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Effect of Etchants on Superalloy HIP Oxide Scales

A study on the effect of different pickling chemicals

Master's Thesis in Material Chemistry

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CHALMERS UNIVERSITY OF TECHNOLOGY
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Cover: Scanning electron microscope image of the cross section of an LPBF Alloy 718 sample that has been hot isostatically pressed and then pickled in HCl. Magnified 20,000x.

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Abstract

In the aviation industry producers are met with high standards for material specifications. This has led to the development of new techniques to manufacture parts for airplane engines. One such method is additive manufacturing. Due to the unfavorable microstructure after additive manufacturing the parts have to be post-processed in multiple steps to reach the final structures. These include hot isostatic pressing and an etching process. When manufacturing parts in nickel-based superalloy Inconel 718 oxides can form during hot isostatic pressing. These oxides need to be removed from the part to meet standards. This is done in a two-step etching process, pickling and etching.

In this project different pickling solutions are evaluated for their ability to remove the oxide scales formed during hot isostatic pressing of the nickel-based superalloy Inconel 718. Additionally the chemical composition of the oxides and bulk material is determined. The pickling solutions used were HCl, HF mixed with HNO₃ and Metex M-629 used with electrolysis. Analysis of the oxides was made using an optical microscope, laser microscope, a scanning electron microscope and energy-dispersive x-ray spectroscopy.

The results show that no matter the pickling solution the etching solution removes all of the surface oxides. Among the pickling solutions, HF/HNO₃ had the strