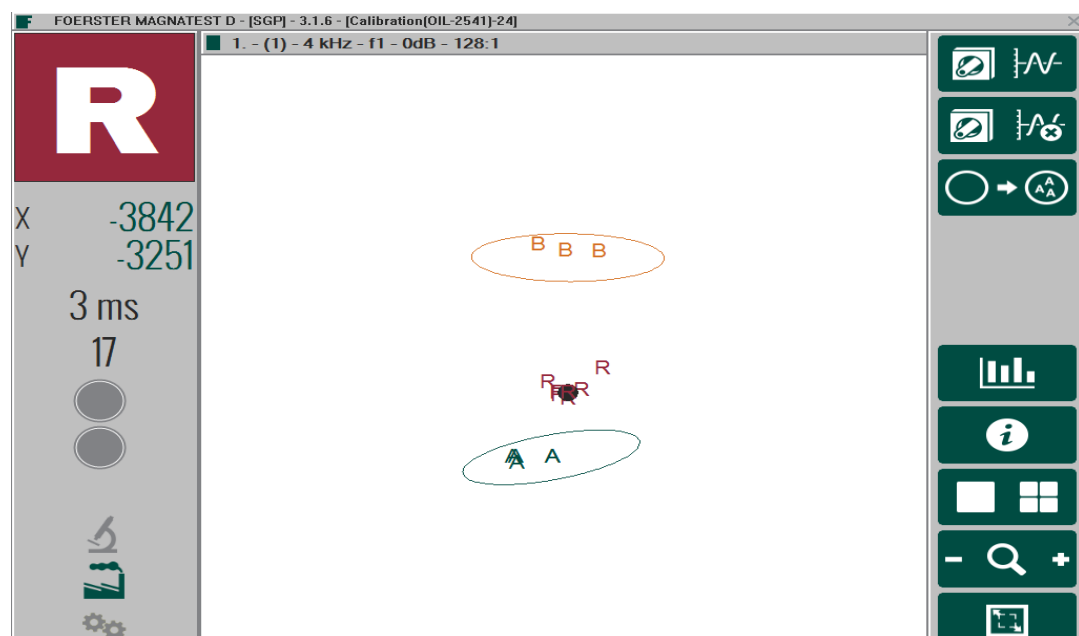


Figure 23 - MAGNATEST output screen for oil-quenched SS 2541 samples

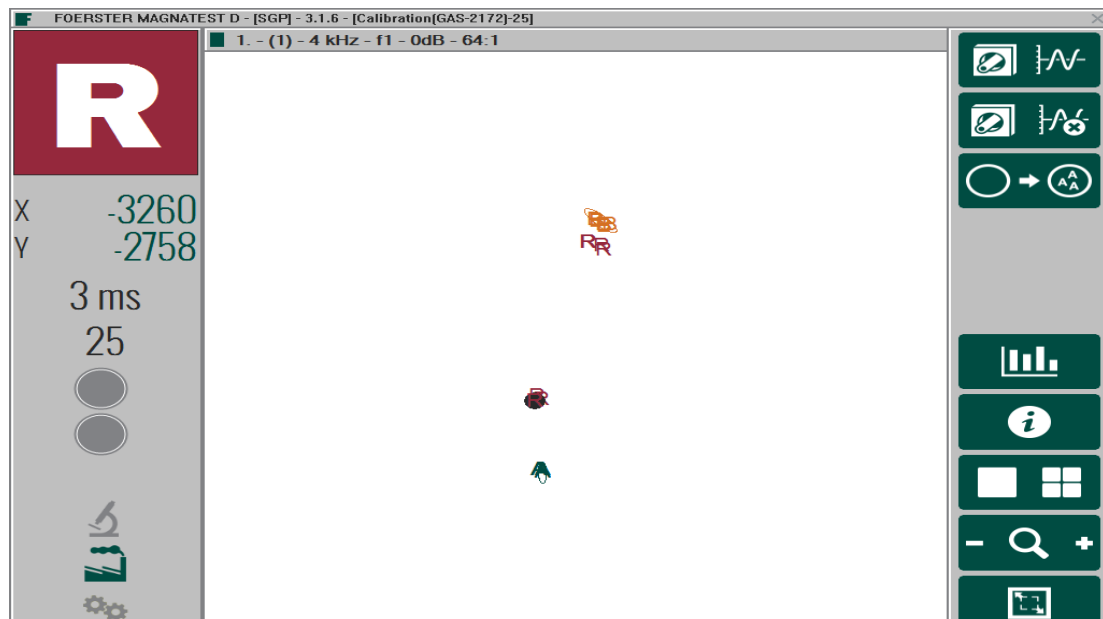


Figure 24 - MAGNATEST output screen for gas-quenched SS 2172 samples

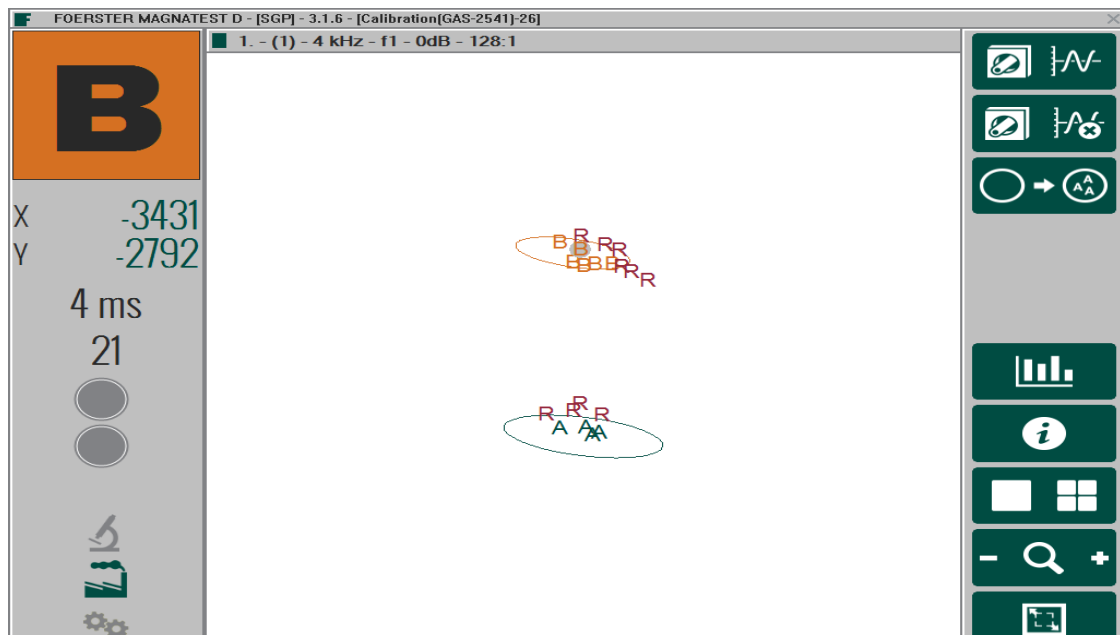


Figure 25 - MAGNATEST output screen for gas-quenched SS 2541 samples

4.3 Metallographic Results

4.3.1 Optical Microscopy

As a completion to the introductory survey, the rest of the metallographic study comes as a continuation to support the previous microscopy results. Therefore, two other samples from each demi-batch were prepared for microscopy (respectively samples 09 and 10). The results obtained from these two-last set of samples were close to those of the first ones. Therefore, it was obvious that the compound layer thickness within each respective batch has some sort of homogeneity as shown in the average values represented in table 20 below. Therefore, the sample preparation of the entire set of samples was deemed as unnecessary.

Table 20 - Entire set of metallographic results

Quenching Medium	Process Number	Steel Grade	Microscopy Measurements (Samples 01)	Microscopy Measurements (Samples 09)	Microscopy Measurements (Samples 10)
OIL	1902 - 964	SS 2172	15,15	14,77	14,96
		SS 2541	13,34	13,30	13,43
	1902 - 270	SS 2172	15,94	16,22	15,05
		SS 2541	13,55	13,31	13,45
	1901 - 716	SS 2172	16,49	16,50	16,65
		SS 2541	13,86	14,53	13,40
	1902 - 521	SS 2172	25,56	24,79	25,21
		SS 2541	22,45	22,85	22,75
GAS	1901 - 184	SS 2172	9,68	9,50	9,77
		SS 2541	9,39	9,53	8,56
	1902 - 092	SS 2172	11,45	11,79	11,32
		SS 2541	10,77	10,53	10,47
	1901 - 223	SS 2172	20,51	20,63	20,07
		SS 2541	16,96	16,39	15,63

GAS	1902 - 289	SS 2172	22,15	20,69	22,84
		SS 2541	20,75	20,01	19,77

From these results, it seems that the use of the first samples only was reliable for comparative studies. To get a clear visual inspection about the metallographic study of samples 09 and 10, please refer to appendix 09 with all remaining optical microscopy images.

4.3.2 Scanning Electron Microscopy

Regarding the results from the SEM study conducted over a sample from batch 1901 - 223, the channels formation near the surface of the compound layer were very clear. These channels form beginning from the upper part of the compound layer and ends towards the surface. Therefore, the information mentioned in the literature review part and illustratively in figure 02 (right) was experimentally proven. For the rest of the SEM images please refer to figure 26 below and appendix 10.

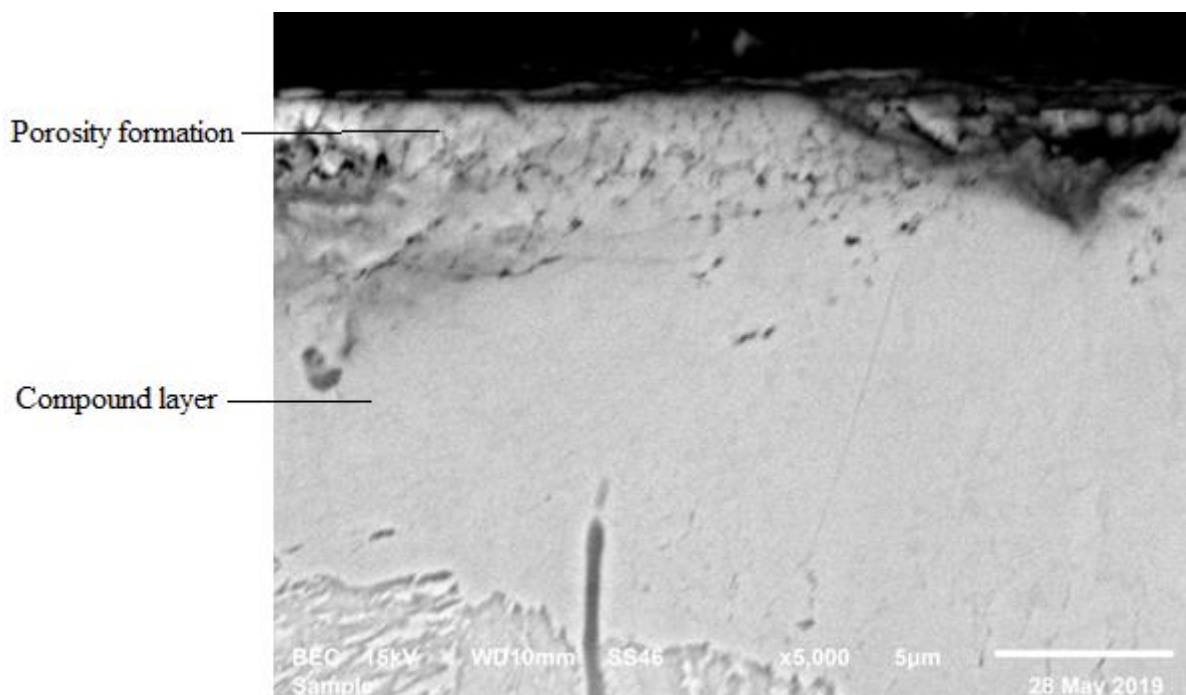


Figure 26 - Scanning electron microscopy image of an SS 2541 sample, 5000x

5 Discussion

In this part, the focus will be aiming at discussing the different results from section 04 in a more in-depth analysis. This part will encompass the use of both NORTEST 2060 and MAGNATEST D 3.623 EC devices. On the other hand, the metallographic study will be discussed in detail with spotlights over the compound layer and porosity level characteristics vis-à-vis the use of EC as an NDE method.

5.1 Nondestructive Evaluation

5.1.1 NORTEST 2060 Assessment

Overview - As expressed in the results section, the evaluation of the first eddy current equipment followed a certain methodology. Therefore, the discussion section will follow the same steps to assess the characteristics of the NORTEST 2060 nondestructive device. This methodology includes mainly the frequency of the device and the compound layer thickness different measurements such as the full test, stability over time along the system variation tests.

Frequency - The use of the USB oscilloscope was dedicated to the determination of the frequency value and waveform. The results shown in section 4.2.1 proved the appearance of two different signals; one variable frequency was expected while another signal with higher frequency and smaller bandwidth was totally unexpected. While brainstorming about this matter, two explanations were believed to maybe be the cause for such frequency signals. The first explanation entails that the first signal (one variable) represents the first connector on the back of the NORTEST 2060 hardware. Indeed, the probe used for this project was linked to that specific connector. However, the second signal which looked more of a pulsed nature was believed to be a result of the second connector port which was not linked to any probe. For more information about the connections of NORTEST 2060, please refer to appendix 04. On the other hand, the second explanation leans towards the possibility that the second signal is nothing but a disturbance from either the material itself or the environment in which it is present. Basically, the difference between the two opinions is that the first sees some order in the signals in question while the second thinks those changes do not represent any. This segment of the project was discussed with a handful number of people, yet the interventions were more of guesses than actual answers. The suggestion that imposes itself here is to check NORTEST 2060 hardware from the inside as there was no serial number on the backside of the instrument. Such a move was not possible, as the instrument is still being used by Bodycote.

Compound Layer Measurements - As the core task of this thesis project requires it to be, various nondestructive measurements using the first nondestructive equipment (NORTEST 2060) gave a set of results as expressed in section 4.2.1. These sets of results were quickly presented then, yet they had to be further discussed. The use of statistical software to achieve that was deemed as essential to get all the required discussions. Consequently, different tools from JMP14 Pro software were adopted such as a graph builder, a control chart builder, a gauge repeatability and reproducibility investigation along an analysis of variance for operators' performance.

The **full test** results could be tracked down to table 14 in section 4.2.1. The main comparisons made for this specific test were between two types of calibrations (Old = Separation based on steel grade and quenching medium & New = Separation based on steel grade only) and to the actual compound layer thicknesses from microscopy evaluation. Doing such an evaluation could help assess the most precise and reliable type of calibration to follow in the future. Through a dedicated use of the statistical software JMP14 Pro, the graph represented in figure 27 below gave an insight over that matter through a comparison of the regression equations belonging to the different quenching mediums under the two distinctive types of calibration. It is noticed from these regression equations included that the slopes from the old calibration measurements (OIL = 94.65% & GAS = 92.29%) with respect to microscopy values are bigger than those for the new calibration ones (OIL = 78.54% & GAS = 76.21%). This fact could be explained as the trueness representation of each calibration type in comparison to the actual compound layer thickness (microscopy values); which proved to be higher when using the old calibration. Therefore, one can assess that separating between the steel grades and quenching mediums is indeed a better option for more accuracy and precision. Such information must be noted as the efficiency of any future measurements would take benefit of this tip.

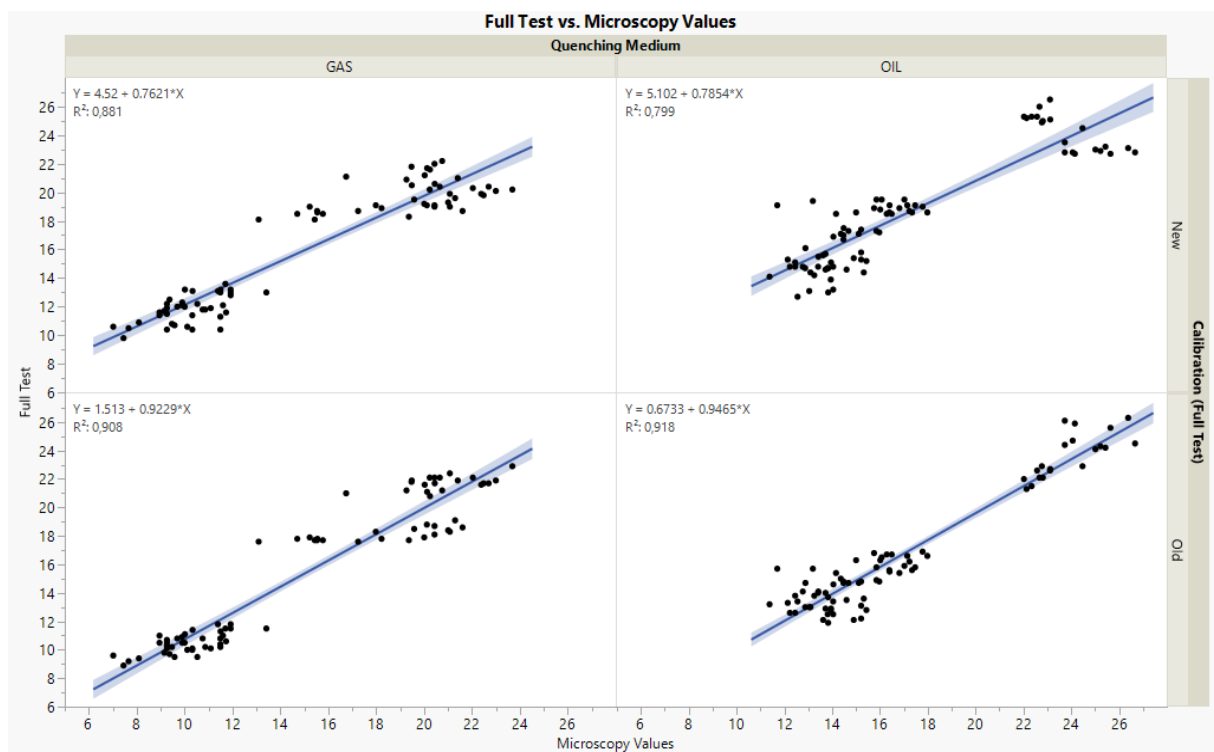


Figure 27 - Correlation between the types of calibration and microscopy values

Moreover, a discussion regarding the **stability over time** was necessary since the evaluation of the set of results was not satisfying enough. Therefore, a control chart builder was used to assess the variation over time between respective subgroups (X-bar chart) and within each one of them (R chart). Each subgroup represents measurements over a sample belonging to a specific quenching medium and steel grade over the period of a month as shown in figures 18, 19, 20 and 21. This study was conducted using JMP14 Pro software as well as shown in figure 28 below with X-bar chart on the top and R-chart on the bottom. The first striking

deduction would be that all measurements are within the upper and lower control limits for both types of charts. This fact imposes that the measurements using NORTEST 2060 keeps being valid even after a month of use. This test alone assessed that the use of the EC device is still valid, while changes within the subgroup (lower graph = R-chart) are noticed to be less intense than between one another (upper graph = X-bar chart). The reason for such a result is that fluctuations within measurements of the same sample will naturally be less than that between different samples. Therefore, it is suspected that the repeatability of the EC device would not cause severe variation when it comes to measurements within a respective subgroup. This study was rewarding indeed, yet another test had to be conducted.

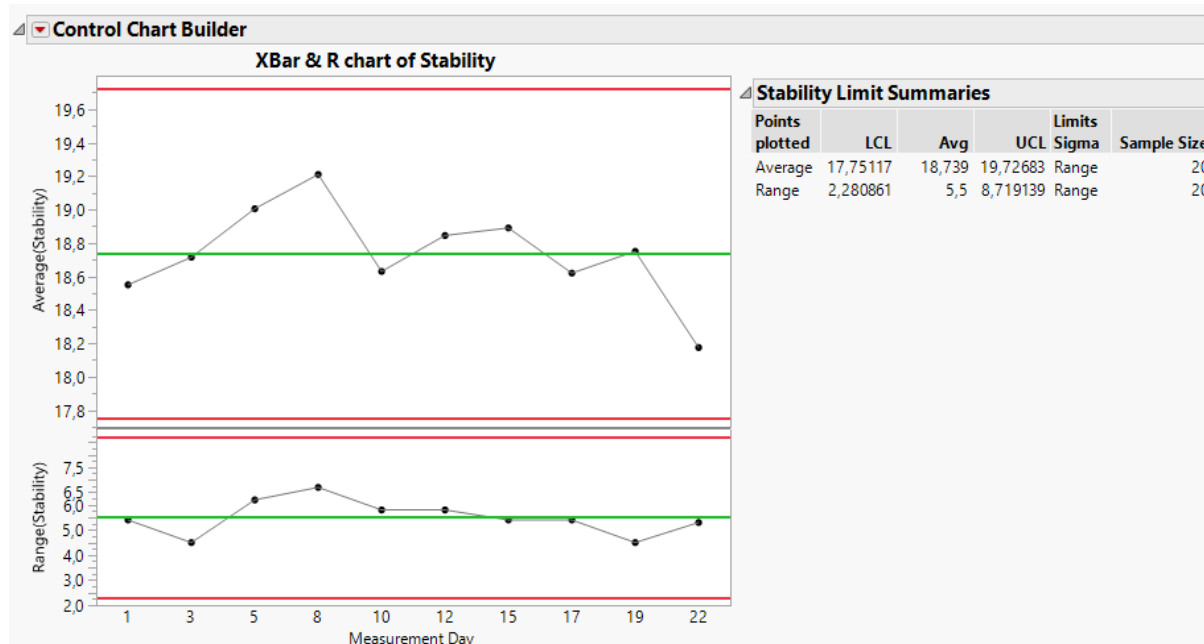


Figure 28 - Stability measurements over time (R chart and X-bar one)

On the other hand, it was still necessary to determine the *sources of variations* along their respective contributions under that matter. Indeed, variations from the operator, the instrument and the parts themselves were expected to show up. However, clarifying the percentages of each contribution was necessary to assess the instrument repeatability and reproducibility. These pieces of information could be found in figure 29 below. In fact, the percentages presented within the figure below prove that the assumption made previously is not fully correct. It seems that the contribution of the repeatability in the variation of the measurement system exceeds 5% and constitutes most of the gauge repeatability and reproducibility. In fact, the reproducibility factor is very low amongst the operators that it could be neglected. Such results prove that the test setup used for NORTEST 2060 helped drastically decrease the operator contribution to the variation in general. Moreover, it was interesting to zoom in over the actual closeness of the operators' measurements further. Therefore, a one-way analysis was pursued and was believed to be a good choice for this thesis work. As shown in figure 30 below, the means and standard deviations from one operator to the other have almost the exact same values. This backs up the theory that any variation within the system due to the operator would be quite small and can be neglected thanks to the NORTEST 2060 setup which does not allow much difference between the operators' performance in general. However, the

setup is ought to be used correctly for such result. From the measurements experience of this thesis project, it was of the utmost necessity to take benefit of the damper as shown in figure 07 and appendix 04. This had allowed a more stable set of measurements and apparently almost annihilated the fact that the operator may be a source of variation.

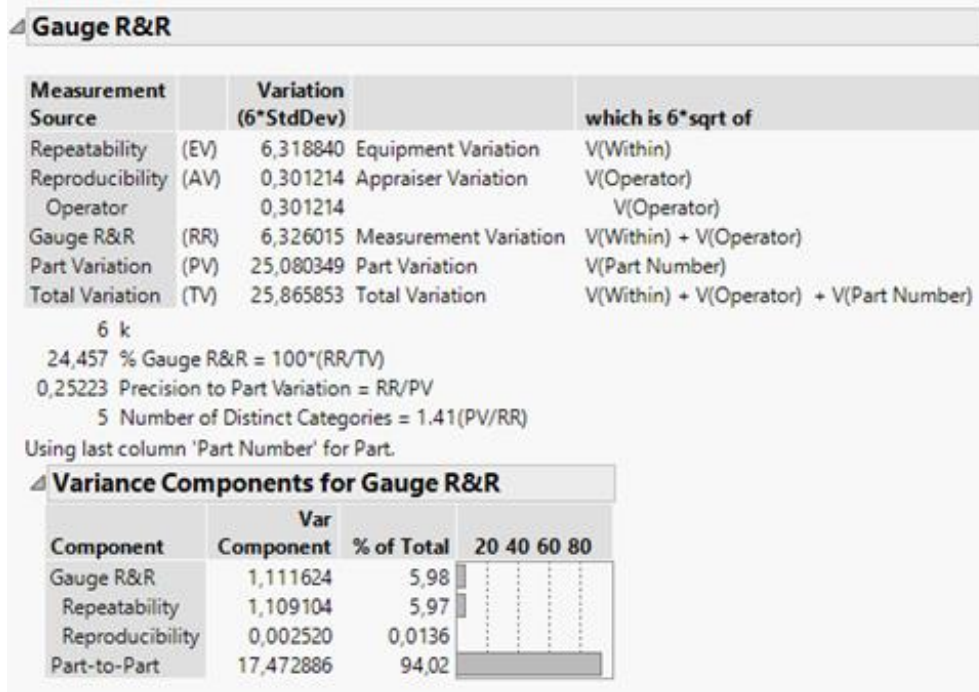


Figure 29 - Variance components for gauge repeatability and reproducibility

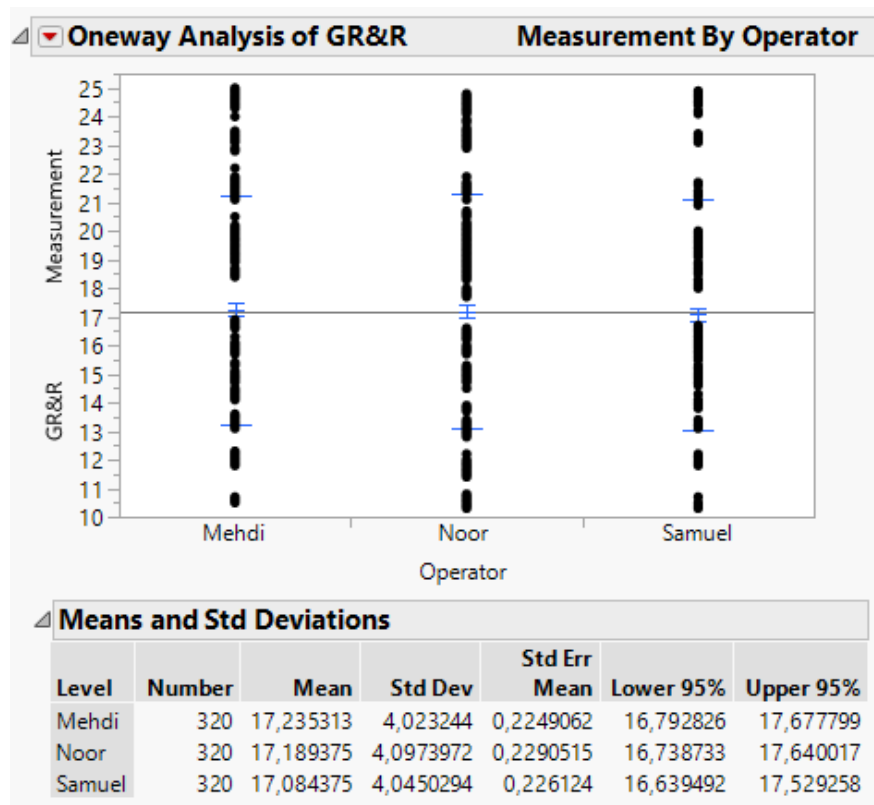


Figure 30 - One-way analysis of the operators' performance

5.1.2 MAGNATEST D 3.623 Assessment

Basically, MAGNATEST D 3.623 is one of the EC equipment which were attentively investigated along the duration of this thesis work. The results shown in section 4.2.2 regarding this EC equipment proved that it can be used for comparative studies of the different compound layer thicknesses. Indeed, a thorough calibration that separates both quenching medium and steel grade could help define the range of thicknesses to which it belongs. However, this general rule was broken under two different occasions which helped prove two main ideas. The first idea as shown in figure 23 demonstrated clearly that if the compound layer thicknesses had very close values, then one cannot comparatively assess the set of results as they may superpose. Regarding the second idea, figure 25 shows clearly that a higher porosity level can cause a higher signal registered than the one expected. This high level of porosity than the expected one can be noticed mainly in samples within batch 1901 - 223. For more details about this matter, please refer to section 5.2.2 below. However, it is important to mention that this signal behavior was noticed with the high alloying element steel sample (SS 2541) but was pretty much absent within the lower alloying element sample (SS 2172). The assessment of this EC equipment was quite interesting for the scope of this project, as it gave a concrete understanding of the normalization principles and impedance plane changes. Finally, the use of such a low frequency (4 kHz) was of concern compared to the NORTEST 2060 one (1 MHz). Therefore, it was believed that such frequency may probably reflect information about the diffusion zone instead of the compound layer one. Nevertheless, it is believed that the compound layer and diffusion zone can be correlated to each other. Consequently, the comparative study could be proven as acceptable.

5.2 Metallographic Evaluation

5.2.1 Compound Layer Thickness

Overview - After conducting all the nondestructive measurements, the project encompassed the use of conventional quality evaluation methods. Although only half of the sample is hot mounted, it was preferred to leave it till the end of the nondestructive measurements phase. The reasons for such precaution can be summarized into two main points. The first one would be the use of a specific liquid during cutting which could maybe lead to some changes regarding the cleanliness of the sample surface. While on the other hand, half a sample is difficult to measure using NORTEST 2060 probe as it may be difficult to succeed in avoiding any measurements over the edge of a demi-sample; especially an SS 2172 one. Such concerns explain one of the reasons why the full tests did not include the first samples, since the thesis work had to undergo the introductory survey at the very beginning.

Introductory Survey - Basically, the most important metallographic evaluations were surprisingly deducted from this preliminary study. First, it proved that the compound layer thicknesses could have quite different values and therefore multiple measurements had to be taken and averaged. The decision had settled on five measurements for each sample. The fluctuations in measurements could be high enough that the range between the minimum value and the maximum one could reach a value of 4 μm . Second, the grinding and polishing of samples from one side was deemed as important to get better microscopy images. In fact,

skipping this step could lead to more fluctuations in microscopy values as the samples used had a wavy surface. Nevertheless, it is also recommended that NORTEST 2060 probe investigates flat surfaces for better accuracy. Moreover, it was proved that sustaining the compound layer for good microscopy imaging could take benefit from wrapping the demi-sample before mounting it. The reason for this is the possible damage that could occur due to consequent grinding and polishing after sample preparation. On the other hand, this study offered an insight over the impact of the different parameters that could affect the final compound layer thickness. These parameters were respectively the holding and quenching properties along the steel grade used.

Rest of Metallographic Study - The discussions resulting from this study were first noticed during the introductory survey but were eventually proved further. The results showed that the differences in compound layer thicknesses have a direct relationship vis-à-vis the holding time. In fact, the higher the holding time would naturally lead to a thicker compound layer. Moreover, the steel grades have a clear correlation with the thickness of the final compound layer. This correlation relies mainly over the alloying elements (Cr, Mo, V, Mn and Ni) percentages within the component in question. In the case of this project, it was clearly shown from figure 11 that SS 2172 samples result in higher compound layer thicknesses after nitrocarburizing than SS 2541 ones. In fact, one can ascertain that the higher the amount of alloying elements the thicker the compound layer. It is also noticed that the gap between the two steel grades increase with a rise in holding time as explained previously. For a clear representation of the alloying element percentages please refer to table 05.

5.2.2 Porosity Level

In addition to the compound layer thickness, another property was deemed as extensively important in the EC detection and correct interpretation of the compound layer thickness. For this study, the values of the different porosity levels were investigated as shown in table 13. In fact, such values were the reason why a clear interpretation was reached. The first step towards that interpretation was to separate the different porosity levels of the different quenching mediums and steel grades. These calculated values could be found in figures 31, 32, 33 and 34 below.

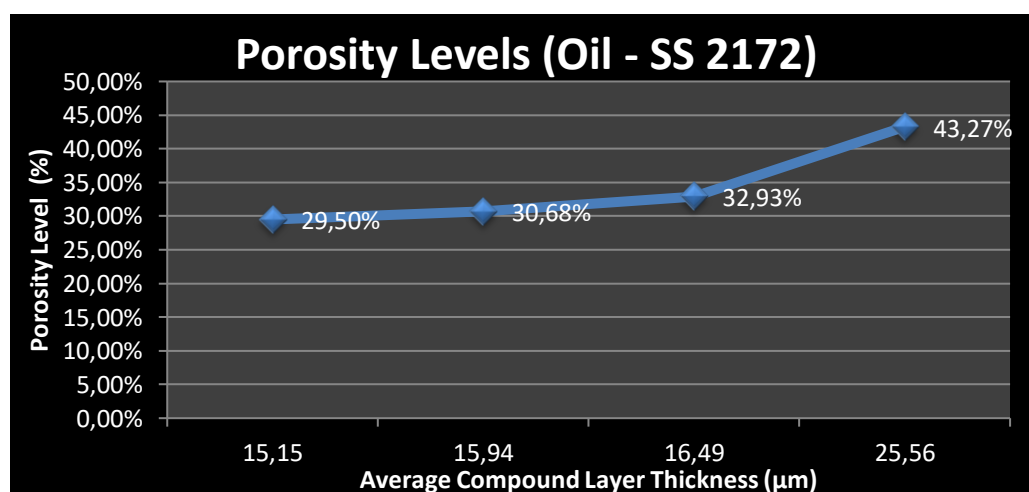


Figure 31 - Porosity levels of oil-quenched SS 2172 samples

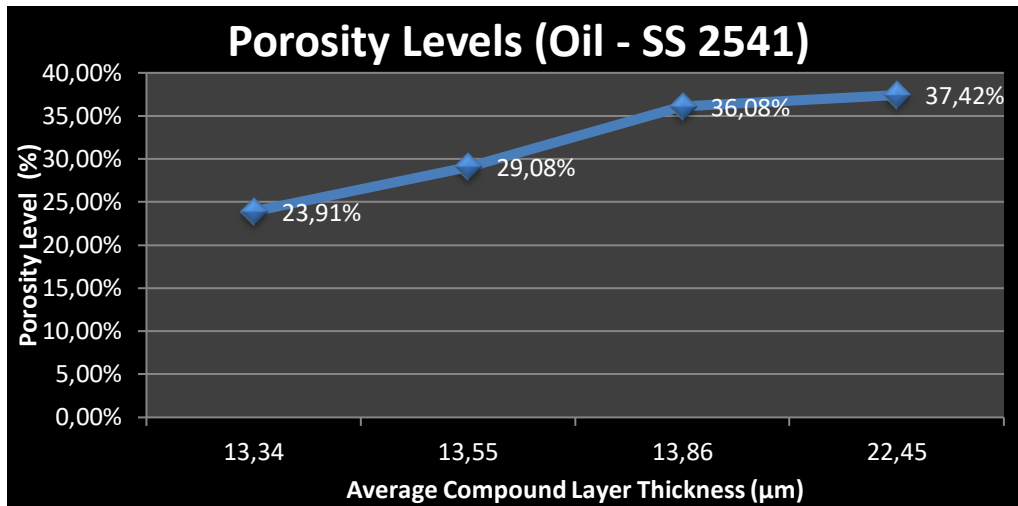


Figure 32 - Porosity levels of oil-quenched SS 2541 samples

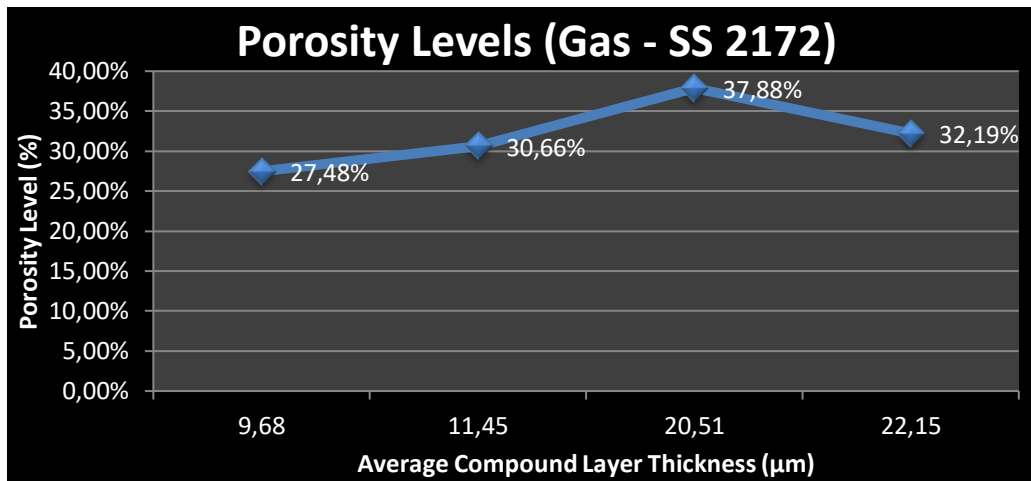


Figure 33 - Porosity levels of gas-quenched SS 2172 samples

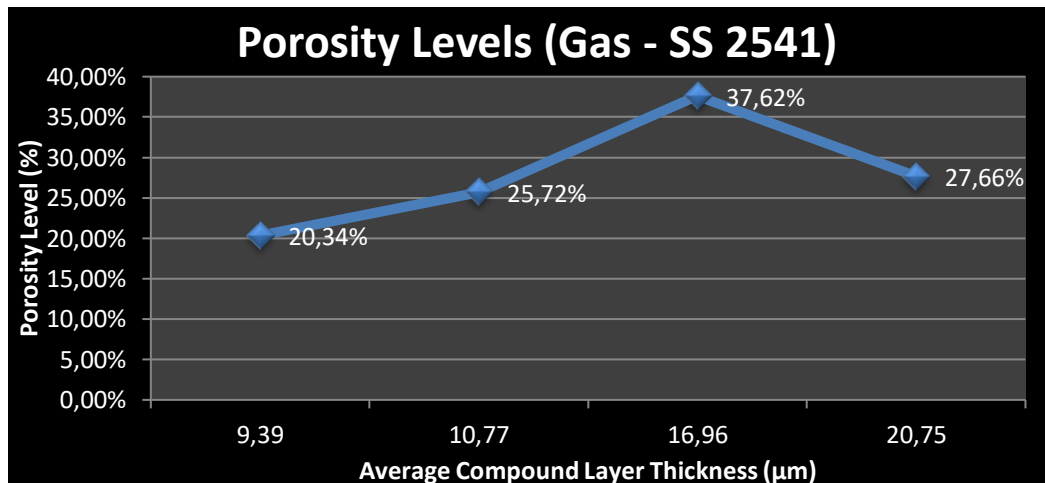


Figure 34 - Porosity levels of gas-quenched SS 2541 samples

From porosity level figures above, one could eventually assess the behavior of the formation of the porous zone on the surface of the compound layer. The porosity layer thickness gets bigger with a higher compound layer thickness. Naturally, all SS 2172 samples have a higher porosity thickness than the SS 2541 present on the same batch. However, looking into

the topic from such an angle would not lead in a way to a worthy discussion. Therefore, the focus was dedicated towards the porosity level instead as a percentage of the porosity thickness within each compound layer. The first remark would be that the porosity level increases respectively and linearly with a rise in the compound layer thickness in general. However, this general rule failed with the third samples of each steel grade under the gas quenching medium as shown in figures 33 and 34. This deviation from the general rule was found out to be a direct result of the higher quenching time of 150 minutes compared to that of 105 minutes for the other samples. See appendix 03. Such a deviation was determined during the nondestructive evaluation as well using MAGNATEST D 3.623 device as partially shown in figure 25. As for NORTEST 2060 results, the calibration of gas-quenched samples included SS 2172 and SS 2541 samples from batch 1901 - 223 in favor of a homogeneous nondestructive study.

So far, the study managed to give very interesting discussion points about the porosity formation and its behavior under different circumstances both theoretically and experimentally. However, this was still not enough to compare between the porosity formation under oil-quenched samples and gas-quenched ones. In fact, such a comparison was not easy to pull out because when the porosity levels from both quenching mediums were put next to each other a simple obstacle yet a challenging one presented itself. This challenge is simply represented in the different compound layer thicknesses between the samples of the two different quenching mediums. Therefore, it was necessary to adopt a forecasting function using excel. Such a forecasting function would allow an approximate determination of the possible porosity level that would take place under the whole set of compound layer thicknesses. Then, the forecasted porosity levels which have real values to oppose to are compared to each other to assess the reliability of the forecast. Regarding the forecast method followed, all the real porosity levels were considered except for those related to batch 1901 - 223 since they represent a deviation from the acknowledged trend as mentioned in the previous paragraph. For more details about the different values of the forecasted porosity levels side by side with the real ones along the difference between the two values, please refer to appendix 11. Moreover, figures 35 and 36 representing the forecasted porosity levels versus the respective compound layers can be found below.

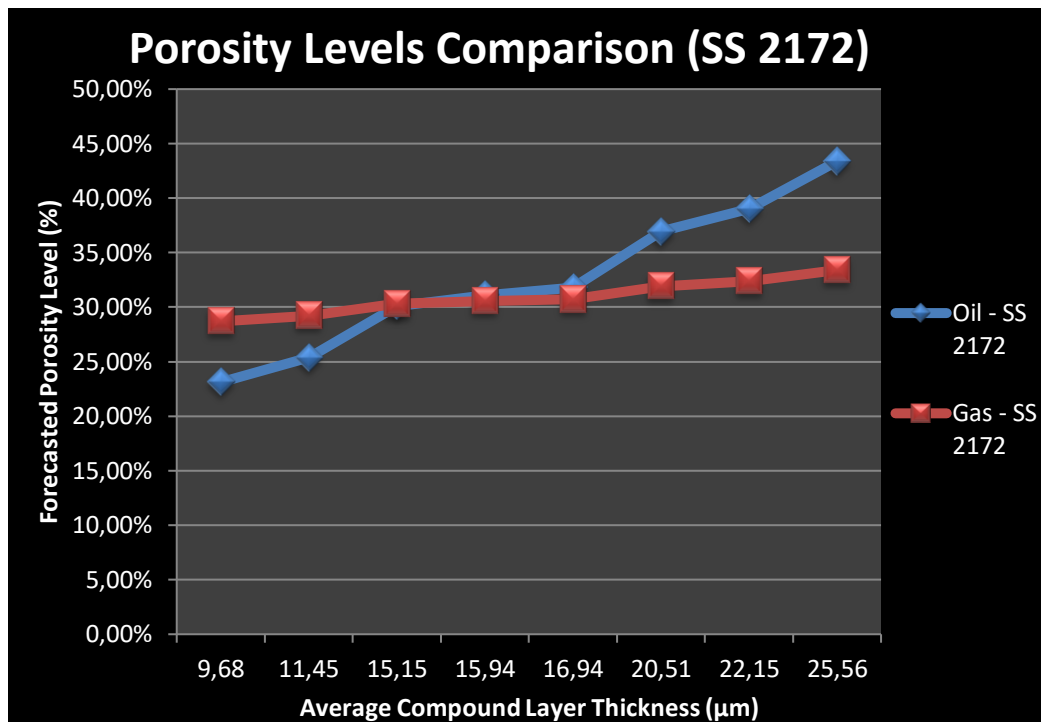


Figure 35 - Porosity levels comparison for SS 2172 samples

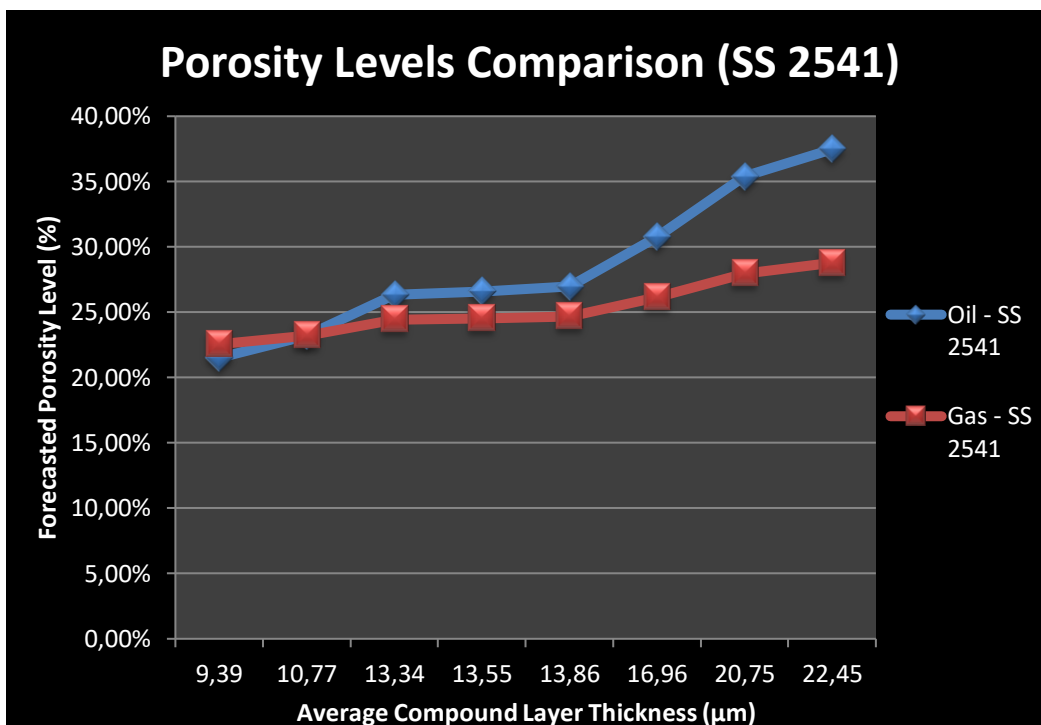


Figure 36 - Porosity levels comparison for SS 2541 samples

From the figures above, it was shown that the porosity levels for both steel grades demonstrate a more stable increase for gas-quenched samples than oil-quenched ones. Moreover, the gas-quenched samples start with a higher porosity level at the beginning. However, this situation changes after a certain compound layer thickness (15 μm for SS 2172 while only 11 μm for SS 2541) as the oil-quenched samples start getting a higher porosity level than the gas-quenched ones. In fact, this gap starts getting bigger and bigger with the increase

in compound layer thickness. Such a distinction goes mainly to the different formation steps of pores under the two quenching mediums. In fact, it was expected for oil-quenched samples to have a lower porosity level than gas-quenched ones over the entire range of compound layer thicknesses. Nevertheless, figures 35 and 36 surprisingly did not go fully in accordance with that. The idea was that oil-quenched samples went through lower duration of heating time and quenching one compared to gas-quenched specimens, therefore a lower porosity level was expected in general. Such an assumption was proved right at the beginning of the graphs in figures 35 and 36 but was shown to be inaccurate for the rest of it. The forecasted porosity level percentages of the oil-quenched samples overpass those of the gas-quenched ones after a certain compound layer thickness. Therefore, one could assess this situation by suggesting that maybe the general rule of oil-quenched samples having less porosity levels than gas-quenched specimens depends mainly on the heating and quenching times along the amount of N within the furnace as well. In fact, it was shown that going beyond a certain level regarding these parameters could practically lead to the inversion of the primary assumption.

6 Conclusions

In this section, a summary of some of the findings is stated along the respective discussion that the thesis project resulted in. The idea was to mention everything in general as to allow the reader to find out in brief about the whole report and the conclusions made about the most noticeable discussed topics. Moreover, suggested future work is included as well for matters which were not fully covered as to allow a smooth continuation of the project if ever proceeded with in the future.

6.1 Summary

Regarding the nondestructive measurements conducted, the following was concluded. First, the frequency used by NORTEST 2060 EC device was defined as 1 MHz with a one variable frequency. Therefore, this value was taken as the minimum frequency required for any new EC replacement for NORTEST 2060. On the other hand, the measurements conducted provided the following information. It was proven through the full test that the separation of the steel grades and quenching mediums during calibration is essential to get better measurements. Moreover, the stability over time test showed that the fluctuations are within the control limits and therefore NORTEST 2060 results were assessed as reliable. Regarding the gauge repeatability and reproducibility analysis, the main reason for the system variation was naturally the part-to-part aspect. However, the contribution of the repeatability of the EC device was found to be much bigger than that of the reproducibility. Therefore, it was concluded that the NORTEST 2060 setup serves its purpose especially when handled correctly. In fact, the careful use of the damper was found out to be essential to obtain good results. Finally, the means and standard deviations from different operators using the one-way analysis confirmed that they have indeed very close values.

As a summary to the metallographic study, two main points were addressed thoroughly which are respectively the thickness and porosity level of the compound layer. Regarding the thickness, it was proved that the holding time and quenching properties had a detrimental role in determining the compound layer. Therefore, one should keep close attention to such parameters during any planned process. Also, a clear knowledge about the steel grade properties was also deemed as interesting since it was proven that the number of alloying elements for example has a direct impact over the final compound layer thickness. On the other hand, porosity was a crucial parameter in the nondestructive evaluation and therefore had to be investigated metallographically as well. It was shown through the discussion part that the amount of porosity grows as the compound layer thickness experiences a rise. Such a conclusion was already found in various literature reviews but had to be experimentally proven. Also, the contribution of the quenching time in the formation of the porous zone was found out to have a direct relationship; a longer quenching time leads to more porosity. However, what was important to mention as well was the concept of porosity level which basically means the percentage of porosity thickness within a compound layer. This concept was used to determine the difference between the amount of porosity formation under oil and gas quenching circumstances. The results were quite fascinating as it was concluded that oil-quenched specimens have lower porosity than gas-quenched ones under a certain compound layer

thickness. However, such a conclusion starts changing as the compound layer thickness passes a certain value (15 μm for SS 2172 and 13 μm for SS 2541). It was concluded that such a shift basically depends on the amount of N within the furnace alongside the usual properties which are the heating time and quenching one.

6.2 Discussion

Through the work done in regard of this thesis project, one could assess through sections 1.3 and 6.1 that most of the goals were successfully met. In fact, the only ones which were not investigated and discussed are the tasks which aimed at conducting measurements with the NORTEST 2060 EC device replacement known as QUTEC.DELTA.S. Such a limitation was mainly due to the scarcity of such EC equipment in the market in general along the exclusivity of German as the language of description. However, the project was quite successful in determining NORTEST 2060 properties and what to expect from the replacement device in term of uncertainty levels, working principle and frequency properties. Moreover, the measurement methodology followed to assess the suitability of NORTEST 2060 was successful in doing so. Therefore, applying such a measurement methodology on other EC equipment would be quite fruitful as well. On the other hand, the use of MAGNATEST D 3.623 was not disappointing at all as it helped get an overview of how the impedance changes could be reflected into resistance and inductive reactance values. Indeed, the use of the EC equipment from Foerster helped assessing the impact that porosity has over the EC measurements and to experimentally prove it. Furthermore, a clear understanding of the normalization principle helped in determining the most suitable EC equipment to fill the NORTEST 2060 task. In general, the project helped in determining the different parameters that should be investigated when using EC as an NDE technique within the heat treatment business. These properties are mainly holding/quenching times, quenching mediums and steel grades. As a matter of fact, these properties were proved to be direct contributors to the compound layer thickness and porosity level which in turn influence EC readings. On the other hand, it is firmly believed that in case different contributors to the heat treatment community decide to find a permanent use of NDE in quality evaluation procedures, many benefits could be met both economically and environmentally speaking. Indeed, it would allow many changes that would alter the conventional idea about quality check processes. Finally, it is believed that such a project will always require improvements and further work. A set of suggested future steps can be found in section 6.3 below.

6.3 Outlook

As for the future work, it is suggested to first fill the gaps that resulted from this project. The very first step would naturally be the use of QUTEC.DELTA. S over the set of nitrocarburized samples in the same manner that was conducted using NORTEST 2060. Only then a comparative assessment could be made and so the efficiency along reliability of the QUTEC.DELTA. S EC device would be determined. Furthermore, it is still suggested to get a better look over the working principles of NORTEST 2060 and especially the frequency signals. It is ought to find out the reason behind such a frequency behavior. Moreover, a deeper understanding of the normalization principle along others would help determine the functioning

manner of any suggested new EC equipment in a better way. A further study was also suggested when dealing with MAGNATEST D equipment as to determine the correlation between the different thicknesses of the compound layer and diffusion zone. Such a study will help assessing the use of low frequency values vis-à-vis different nitrocarburized specimens and even be used to find out experimentally the correlation between the compound layer and the diffusion zone. This project would have also taken great benefit from the use of simulation methods to determine variables such as electrical conductivity and magnetic permeability of the different constituents of a nitrocarburized steel component namely the compound layer, diffusion zone and base material. It is believed that such a study would help assess closely the behavior of the EC equipment as they rely heavily on these two properties. Therefore, it is suggested to have a closer look on that matter as well. Finally, a continuous search for new equipment would be key into developing further knowledge about the topic. Bearing in mind that the trick of such an application is in fact related to the software that translates the x and y values into final numerical results, a close investigation on how such a software works is believed to be a good start to further understand the principles behind the translations such EC devices like NORTEST 2060 execute to get that final numerical value.

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8 Appendices

In this section, a set of documents, tables and images are being introduced as a completion to the work described above. The content of this part is somewhat a detailed explanation to some of what was previously mentioned.

Appendix 01 - Initial Project Idea and Planning Gantt Chart

The initial project idea was the first well-organized plan to proceed with all along the duration of the thesis. It was presented to the supervisors at the beginning of the thesis work and was agreed on during the first couple weeks of the thesis.

Background A great deal of information about Nondestructive Testing (NDT) and nitrocarburizing would be necessary to work on this project. The experimental part of this project would be mainly performed at Bodycote plant in Angered and partially at RISE IVF lab in Mölndal, while the academic supervision from Chalmers would be led by Gert Persson from the department of Industrial and Materials Science. The duration of the project is set to go roughly from January 7th to June 7th.

Purpose In order to determine the compound layer thickness at different positions all over the test piece, the use of NDT would be the best way to go for it at first. NDT is usually used to reduce costs and lead times associated with surface verification. Eddy current was specifically chosen as the best option for this thesis work as it is a surface NDT technique capable of determining the thickness of surface layers as it does not have a high depth of penetration in general. Nevertheless, the project will eventually require the use of conventional metallographic methods as well. This will imply some sample preparation from cutting to mounting to polishing along microscopy verification. Eventually, a comparison between the measured values and the actual ones will be conducted.

Aim The essential aim of this thesis project is to reach a set of compound layer thickness values from measurements using two different EC equipment which would be eventually compared to actual ones from microscopy. This will allow the conception of a measurement methodology to follow during future thickness measurements along a comparison between the two EC test equipment. If the results were deemed reliable, this project could include the implementation of the measurement settings followed on actual production-related elements.

Work Procedure and Important Steps

Choice of a precise experiment plan

Literature survey

Experimental Study

- ✚ Grind a set of samples from one side for better microscopy images by the end
- ✚ Conduct Eddy Current tests over the set of samples after nitrocarburizing using both test equipment
- ✚ Prepare a chosen set of samples (three to five from each batch)
- ✚ Verify layers using optical microscopy

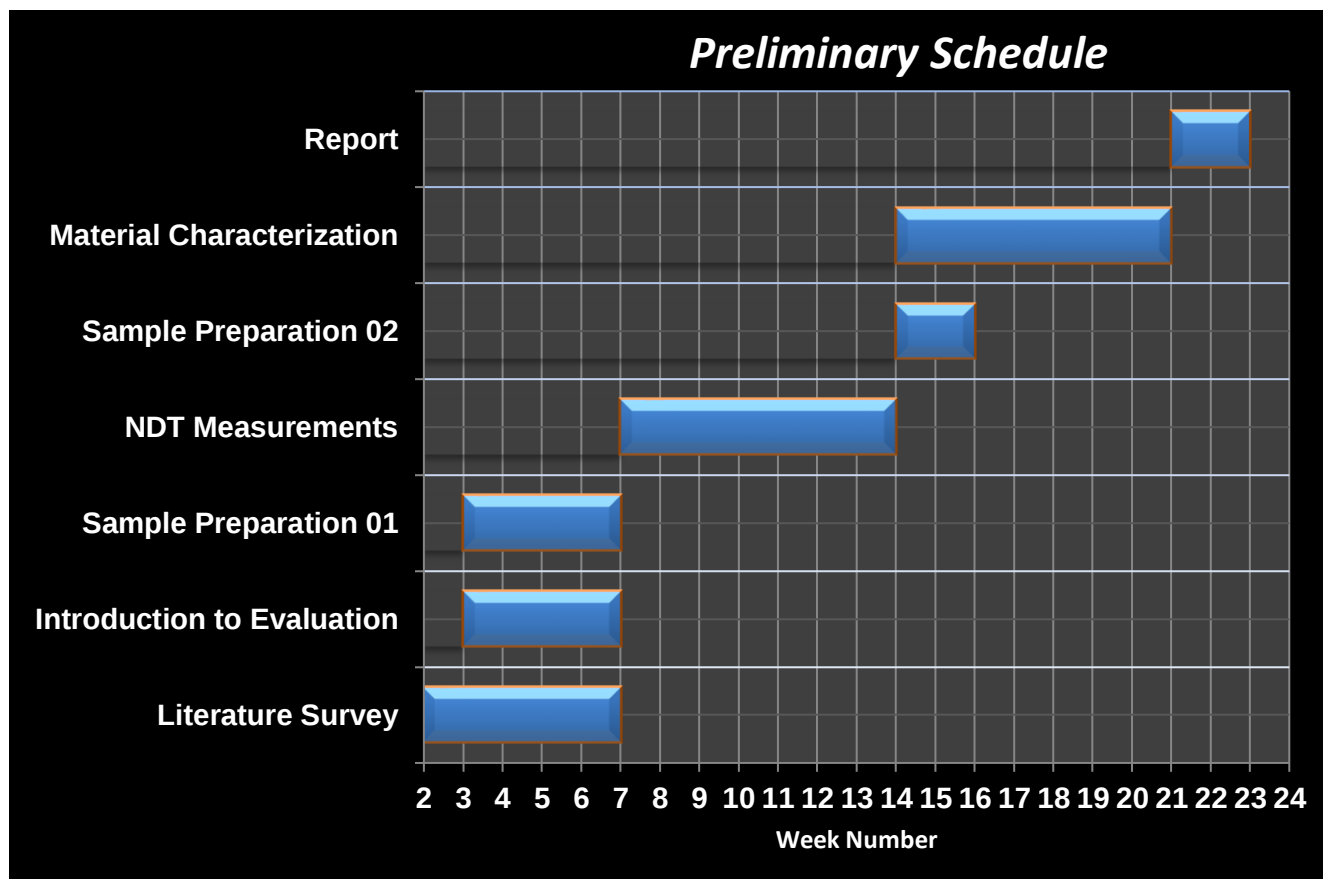
Evaluation of results

- ✚ Analyze Eddy current results for both equipment
- ✚ Develop the expected tolerance of the new Eddy Current equipment
- ✚ Analyze microscopy images of the chosen set of samples

Discussion and Conclusion

- ✚ Comment on the results obtained by both NDT equipment and conventional method
- ✚ Comment on the efficiency of the methodology followed about all the variables in question
- ✚ Use the measurement methodology, if possible, on actual production-related elements
- ✚ Discuss the possibility of implementing the methodology in an actual production environment

Planning Gantt Chart At the beginning of the thesis, an approximate planning chart had to be made as well. This preliminary schedule can be expressed in the Gantt chart below.



Appendix 02 - VärmeBehandlingen Centrum Meeting Summary

VärmeBehandlingen Centrum (VBC) meetings are an opportunity for different Swedish heat treatment companies and research centers to meet and discuss various projects. Each year, the members gather at the Schenströmska Herrgården in Ramnäs, Sweden. During the meeting, members share ideas and try to reach a decision regarding the budget allocation for next year's research projects within heat treatment applications. As a matter of fact, this master thesis project was financed at a previous version of the VärmeBehandlingen Centrum meeting. The project was intended to be completed by year 2018, yet it was sort of delayed due to several reasons. A presentation of the first four weeks of the project was revealed at the VärmeBehandlingen Centrum Meeting on January 31st, 2019. The presentation took 20 minutes and explained briefly what was accomplished so far along the intended steps for the next few months.

During the presentation, several representatives from different companies and research centers were present. Indeed, it was an opportunity to understand the interest that VärmeBehandlingen Centrum members had towards such a project. Moreover, it was very interesting to hear the input, comments and remarks that the audience had to offer. This was a fruitful event for the project as it allowed having a clearer picture of the future steps to be taken. The attendees were from several Swedish companies and research centers such as AGA Gas, Bodycote, Curtiss Wright Surface Technologies, EFD Induction, Epiroc Drilling Tools, GKN, Höganäs, LEAX, Ovako, Parker Hannifin Manufacturing, RISE IVF, Sandvik, Scania CV, Schlumpf Scandinavia, SKF, Swepart, Swerim and Volvo Group.

In the presentation, a set of information about the project was shared with the audience. First, an introduction of the purpose and background of the project was made clear. This intended mainly to catch the attention of the audience. As a matter of fact, they seemed to agree that the use of NDT helps reduce both the cost and lead times of the quality control process. Second, the intended work procedure from nondestructive testing measurements to optical microscopy to the evaluation of results was revealed and explained to the audience. Then, a small introduction to the literature survey topics was presented briefly. This was to clarify the concepts of both nitrocarburizing and NDE methods for those who do not work extensively with one or both these concepts. Then, a validation of the choice of Eddy Current was contested and determined as valid for the scope of the project. Consequently, a clear description of both Eddy Current equipment (Nortest 2060 and Elcometer 456) were made vis-à-vis their respective specifications. Furthermore, a clear description of the test matrix with its five main components (quenching type, steel grades, expected depth range, number of samples and number of test measurements) was clearly explained. Eventually, a set of preliminary results were presented as well showing both NDT measurements using Nortest 2060 and microscopy images. This was followed by a comparison and discussion of preliminary results along their validity. By the end, a set of future steps were unveiled followed by the actual references used for the presentation itself. After the end of the presentation, some of the audience declared that their names were present on the references slide along interesting comments and encouragements.

Appendix 03 - Process Properties

Table - Process properties of oil-quenched samples

OIL - Quenched	Expected Depth Range	8 - 10 μm		10 - 15 μm		10 - 20 μm		+ 20 μm	
	Actual Depth Range (μm)	<u>SS</u> <u>2172</u>	<u>SS</u> <u>2541</u>	<u>SS</u> <u>2172</u>	<u>SS</u> <u>2541</u>	<u>SS</u> <u>2172</u>	<u>SS</u> <u>2541</u>	<u>SS</u> <u>2172</u>	<u>SS</u> <u>2541</u>
		14-16	12-14	12-17	11-15	15-18	12-15	24-27	20-24
	Process Number	1902 - 964		1902 - 270		1901 - 716		1902 - 521	
	Holding Parameters	70min at 580°C		100min at 580°C		135min at 580°C		240min at 580°C	
	Quenching Parameters	20min at 60°C		20min at 60°C		20min at 60°C		20min at 60°C	

Table - Process properties of gas-quenched samples

GAS - Quenched	Expected Depth Range	8 - 10 μm		10 - 15 μm		10 - 20 μm		+ 20 μm	
	Actual Depth Range (μm)	<u>SS</u> <u>2172</u>	<u>SS</u> <u>2541</u>	<u>SS</u> <u>2172</u>	<u>SS</u> <u>2541</u>	<u>SS</u> <u>2172</u>	<u>SS</u> <u>2541</u>	<u>SS</u> <u>2172</u>	<u>SS</u> <u>2541</u>
		9-11	7-10	10-13	9-12	18-22	13-19	19-24	17-21
	Process Number	1901 - 184		1902 - 92		1901 - 223		1902 - 289	
	Holding Parameters	70min at 580°C		100min at 580°C		220min at 580°C		240min at 580°C	
	Quenching Parameters	105min at 100°C		105min at 100°C		150min at 100°C		105min at 100°C	

NORTEST 2060

Operation Manual

(Self-Translated from German)



Figure - NORTEST 2060 setup

Summary

1. Measuring Principle and General Information
2. Device Connections and Setup of the Measuring System
3. Monitor Display and Keyboard
4. Measuring with NORTEST 2060
5. Documentation of Application Numbers
6. Statistics - Functions
7. Options
8. Printer

1) Measuring Principle and General Information

- **Measuring Principle**

The NORTEST 2060 serves to secure the quality of nitrided specimens by measuring the thickness of the compound layer. With the eddy current method, the layer to be measured is brought to a defined distance using a measuring probe through which a high-frequency current flow. The distance is determined by the wear-resistant ceramic or carbide pole of the measuring probe. The magnetic field of the probe induces (on the specimen surface) eddy currents which eventually lead to impedance changes. Such impedance changes are in fact related to the thickness of the compound layer. Eventually, any change regarding the impedance is then processed as an electronic signal in the form of a voltage measurement. After digital conversion, the signal is transferred to the screen as a final numerical compound layer thickness value. Required parameters are determined by a single calibration procedure using determined thickness values. The operation of the device is supported by several functions; the most frequently required are F01 to F10; the ones that are used less frequently are called upon using the function key F09. The measuring tasks used for calibration are combined into groups as the following.

Table - Application groups and their respective application numbers

Application Group	Application Numbers
1	1 ... 20
2	21 ... 40
3	41 ... 60
4	61 ... 80

- **Handling the measuring probes**

The probe has a wear-resistant pole made of ceramic or hard metal. Moreover, it is important to mention that the probe pole dimensions are what determine its sensitivity. Avoiding probe pole sliding over the test piece surface is recommended. In fact, a vertical attachment movement and resurrecting the probe to its initial position after each use is how measurements should be conducted. From time to time, it would also be preferable to clean the probe pole using a soft, alcohol-wetted cloth. This would help remove any abrasives or dust stuck on it.

2) Device Connections and Setup of the Measuring System

- **Device Connections**

The data transfer between the measuring device and the computer takes place via the serial interface COM1. The digitally connected NORTEST 2060 is led out on the back of the device to a 9-pin socket. From there, the connection to the 9-pin plug-in connection takes place. On the back of the device, there are also two circular connectors for measuring probes (at the lower part of the back side). The probe to the left is assigned for application groups 1 and 2, while the one on the right is dedicated for groups 3 and 4. For more details, refer to the figure below.

Connections on the back

Main connection (220/230 V)

Keyboard, Mouse

COM1 interface (connect with NORTEST)

Free connection (do not use)

Printer (use one of the USB connectors)

Monitor

NORTEST Digital output to the COM1 interface

Probe connections

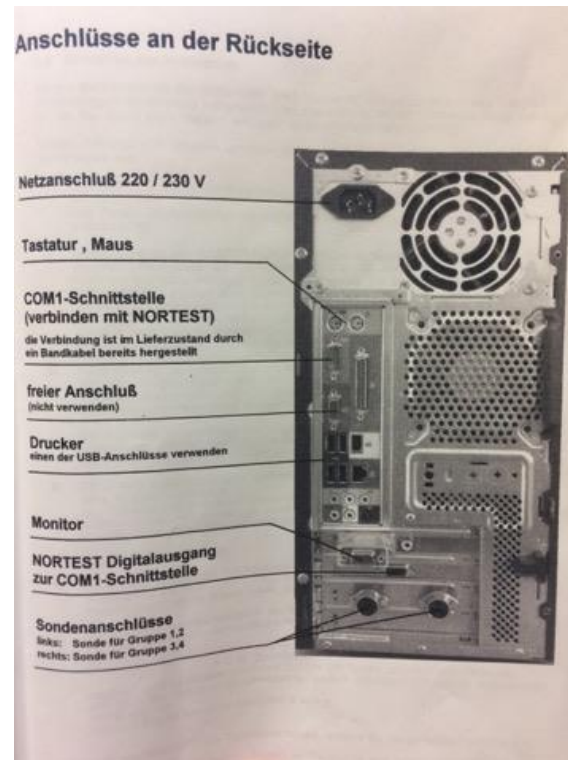


Figure - Illustration of the connections on the back of the hardware

- **Setting up the Measuring Stations**

The NORTEST 2060 software is fully installed and therefore the device is ready for use once received, plugged in and respectively calibrated.

3) Monitor Display and Keyboard

- **Monitor Display**

The monitor displays the following constituents. For a visualization of the monitor display, please refer to the figure below (in German though).

- ⇒ Head Strip - Display the device type and date
- ⇒ Field 01 - Display the symbol for the selected measuring task
- ⇒ Field 02 - Large display of the thickness measurement
- ⇒ Field 03 - Display of the current statistics of the measurement conducted
- ⇒ Field 04 - Display of the operating instructions
(Get adjusted automatically when changing the operating status)
- ⇒ Field 05 - Symbols of the function keys (F1 to F10)

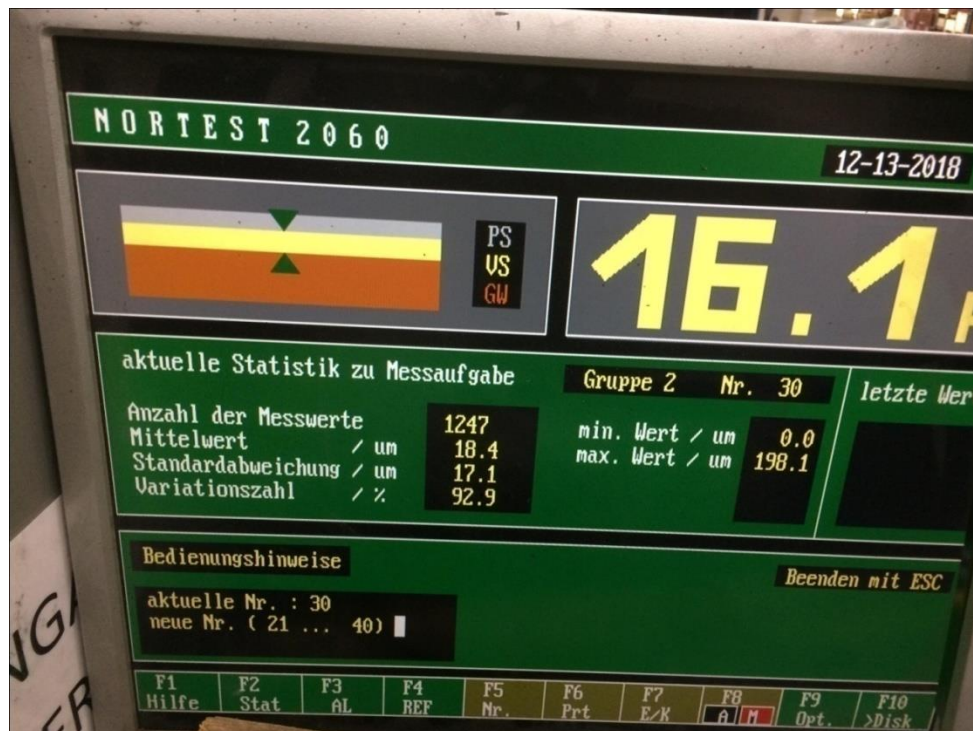


Figure - Monitor display (in German)

- **Keyboard**

On the other hand, the different keys on the keyboard can be used to accomplish a respective set of tasks as shown below.

- ⇒ Space Bar - With this key, the respective next operation is initiated
- ⇒ F01 - Display key functions F1 to F10 on the monitor
- ⇒ F02 - Initiate statistical functions beyond the used ones (shown in field 03)
- ⇒ F03 - Delete outlier
- ⇒ F04 - Initiate a reference measurement (probe against air). It helps get the reference value at layer thickness zero into the computer. Any transition from one application group to another should pass by this step.
- ⇒ F05 - Change of application number within an application group
- ⇒ F06 - Create a printable file named proto.txt
- ⇒ F07 - Calibrate or correct the measuring task. The calibration remains stored for future measurements.
- ⇒ F08 - Choose between automatic and manual data acquisition
 - // Automatic => Output by simply bringing the probe to the measurement position
 - // Manual => Output by bringing the probe to the measurement position and pressing the key RUN
- ⇒ F09 - Call options
- ⇒ F10 - Save all the measurement values on the hard disk as a safety measure. When exiting the program, all data will be backed up to the hard disk.
- ⇒ Esc - Terminate the program

4) Measuring with NORTEST 2060

The probe number is to be understood as a function one. Each probe can have different numbers depending on the intended application. Each group comes assigned with twenty empty application numbers which can be filled by intended calibrations respectively. Before any calibration, keep the probe for at least 40 mm away from the measurement object (against the air) and press RUN. On the other hand, help can be requested using the function key F01, while by using function key F02 and pressing A will display a histogram for interpretation. Finally, a transfer from one application group to another can be done through the function key F04.

A familiarization with the main functions is necessary. As the handling of the probe is very simple, a focus over the program is the only thing which remains for the user to investigate. For each calendar day, a folder is automatically generated in which individual readings, statistical analyses and histograms are stored. One can get into this folder using the window-logo key on the keyboard.

5) Documentation of Application Numbers

The application numbers are not assigned to any specific measuring task, yet they are reserved for a specific application group. Thus, the user has the freedom to assign each application number to a specific measuring task. In the case of NORTEST 2060 at Bodycote Heat Treatment in Gothenburg (for example), the following application numbers are being used.

Table - Application group/number used at Bodycote Heat Treatment in Gothenburg

Measuring Task	Application Group	Application Number
SS 2172 - Oil quenched	1	11
SS 2541 - Oil quenched	1	12
SS 2172 - Gas quenched	2	30
SS 2541 - Gas quenched	2	31

Prior to any calibration, a series of samples with already known compound layer thicknesses are required. These thicknesses must have been determined previously using a conventional method such as microscopy imaging. For a successful calibration, one needs a minimum of four calibration samples (better 5 to 6) for each application number used. The calibration samples should have minimum and maximum values which hold the expected future thickness measurements in between. For example, if the expected thickness measurement is 25 μm . One must preferably use for example the following calibration thicknesses: 14 μm , 20 μm , 24 μm , 31 μm and 37 μm . The values of the calibration thicknesses should avoid being too close from each other. For example, the following calibration thicknesses: 22 μm , 23 μm , 24 μm , 25 μm and 26 μm will not be suitable. As noticed in the example, the difference between the minimum and maximum calibration value is only 4 μm therefore no meaningful calibration can be achieved. A neat calibration would result in more accurate future compound layer thickness measurements. When carrying the calibration steps, measure each sample one after the other and enter the thickness values which were determined through a previous conventional test.

For a better understanding of the calibration procedure, please refer to the steps below.

- Press function key F7 to start new calibration
- Input the application number desired to calibrate (which belongs to the group number currently used). If the number was already calibrated before, the following text will show up: // Number already occupied // Do you want to overwrite this number?
- Decide whether to continue the calibration or not by pressing the respective keys J or N
- Enter the number of the calibration tests that you want to conduct (a minimum of four and a maximum of twenty)
- Place probe on the sample and initiate measurement either automatically or manually when you will have to press the RUN key. When dealing with small samples, it would be preferable to use the manual option to make sure the probe head is entirely touching the sample surface before any measurement. This would allow avoiding any erroneous results in the calibration and therefore ensure the reliability of future measurements.
- Continue doing so till the end of the calibration tests (number determined by the user)

6) Documentation of Application Numbers

NORTEST 2060 has a set of statistical functions which can be summarized in the following.

- Display statistics on the screen (**Displayed continuously**)
 - ~ See sections 03 & 04 for more information
- Print those statistics displayed on the screen (**Call F02 then key D**)
- Display histogram on the screen (**Call F02 then key A**)
 - ~ This statistic function shows a histogram of all measured values displayed in the active application number up to current time.
 - ~ The left side of the output on the screen represents the histogram, while the right side represents the following explanations
 - General Information => Measuring Task
 - Number of Readings
 - Class-Specific Information => Group Number
 - Group Limits
 - Number of Values in the Group
 - ~ To return to normal operation, press RUN
- Print the histogram (**Call F02 then key H**)
- Clear statistics memory (**Call F02 then key L**)
 - ~ This function is used to delete all statistical memories of the actual application number (even the histogram memory)
 - ~ It is used when a new series of measurements is planned
 - ~ The statistics are not stopped with ESC key as they will be stored on the drive
- Clear a runaway (**Call F03**)

- ~ Delete an outlier during a measurement procedure. The value deleted is indicated in “last values” section by a red star in front of the reading.
- ~ This value gets deleted from both the statistics storage and histogram memory.

7) Options

The main functions offered by the equipment are called upon via function keys from F01 to F10. Others less frequently needed can be called upon using the function key F09. These less frequently needed options can be summarized in the following two tables.

Table - First part of the secondary functions

Options	Description	Call Option
Analog Digital Converter (ADC) Direct Display	Show the signal values that the ADC transmits from the electronic signal to the computer.	Call F09 then key A
Measurement Signal	Display the signal delivered and processed by the electronics before conversion into thickness values.	Call F09 then key M
Characteristics	Display the relationship between the measuring signal and the thickness value.	Call F09 then key K
Numbers Occupied	Display all existing calibrations in the respective application group.	Call F09 then key N
Deletion of Previous Calibration	Delete the calibration parameters and the statistics memory of an application number. This option is used to delete calibrations without re-calibration.	Call F09 then key L

⇒ Continuation of the options will lead to the next table of available options.

Table - Second part of the secondary functions

Options	Description	Call Option
Signal at the End of Automatic Measurement	If this option is selected, an audible signal will be given after taking a measurement.	Call F09 then key S
Automatic Delay	Select the delay time to wait between placing the probe on the target and starting the measurement. This delay time will take effect during automatic reading.	Call F09 then key D
Print Line Length	Choose the number of readings that appears in a print line of the printable file.	Call F09 then key Z
Last Used Application Length	Show the last calibrated application number for control purposes.	Call F09 then key B
Printer Control On/Off	Enable or disable the creation of printable files.	Call F09 then key P

⇒ Before returning to normal operation, a reference measurement (probe against air) is made. Hold the probe away from the sample surface by at least 40 mm and press RUN. This takes the user back to the normal operation.

8) Printer

For each calendar day, a folder is created automatically named after that date (MM-DD-YY). For NORTEST 2060, the following files shown in the table below constitute the elements of the folder which can eventually be printed. These files are structured as text files and can be edited into notepad as well.

Table – NORTEST 2060 data packages

File Name	Representation
Proto.TXT	Log Header
Line.TXT	Individual Measured Values
Stat.TXT	Statistical Evaluation
Histo.TXT	Histogram

Appendix 05 - Images of the First Nondestructive Equipment



Figure - NORTEST 2060 test setup at Bodycote Heat Treatment in Gothenburg



Figure - NORTEST 2060 probe tip



Figure - Other NORTEST setup



Figure - Other NORTEST probe like that of the NORTEST 2060

Appendix 06 - Rest of Tomography Images of a Supposedly Identical Probe

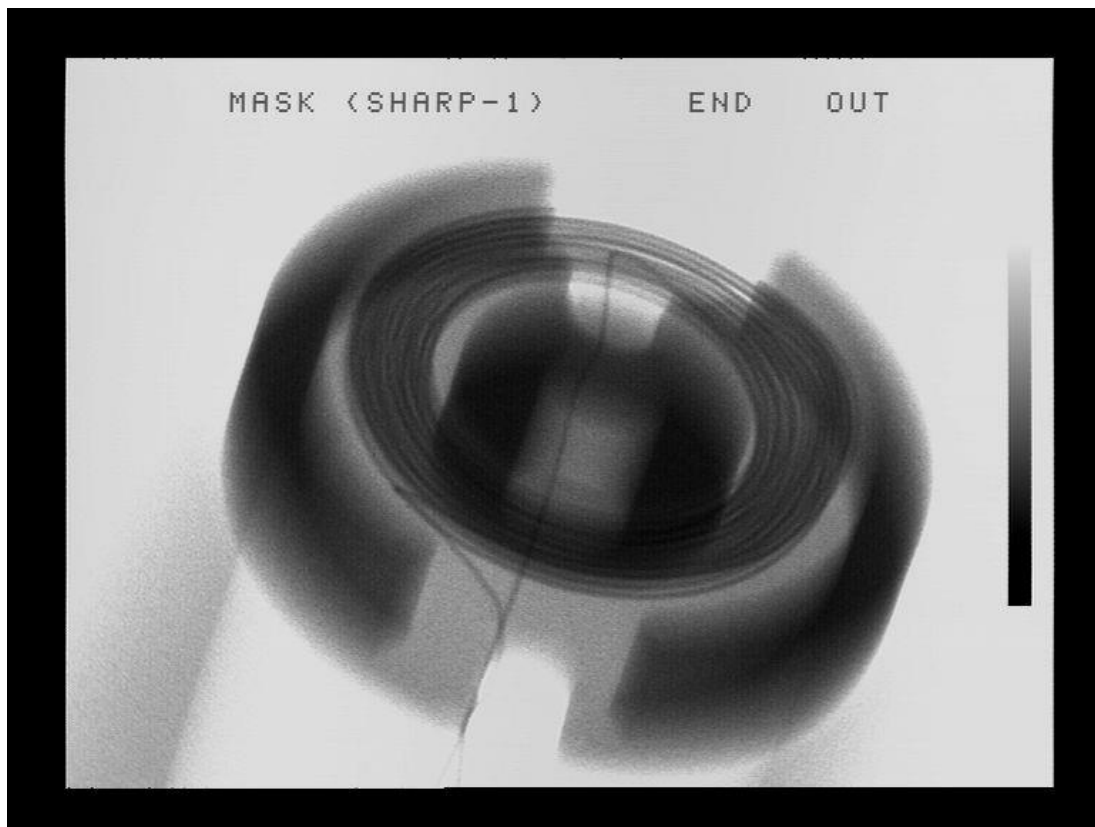


Figure - Probe tip view from the bottom

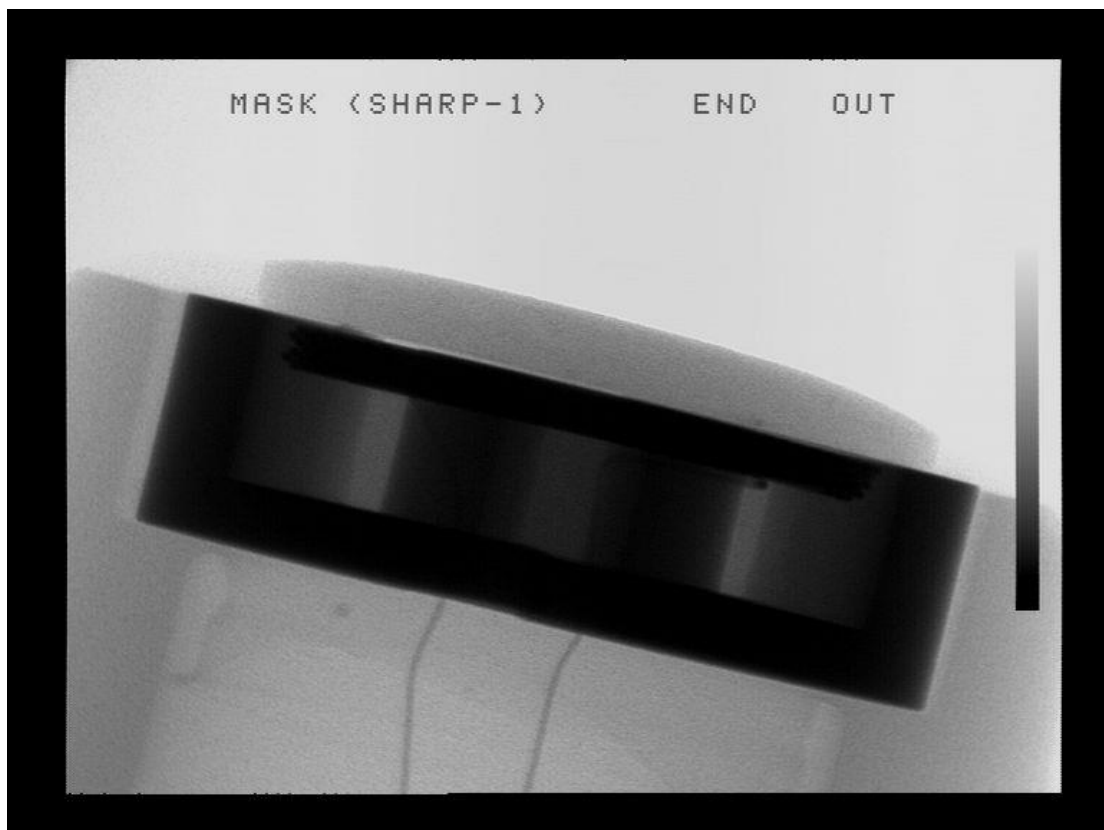


Figure - Probe tip view from the side

Appendix 07 - Images from the Introductory Survey



Figure - First samples from batch number 1902 - 964 (SS 2172 above and SS 2541 below)

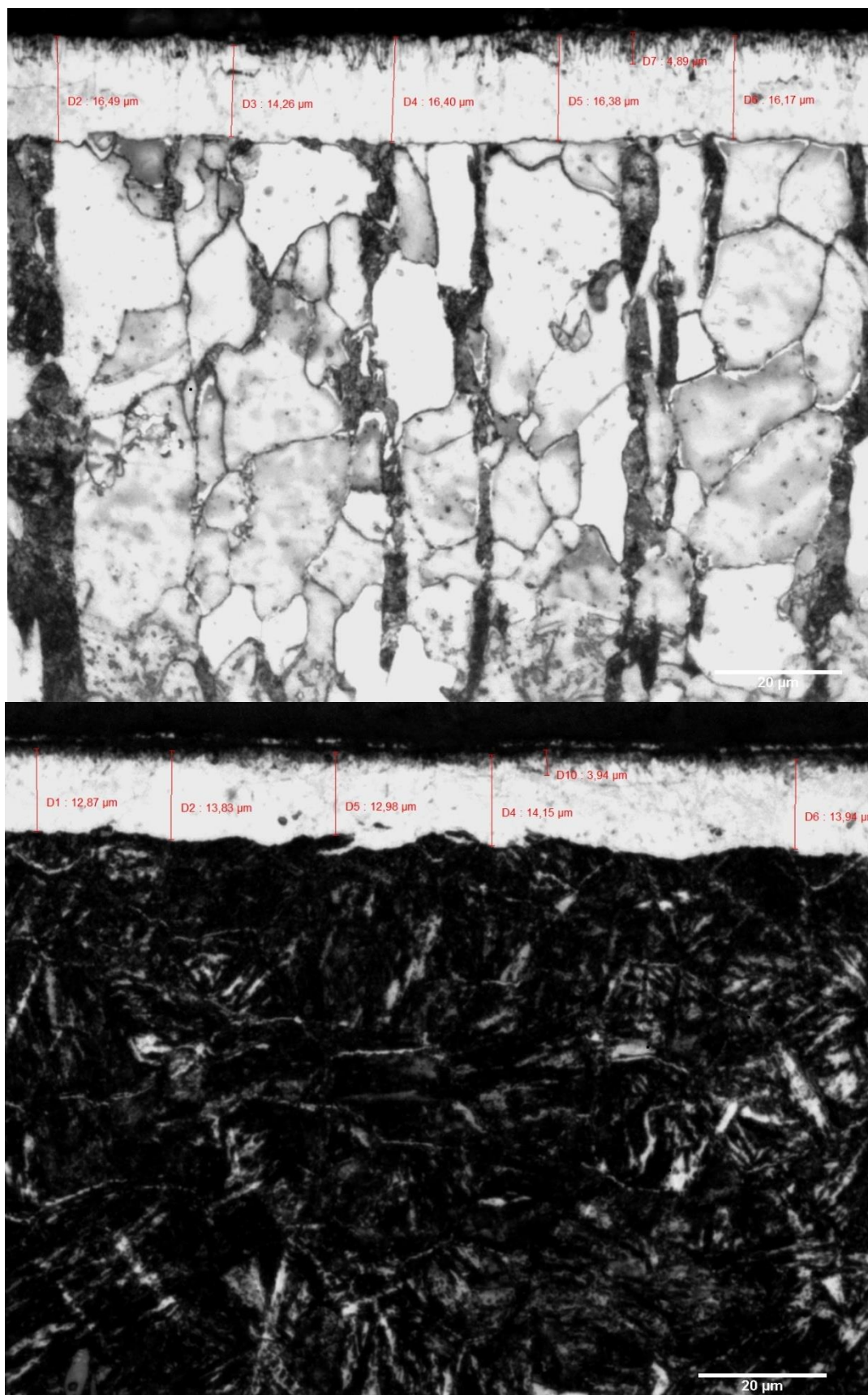


Figure - First samples from batch number 1902 - 270 (SS 2172 above and SS 2541 below)

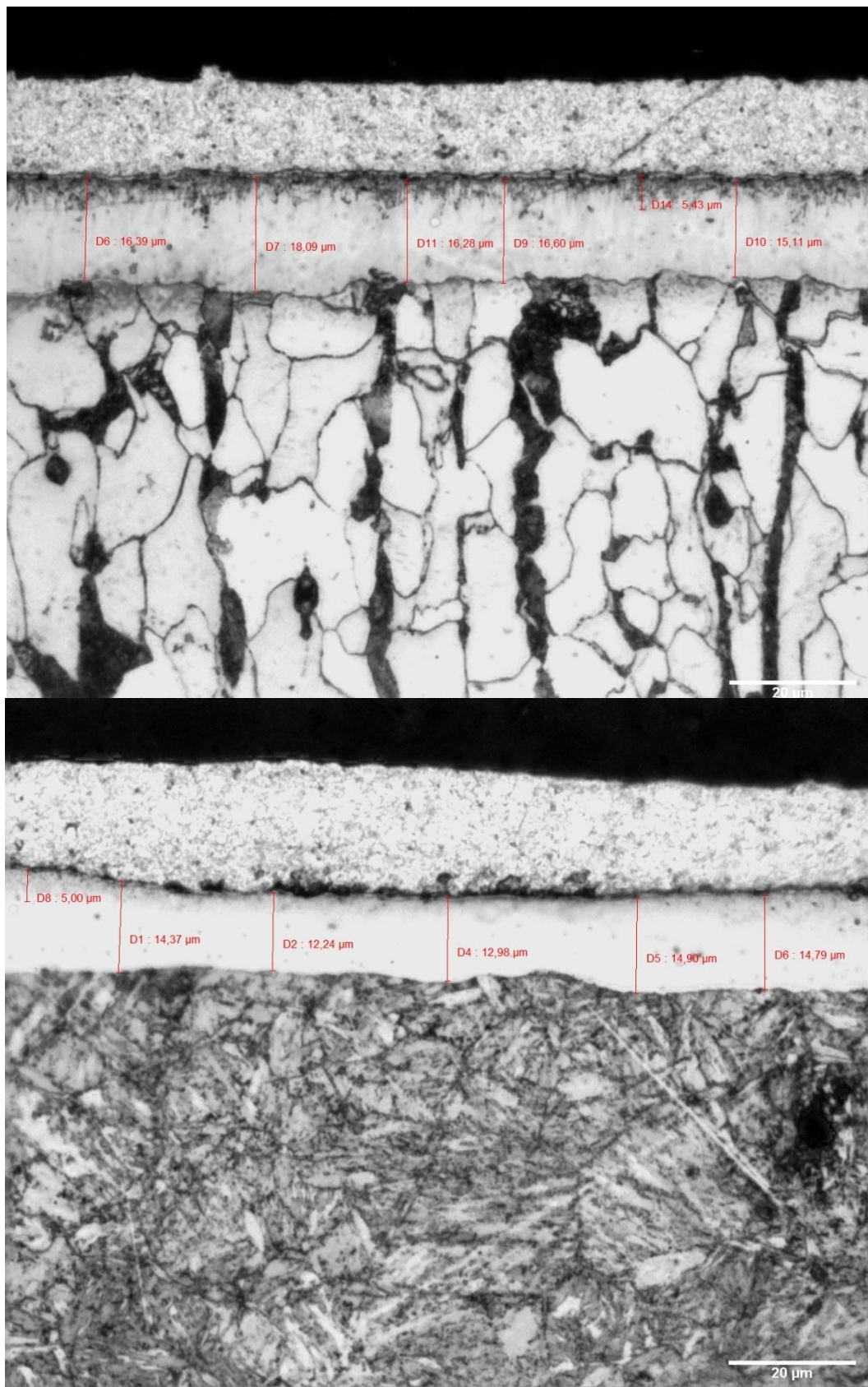


Figure - First samples from batch number 1902 - 716 (SS 2172 above and SS 2541 below)

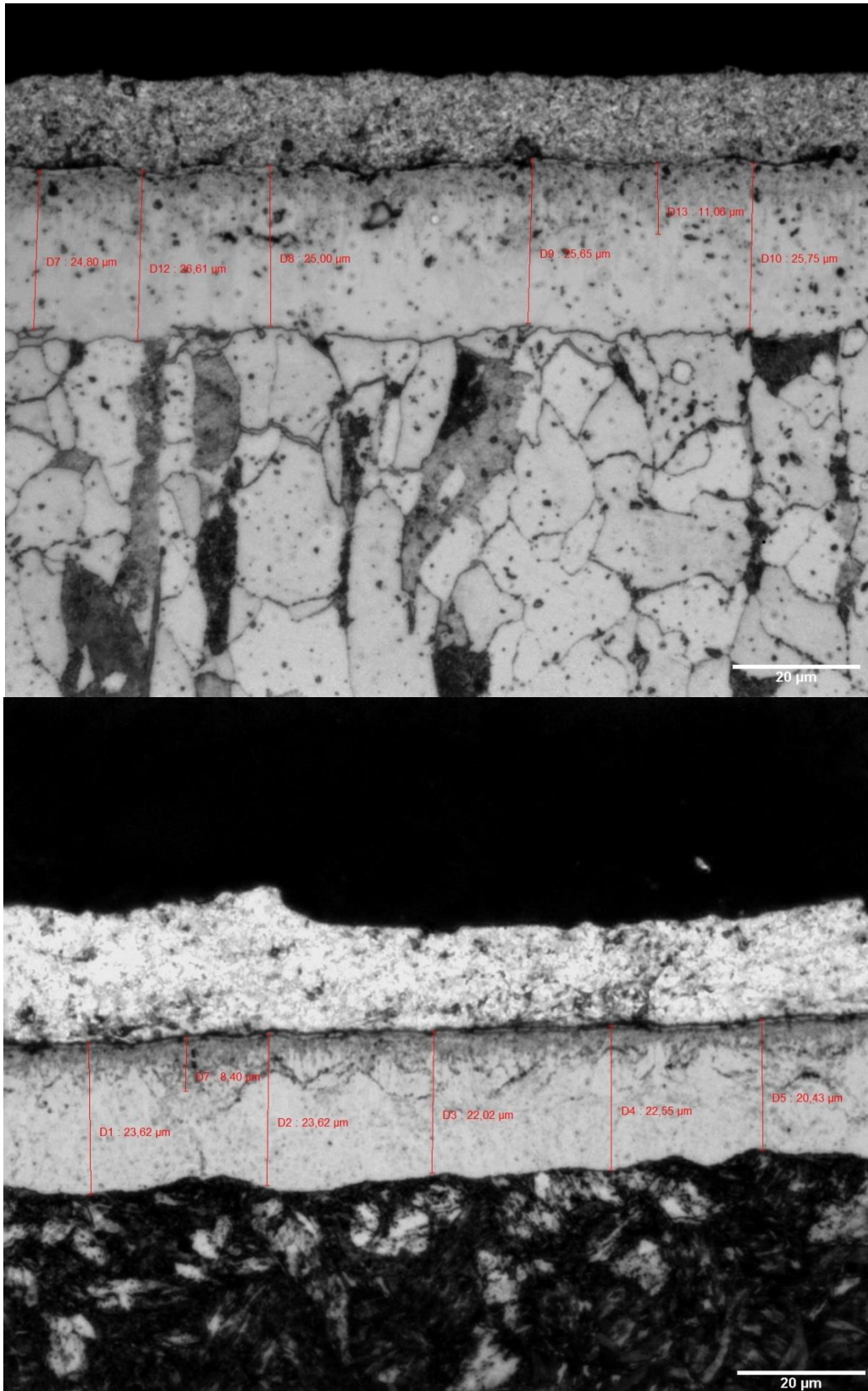


Figure - First samples from batch number 1902 - 521 (SS 2172 above and SS 2541 below)

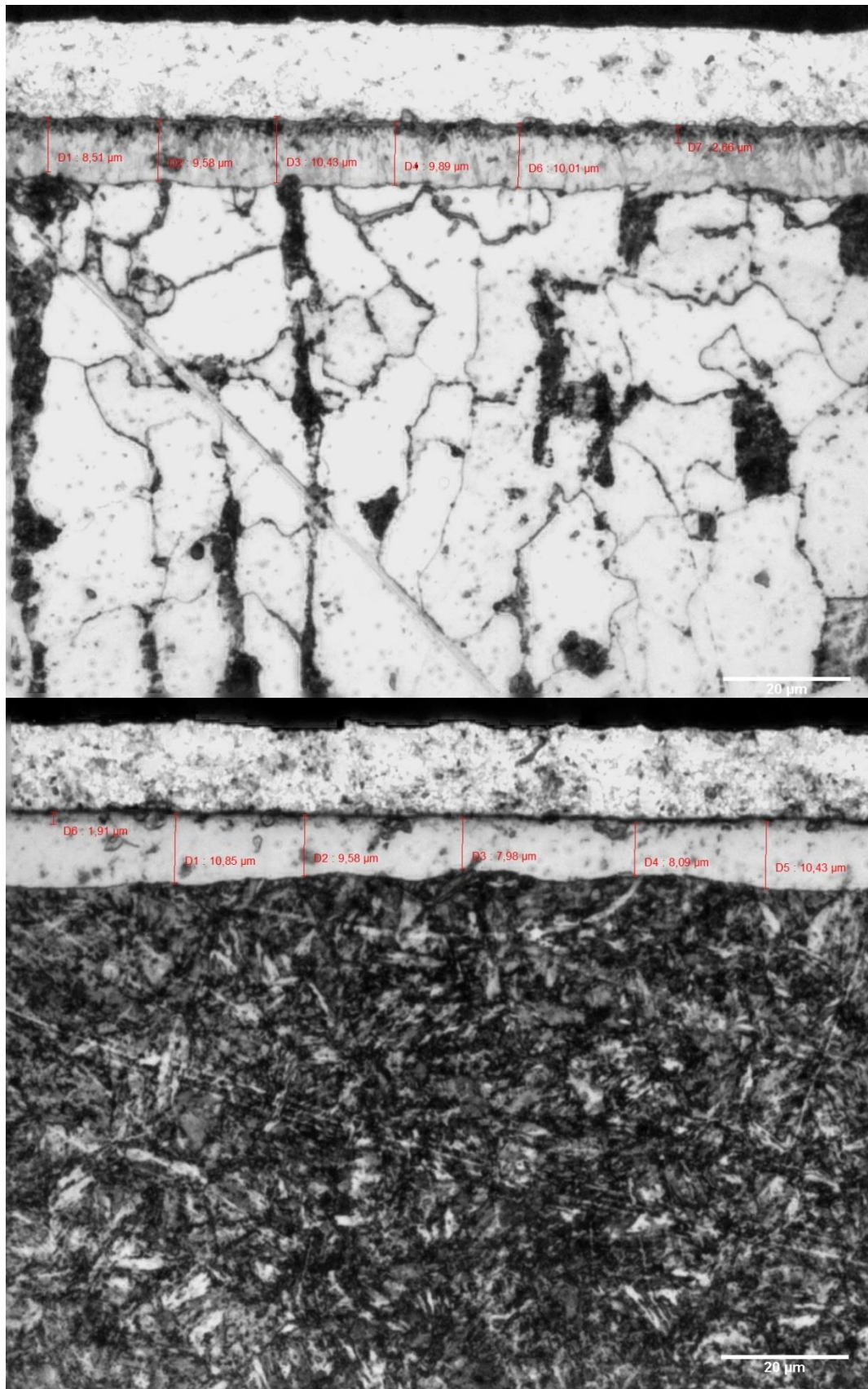


Figure - First samples from batch number 1902 - 184 (SS 2172 above and SS 2541 below)

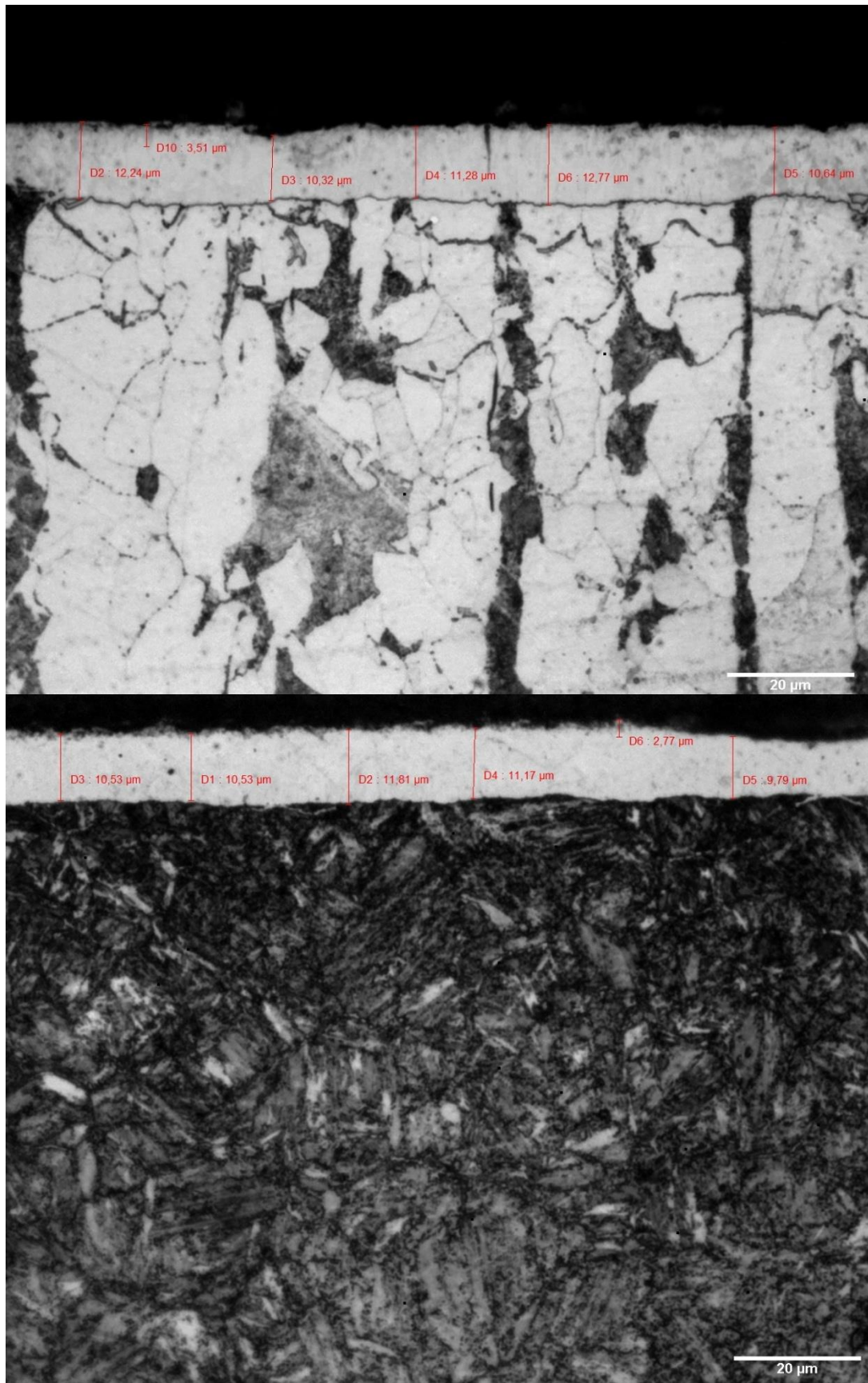


Figure - First samples from batch number 1902 - 092 (SS 2172 above and SS 2541 below)

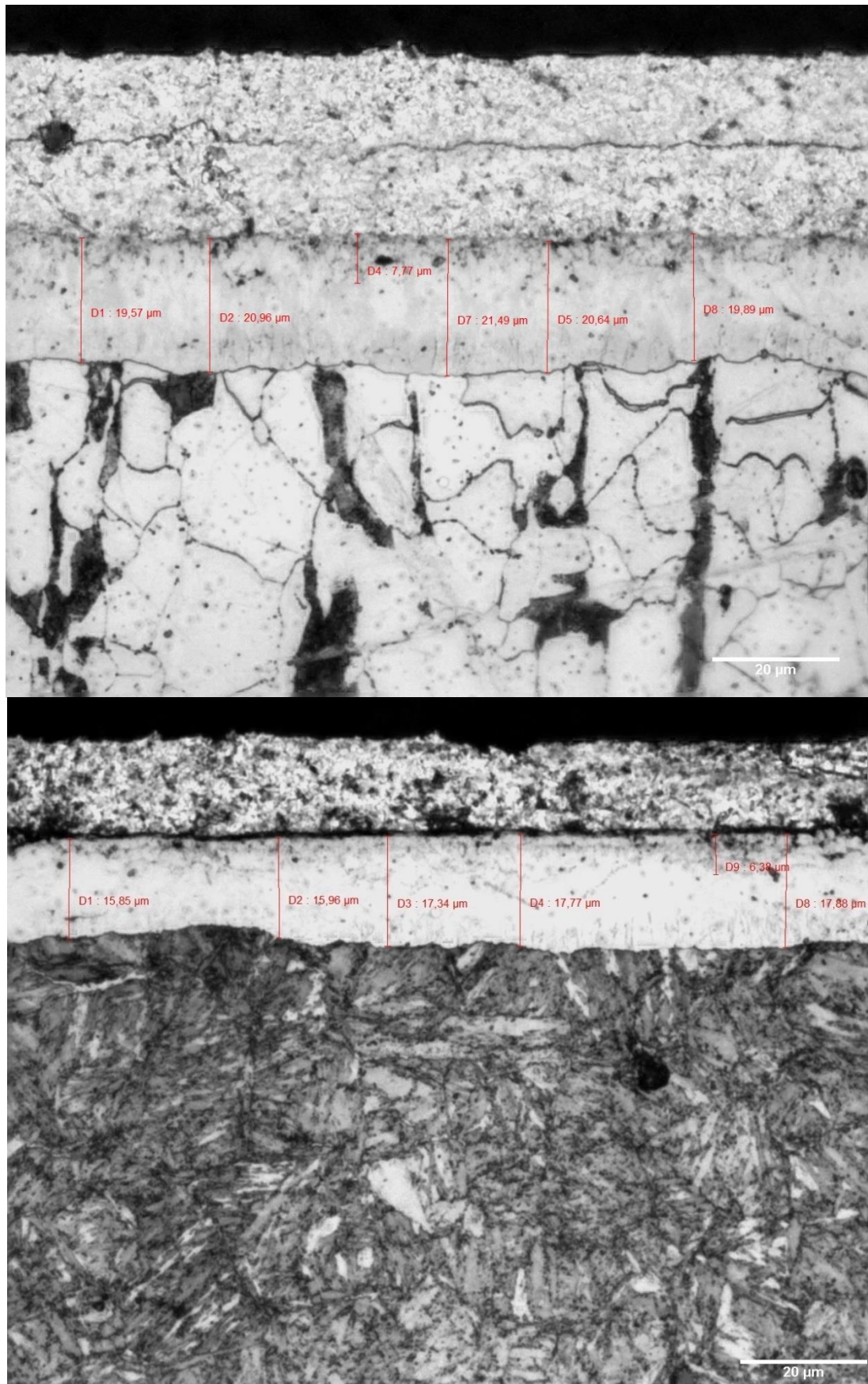


Figure - First samples from batch number 1902 - 223 (SS 2172 above and SS 2541 below)

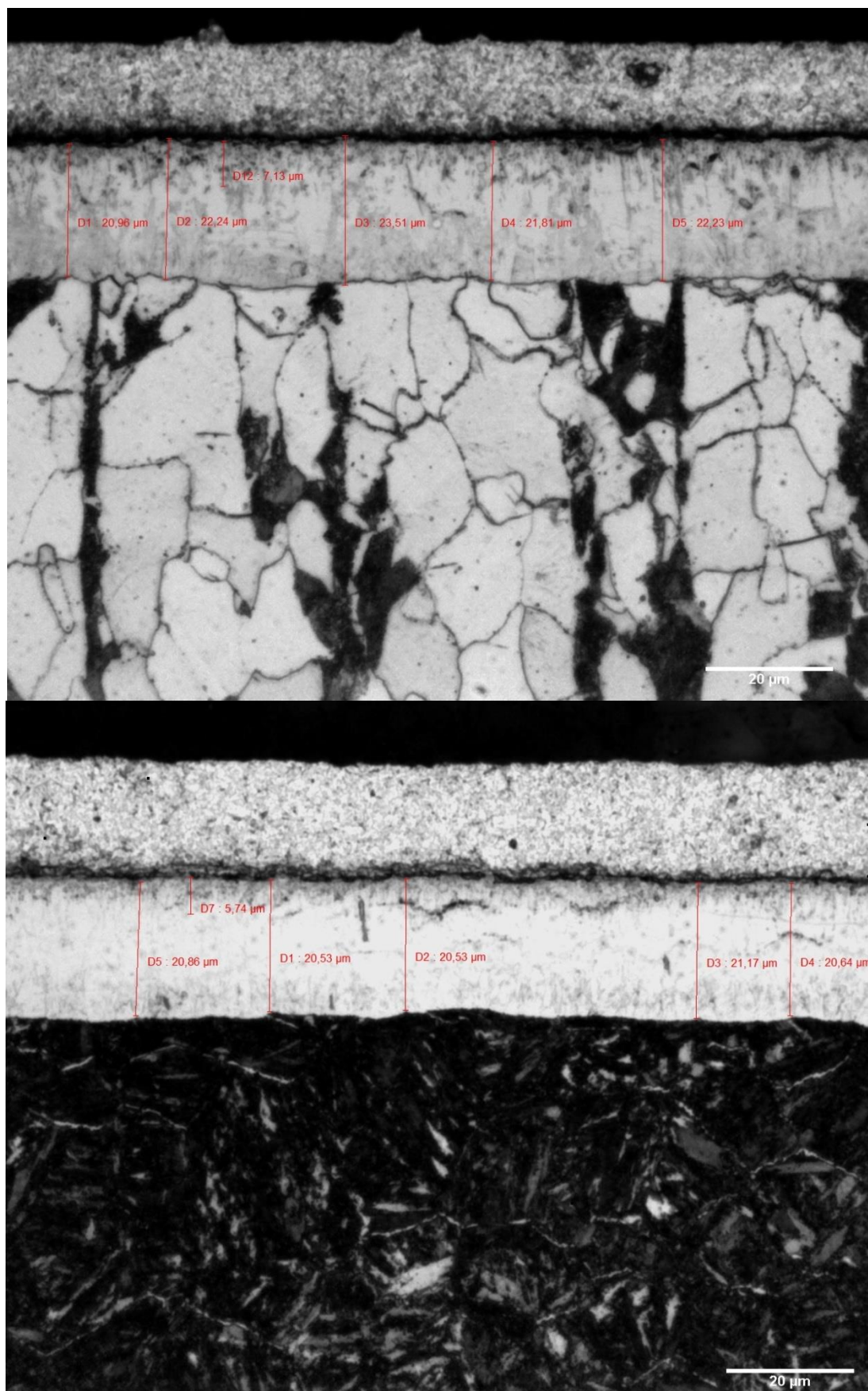


Figure - First samples from batch number 1902 - 289 (SS 2172 above and SS 2541 below)

Appendix 08 - MAGNATEST D 3.623 Numerical Results

The results are divided into four different categories based on both quenching medium and steel grade. Three of the x-values and y-values were taken in the case of each set of measurements over a specific sample (each one of the sixteen samples used).

Table - MAGNATEST results for oil-quenched SS 2172 samples

Sample Number	Measurement Number	Microscopy Values	x-values	y-values
1	1	15,15	-3658	-3174
1	2		-3653	-3175
1	3		-3657	-3181
2	1	15,94	-3606	-3066
2	2		-3615	-3065
2	3		-3624	-3064
3	1	16,49	-3658	-3021
3	2		-3658	-3016
3	3		-3643	-3026
4	1	25,56	-3576	-2769
4	2		-3554	-2764
4	3		-3564	-2767

Table - MAGNATEST results for oil-quenched SS 2541 samples

Sample Number	Measurement Number	Microscopy Values	x-values	y-values
1	1	13,34	-3881	-3306
1	2		-3853	-3306
1	3		-3853	-3306
2	1	13,55	-3851	-3250
2	2		-3849	-3251
2	3		-3848	-3253
3	1	13,86	-3817	-3230
3	2		-3868	-3245
3	3		-3856	-3242
4	1	22,45	-3863	-3124
4	2		-3820	-3130
4	3		-3844	-3129

Table - MAGNATEST results for gas-quenched SS 2172 samples

Sample Number	Measurement Number	Microscopy Values	x-values	y-values
1	1	9,68	-3256	-2878
1	2		-3250	-2878
1	3		-3255	-2878
2	1	11,45	-3257	-2744
2	2		-3266	-2743
2	3		-3255	-2767
3	1	20,51	-3187	-2478
3	2		-3169	-2485
3	3		-	-
4	1	22,15	-3176	-2441
4	2		-3179	-2438
4	3		-3165	-2450

Table - MAGNATEST results for gas-quenched SS 2541 samples

Sample Number	Measurement Number	Microscopy Values	x-values	y-values
1	1	9,39	-3445	-2945
1	2		-3427	-2944
1	3		-3418	2949
2	1	10,77	-3454	-2933
2	2		-3431	-2924
2	3		-3435	-2930
3	1	16,96	-3414	-2788
3	2		-3430	-2781
3	3		-3404	-2793
4	1	20,75	-3429	-2806
4	2		-3421	-2805
4	3		-3436	-2803

Appendix 09 - Rest of Images from the Metallographic Study

Microscopy images from samples 09;

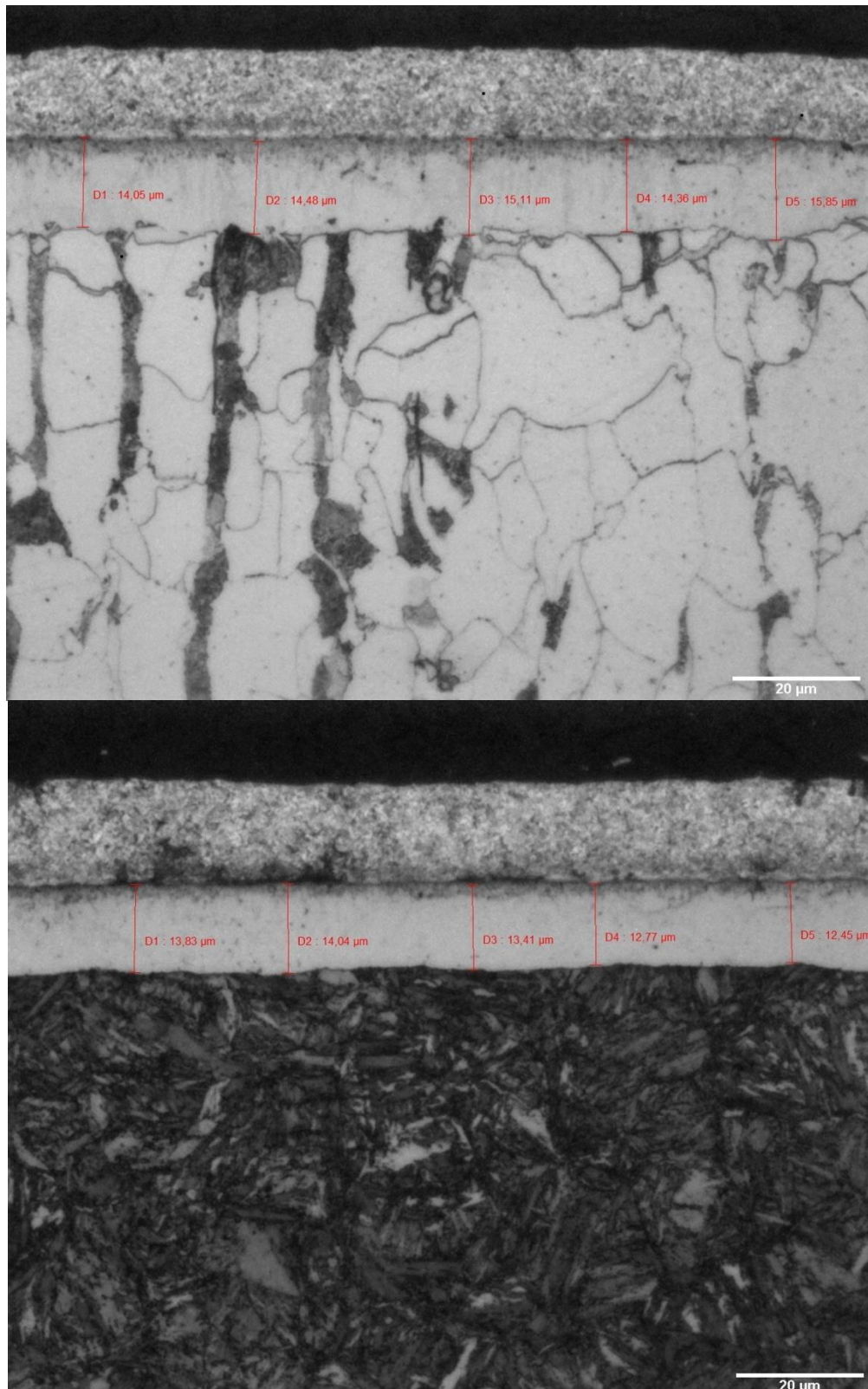


Figure - Samples 09 from batch number 1902 - 964 (SS 2172 above and SS 2541 below)

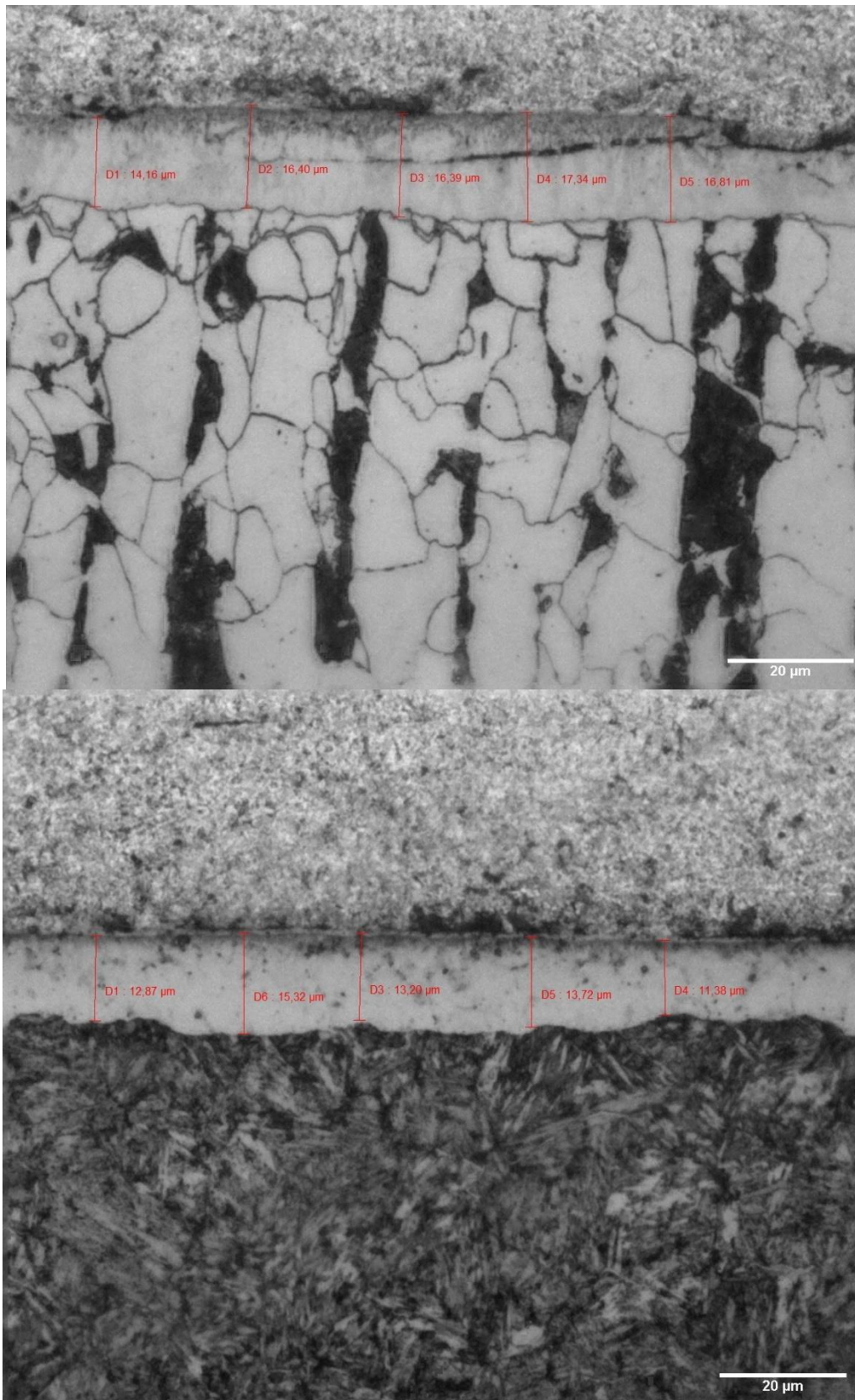


Figure - Samples 09 from batch number 1902 - 270 (SS 2172 above and SS 2541 below)

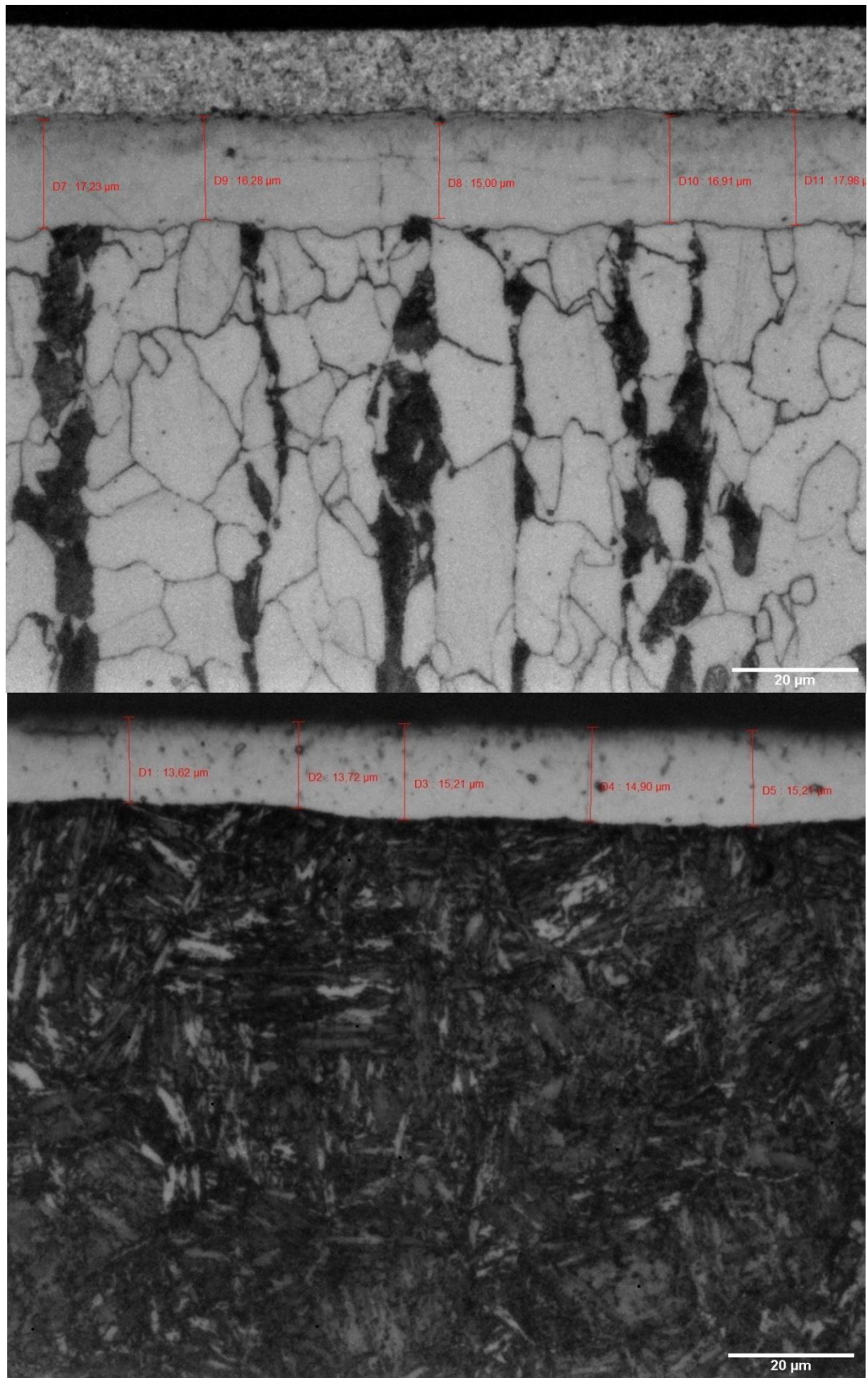


Figure - Samples 09 from batch number 1902 - 716 (SS 2172 above and SS 2541 below)

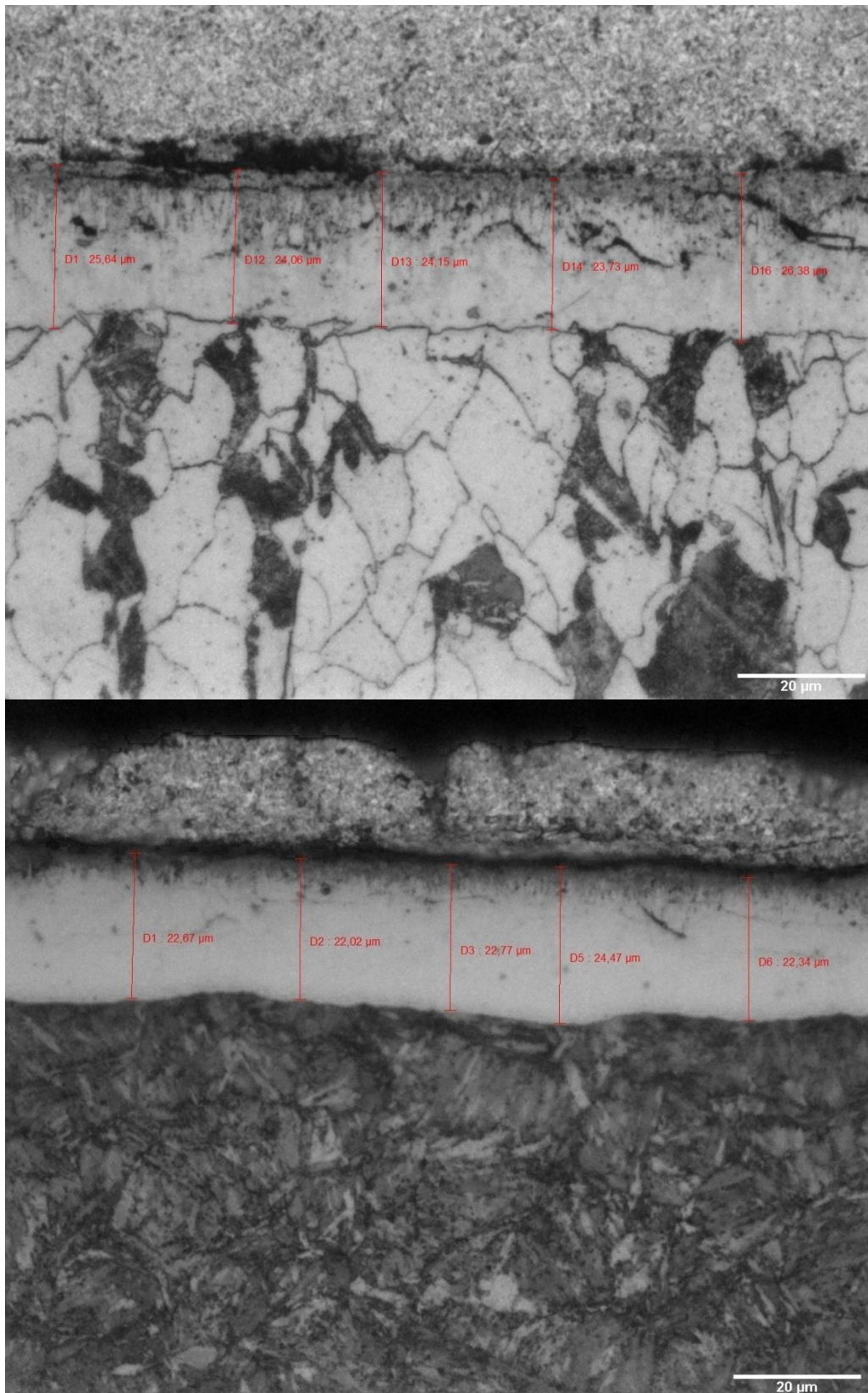


Figure - Samples 09 from batch number 1902 - 521 (SS 2172 above and SS 2541 below)

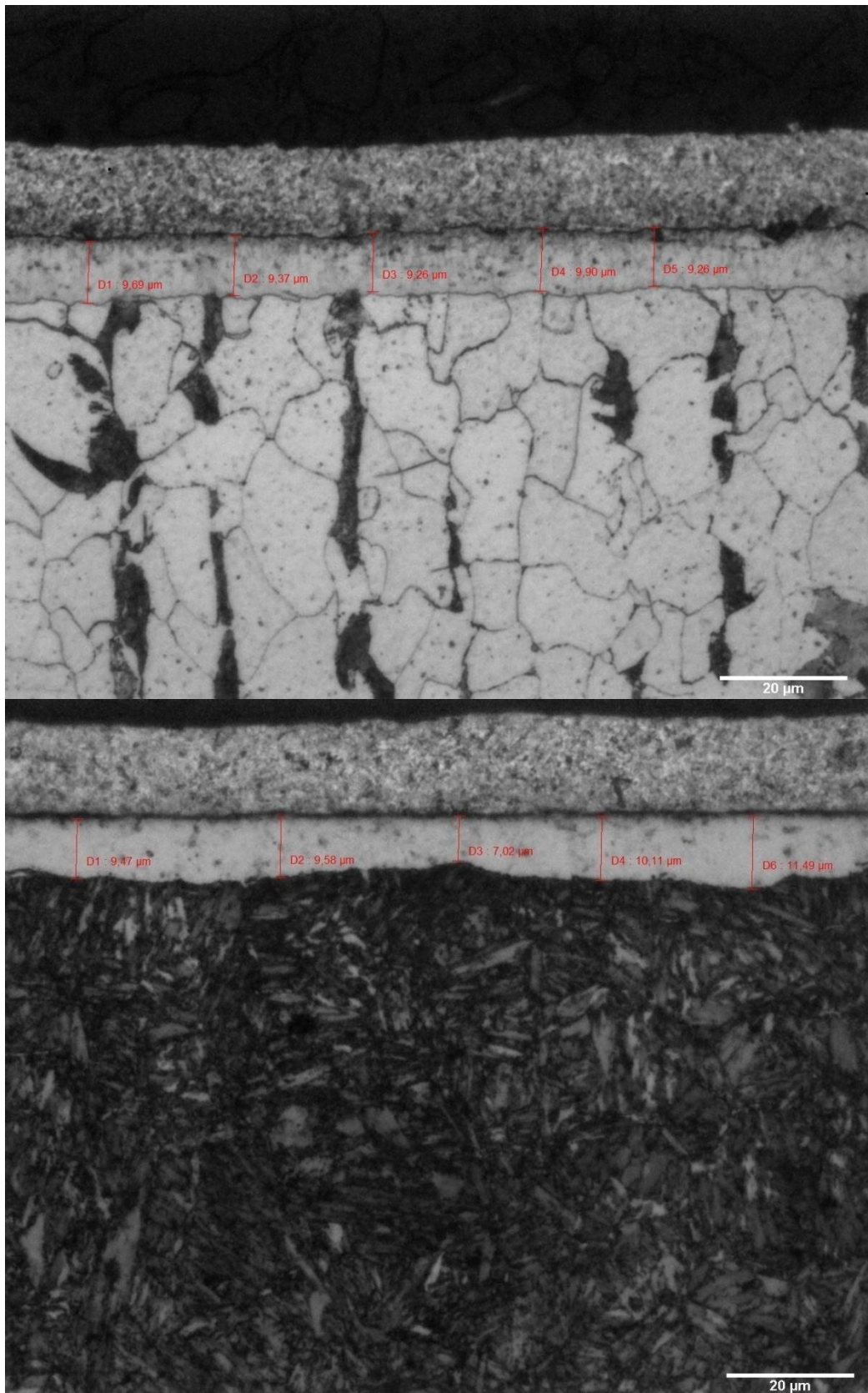


Figure - Samples 09 from batch number 1902 - 184 (SS 2172 above and SS 2541 below)

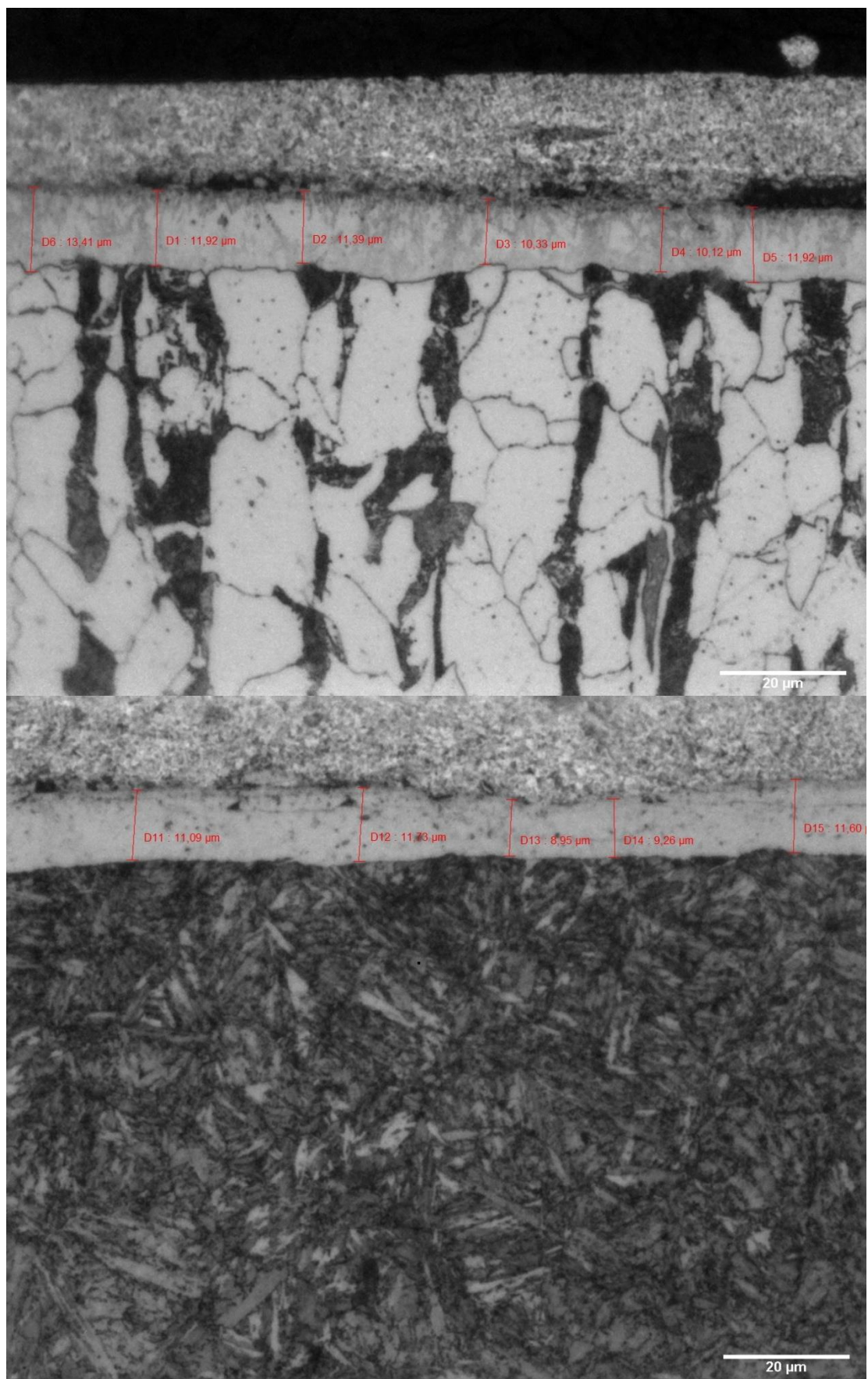


Figure - Samples 09 from batch number 1902 - 092 (SS 2172 above and SS 2541 below)

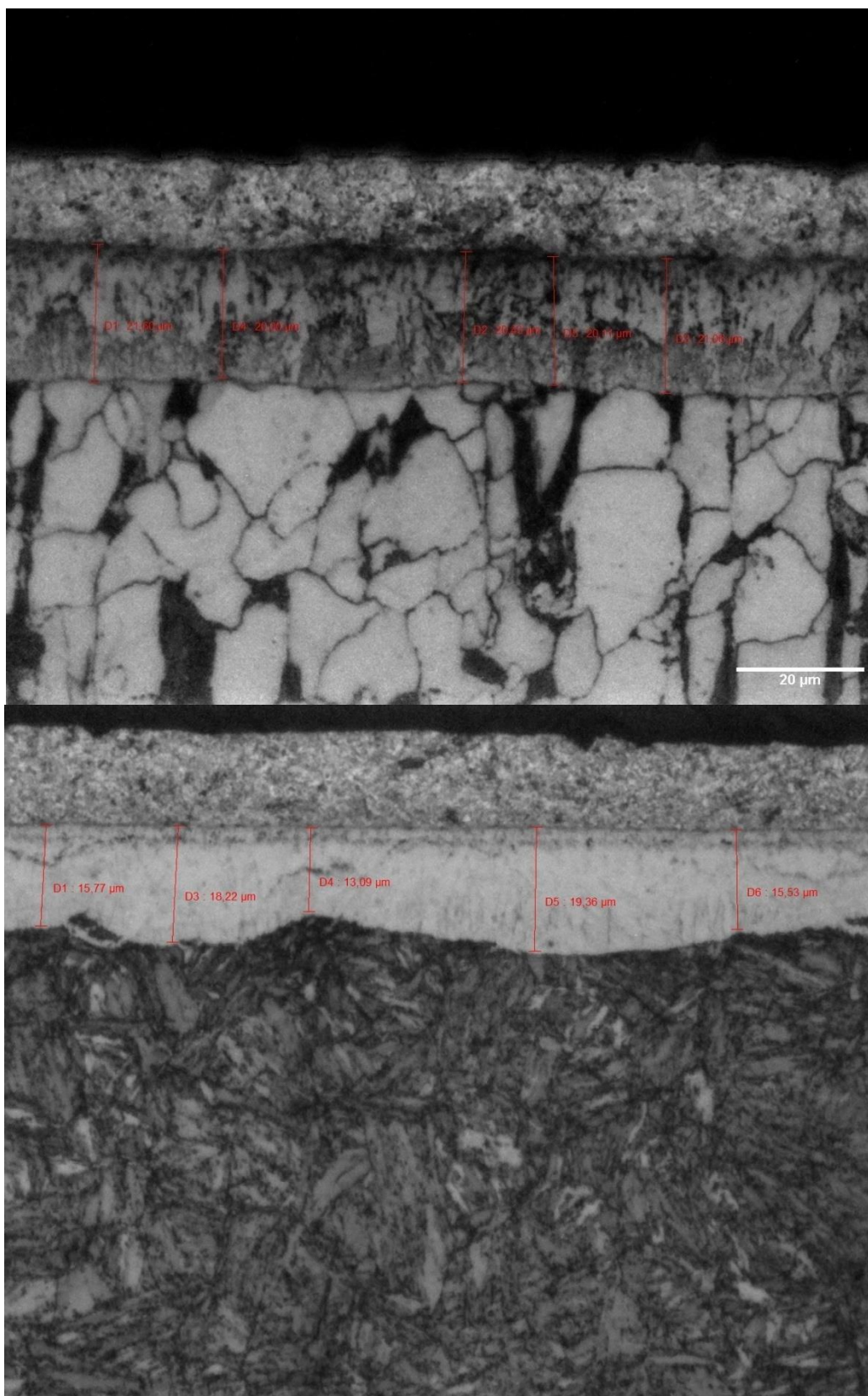


Figure - Samples 09 from batch number 1902 - 223 (SS 2172 above and SS 2541 below)

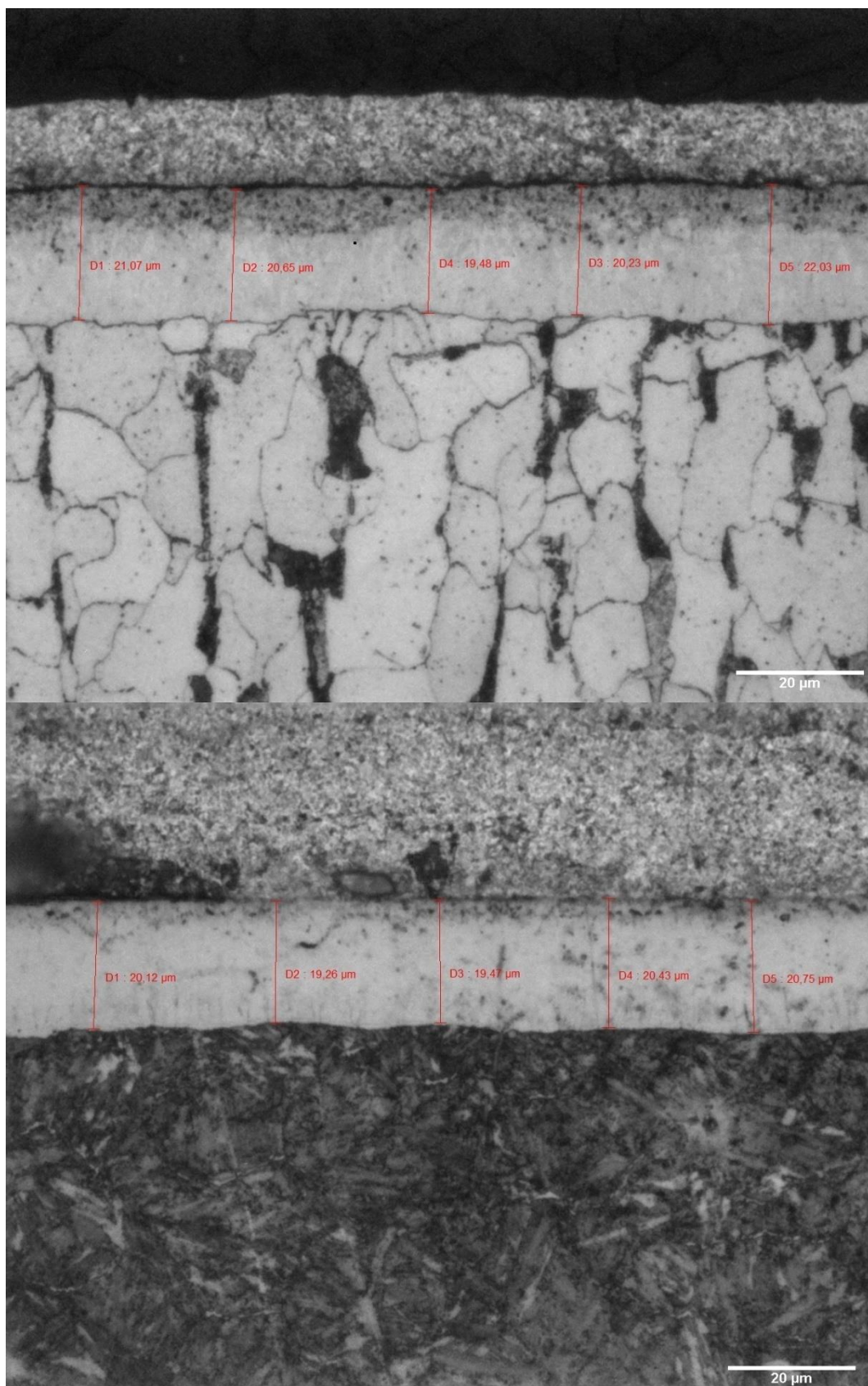


Figure - Samples 09 from batch number 1902 - 289 (SS 2172 above and SS 2541 below)

Microscopy images from samples 10;

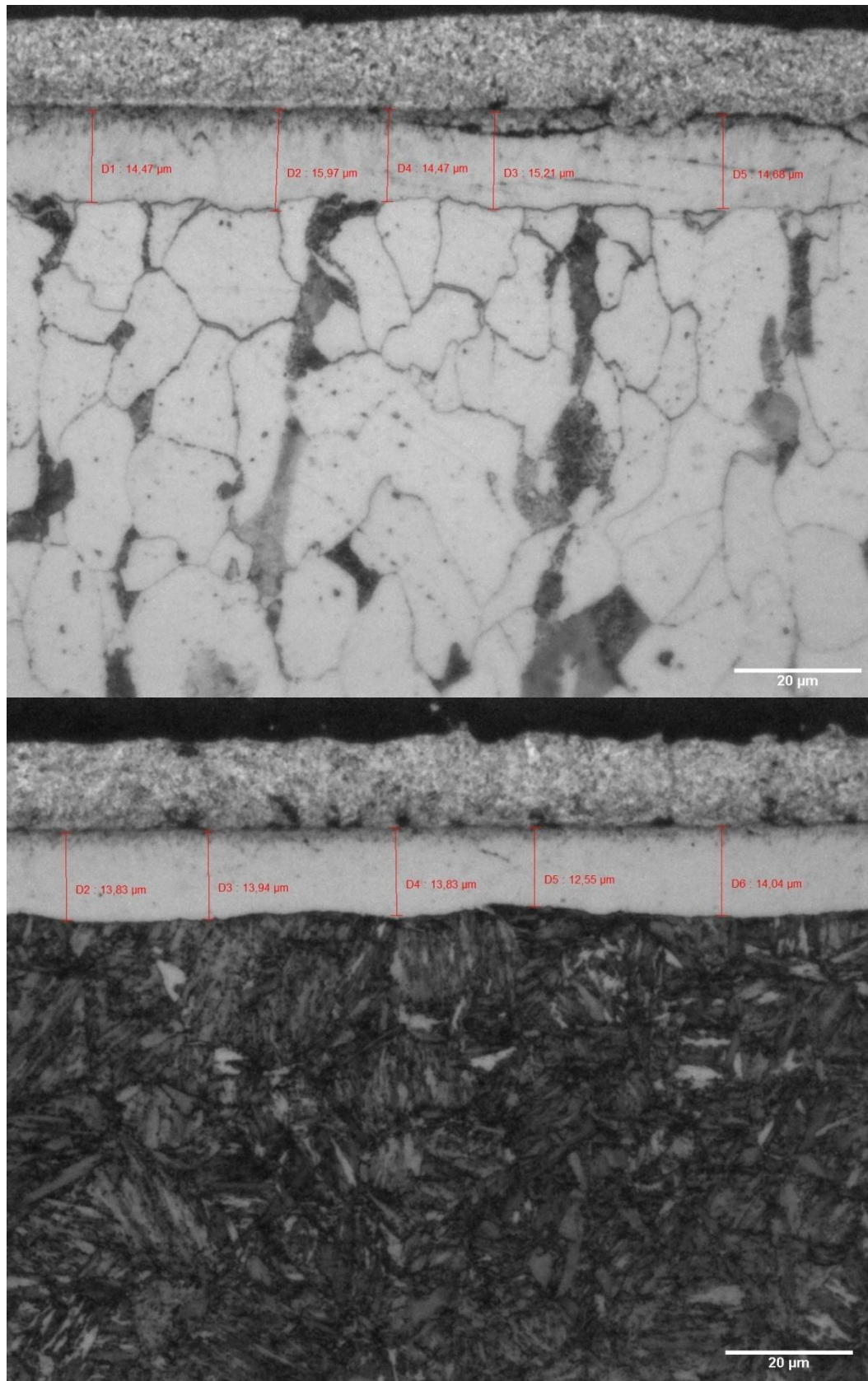


Figure - Samples 10 from batch number 1902 - 964 (SS 2172 above and SS 2541 below)

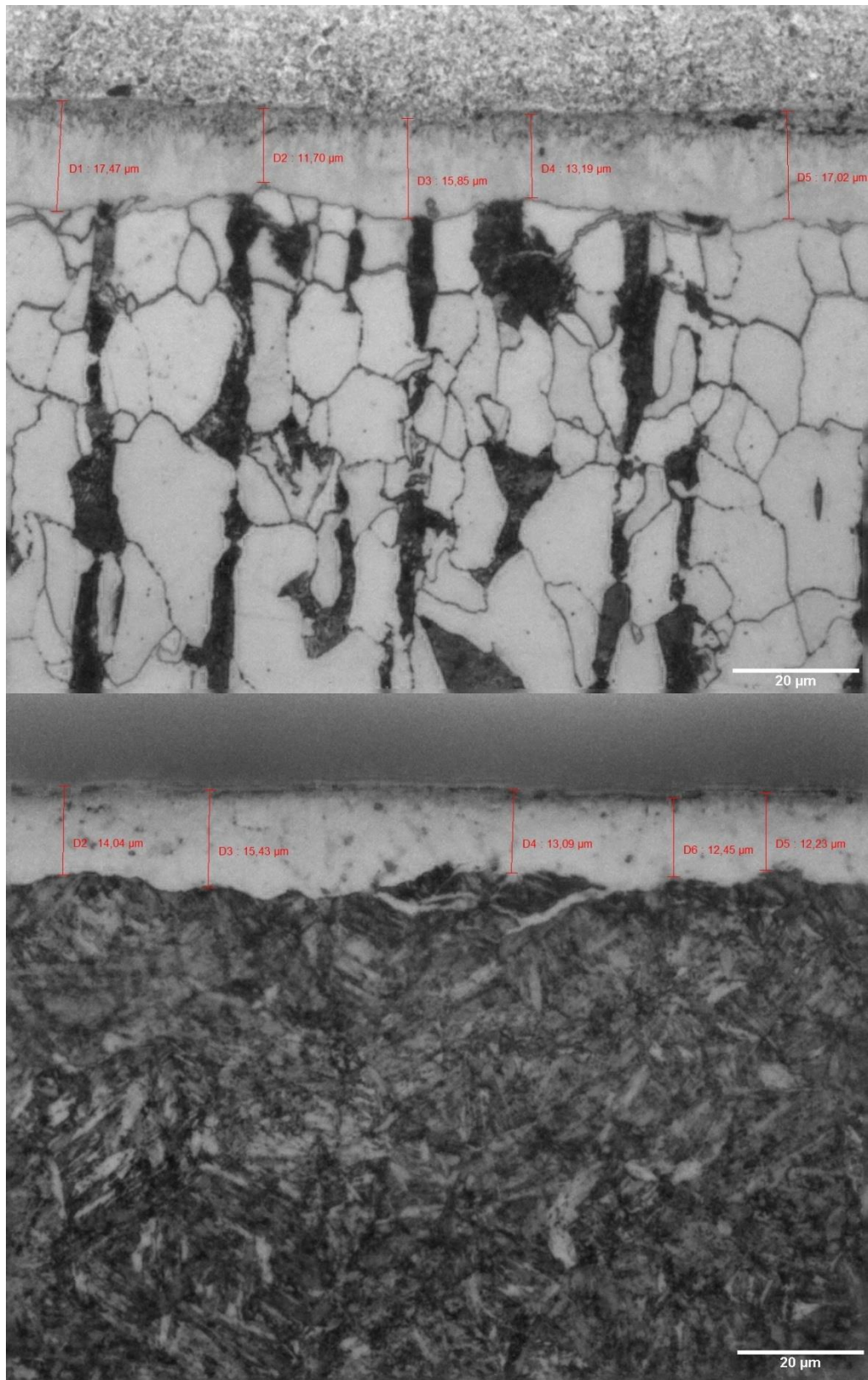


Figure - Samples 10 from batch number 1902 - 270 (SS 2172 above and SS 2541 below)

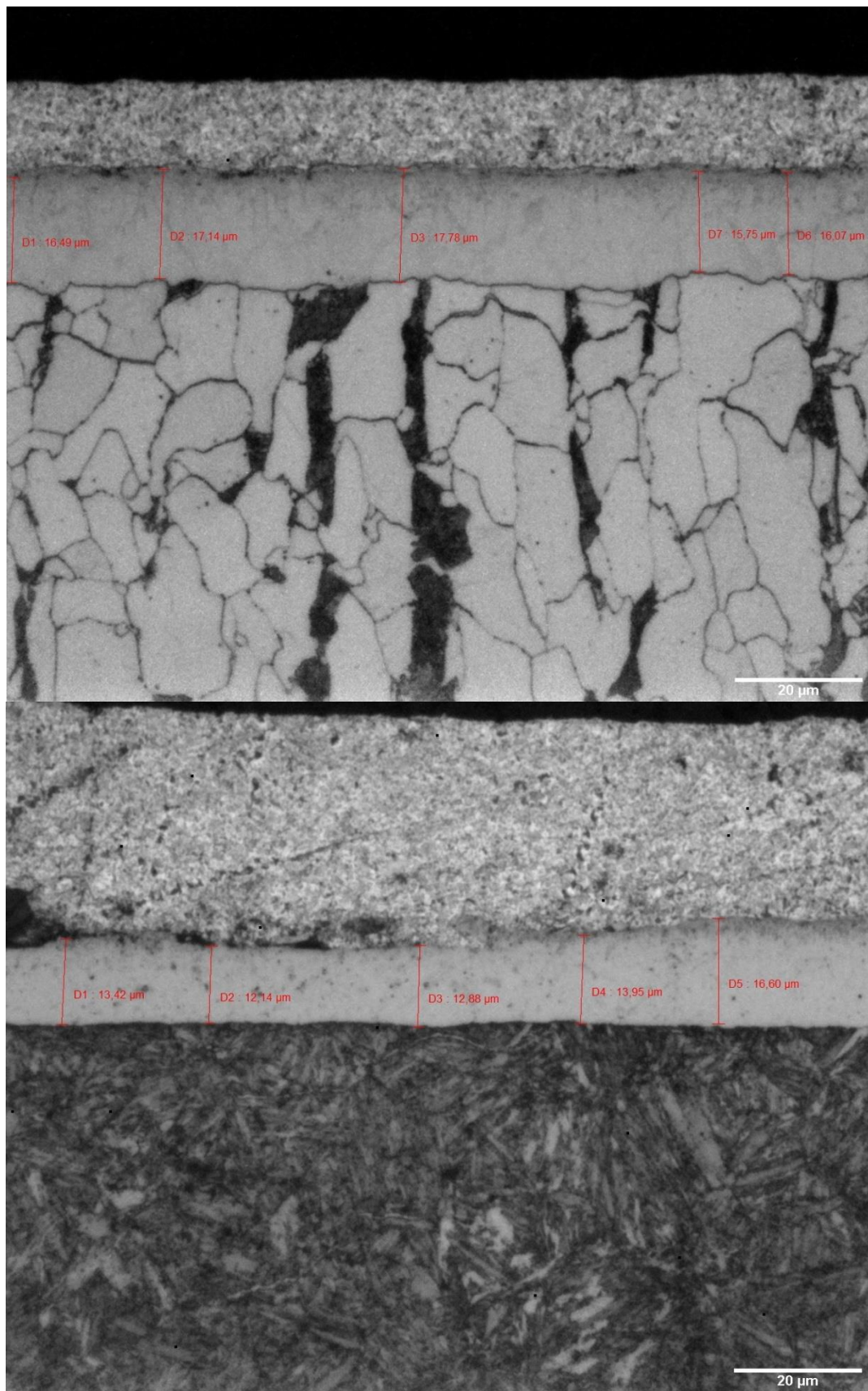


Figure - Samples 10 from batch number 1902 - 716 (SS 2172 above and SS 2541 below)

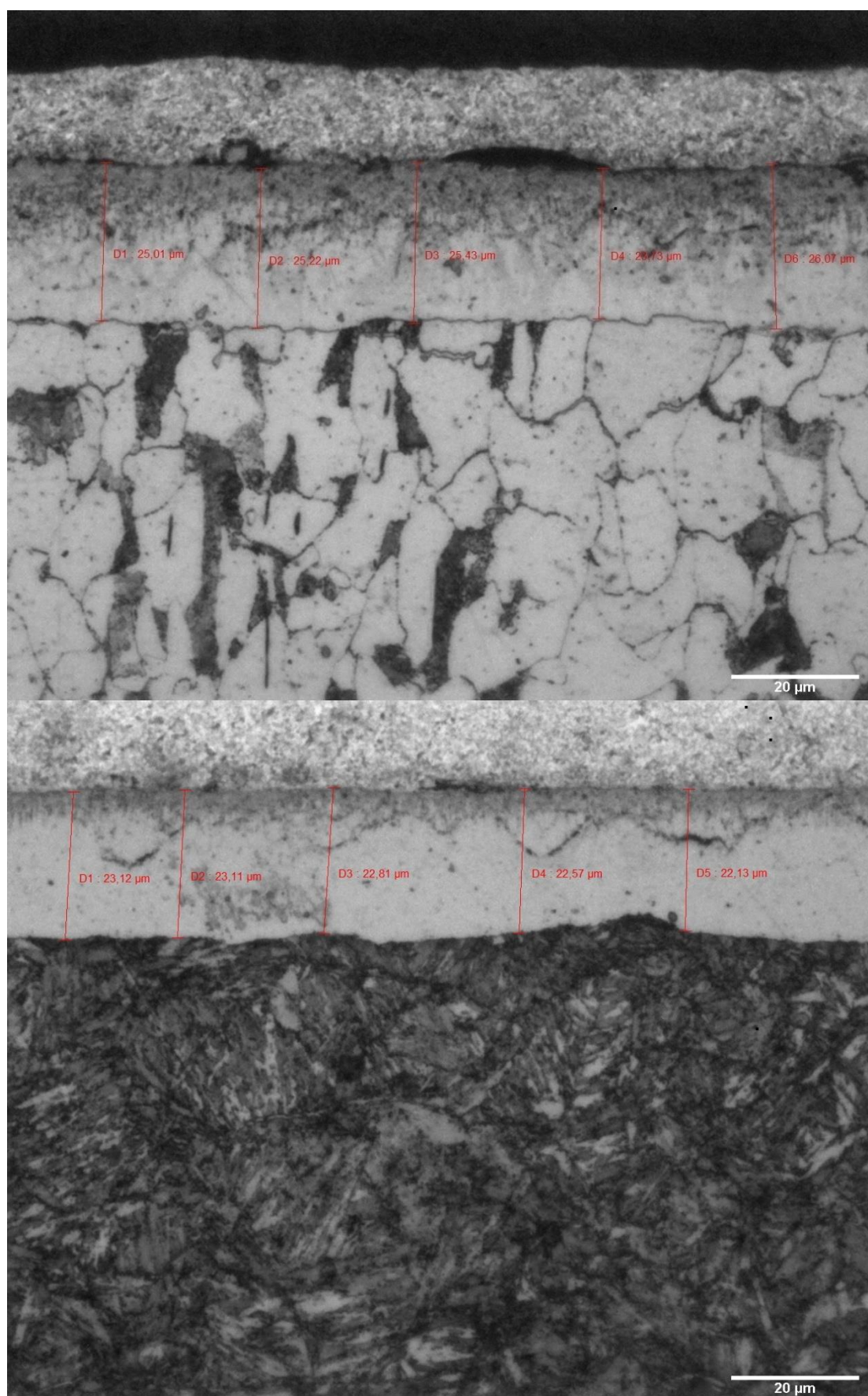


Figure - Samples 10 from batch number 1902 - 521 (SS 2172 above and SS 2541 below)

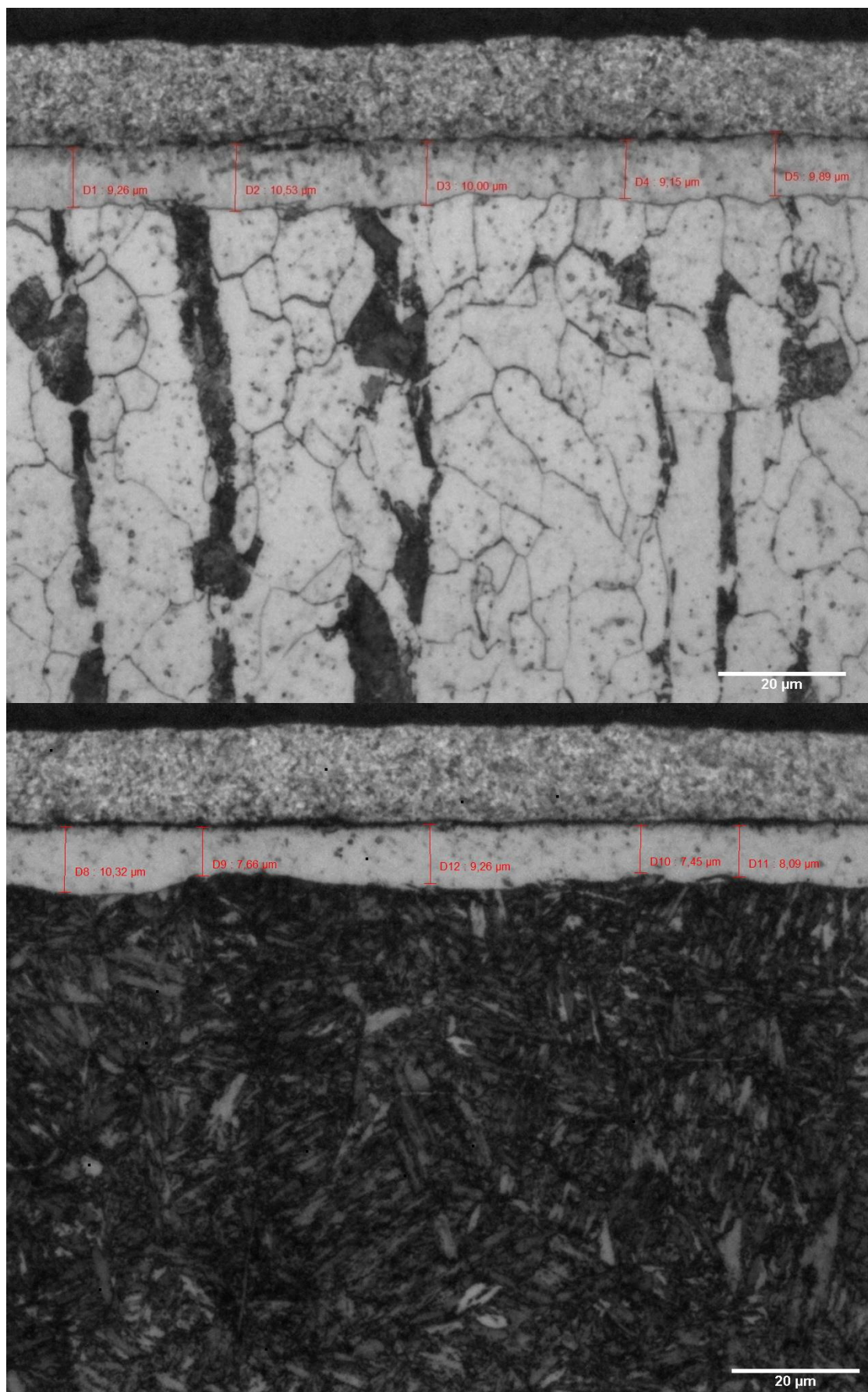


Figure - Samples 10 from batch number 1902 - 184 (SS 2172 above and SS 2541 below)

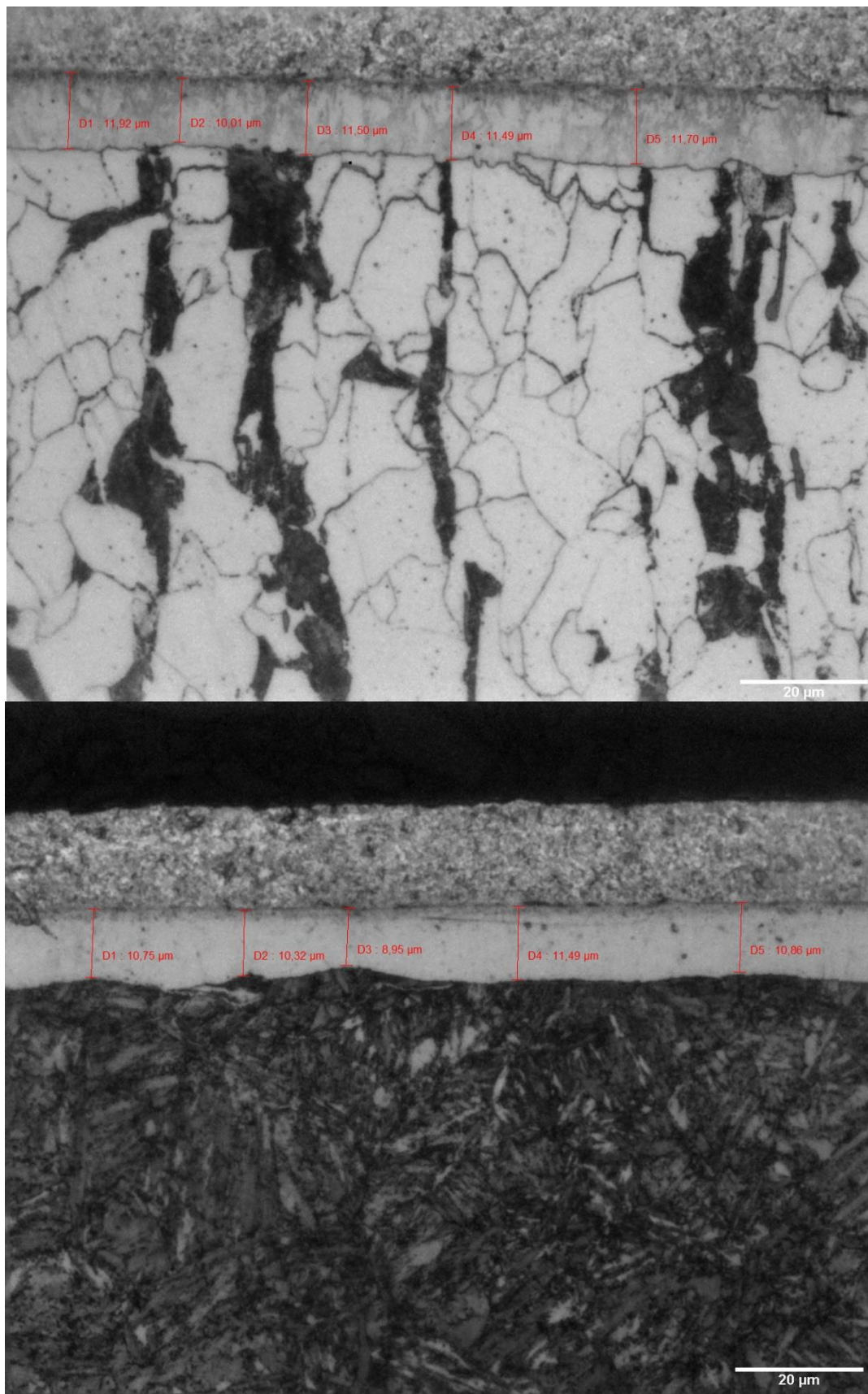


Figure - Samples 10 from batch number 1902 - 092 (SS 2172 above and SS 2541 below)

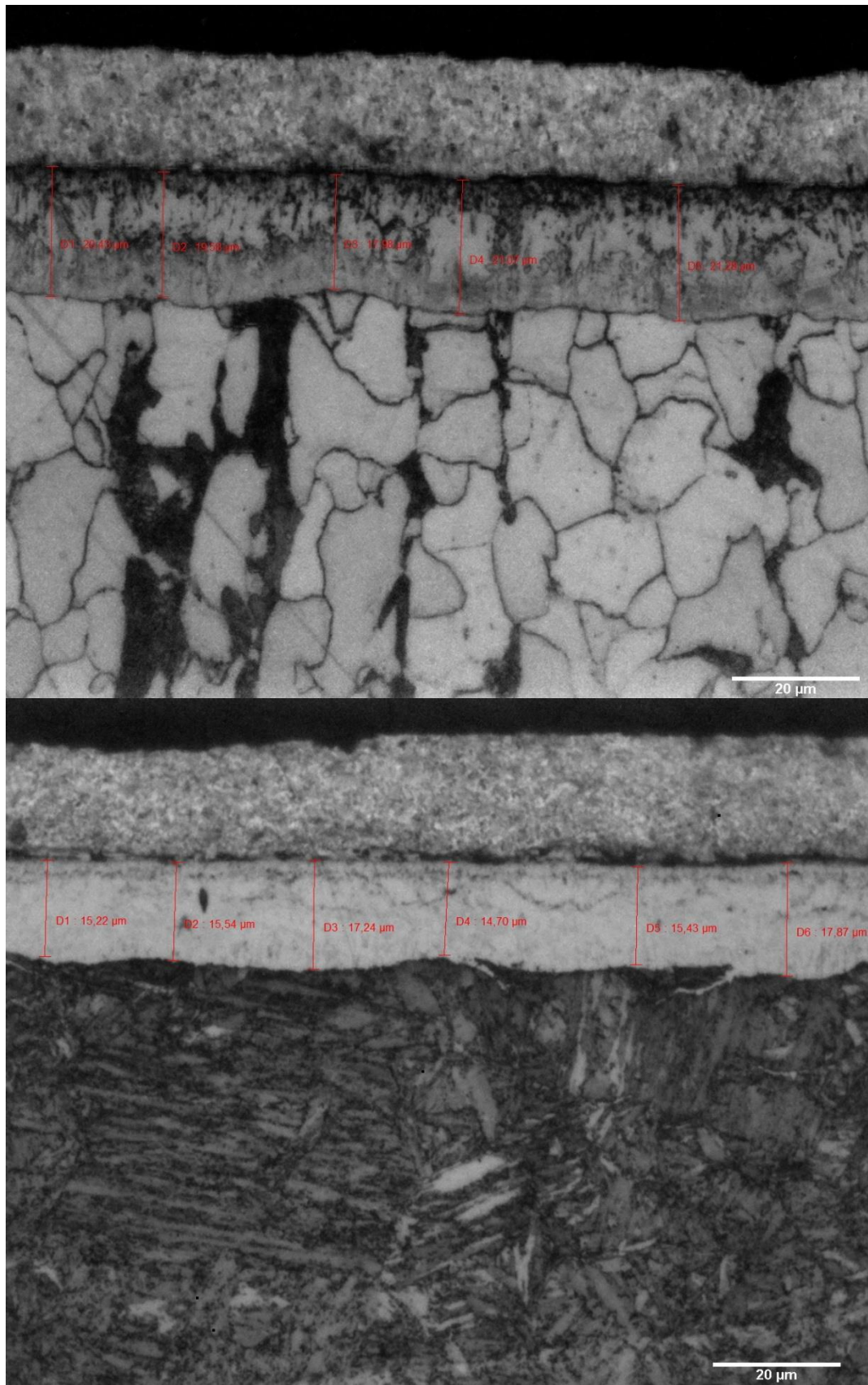


Figure - Samples 10 from batch number 1902 - 223 (SS 2172 above and SS 2541 below)

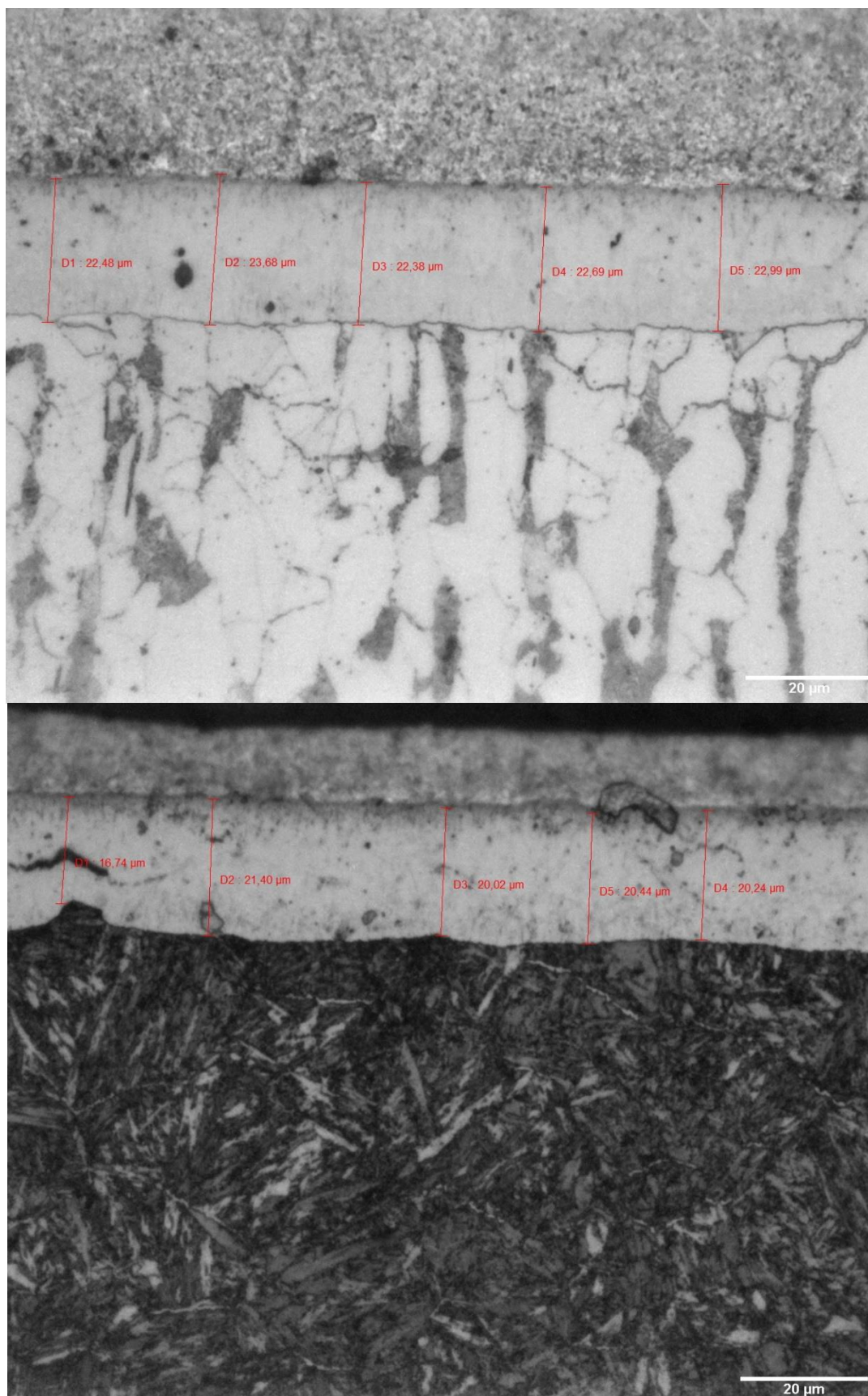


Figure - Samples 10 from batch number 1902 - 289 (SS 2172 above and SS 2541 below)

Appendix 10 - Set of Scanning Electron Microscopy Images

Additional SEM images are added in this appendix with lower and higher magnifications than the one represented by figure 26.

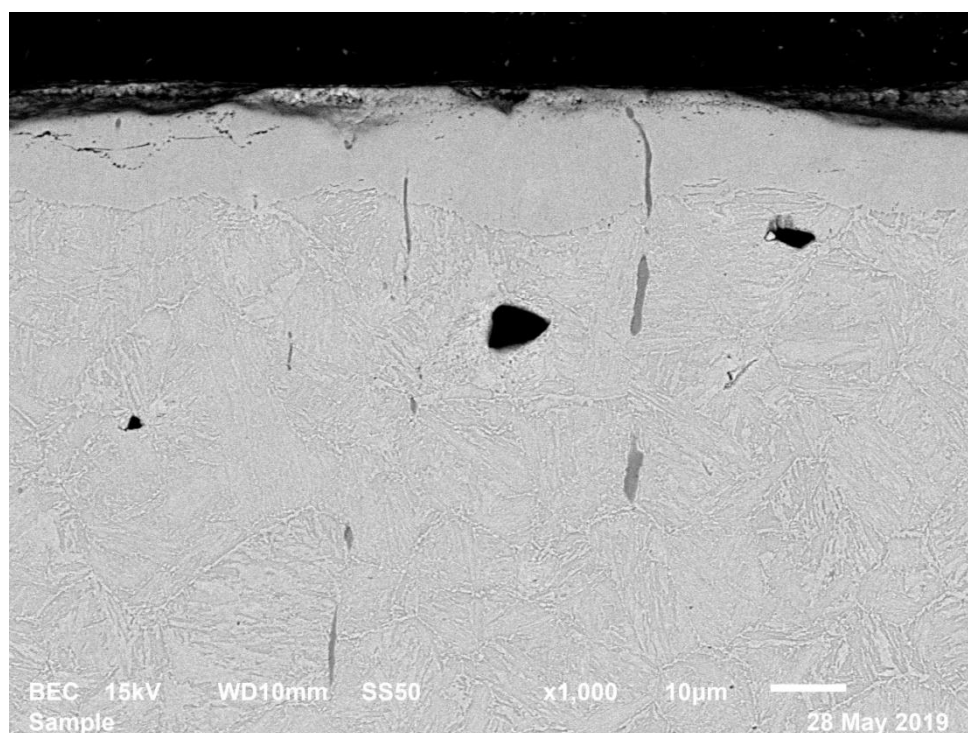


Figure - Scanning electron microscopy image of a sample from batch 1901 - 223, 1000x

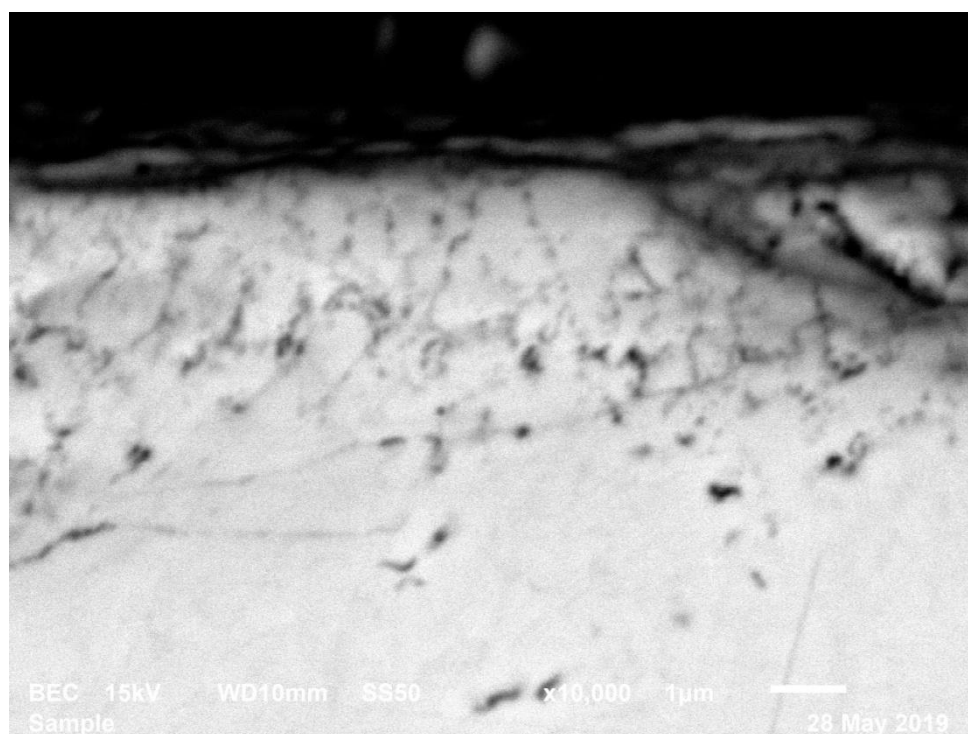


Figure - Scanning electron microscopy image of a sample from batch 1901 - 223, 10000x

Appendix 11 - Comparison of Real and Forecasted Porosity Levels

Table - Real and Forecasted Porosity Levels within oil-quenched SS 2172 samples

Compound Layer Thickness	Real Porosity Level	Forecasted Porosity Level	Difference
9,68	-	23,11%	-
11,45	-	25,37%	-
15,15	29,50%	30,09%	0,59%
15,94	30,68%	31,10%	0,42%
16,49	32,93%	31,80%	1,13%
20,51	-	36,94%	-
22,15	-	39,03%	-
25,56	43,27%	43,38%	0,11%

Table - Real and Forecasted Porosity Levels within oil-quenched SS 2541 samples

Compound Layer Thickness	Real Porosity Level	Forecasted Porosity Level	Difference
9,39	-	21,51%	-
10,77	-	23,20%	-
13,34	23,91%	26,34%	2,43%
13,55	29,08%	26,59%	2,49%
13,86	36,08%	26,97%	9,11%
16,96	-	30,76%	-
20,75	-	35,40%	-
22,45	37,42%	37,47%	0,05%

Table - Real and Forecasted Porosity Levels within gas-quenched SS 2172 samples

Compound Layer Thickness	Real Porosity Level	Forecasted Porosity Level	Difference
9,68	27,48%	28,70%	1,22%
11,45	30,66%	29,23%	1,43%
15,15	-	30,32%	-
15,94	-	30,56%	-
16,49	-	30,72%	-
20,51	37,88%	31,91%	5,97%
22,15	32,19%	32,39%	0,20%
25,56	-	33,40%	-

Table - Real and Forecasted Porosity Levels within gas-quenched SS 2541 samples

Compound Layer Thickness	Real Porosity Level	Forecasted Porosity Level	Difference
9,39	20,34%	22,55%	2,21%
10,77	25,72%	23,21%	2,51%
13,34	-	24,43%	-
13,55	-	24,53%	-
13,86	-	24,68%	-
16,96	37,62%	26,16%	11,46%
20,75	27,66%	27,97%	0,31%
22,45	-	28,78%	-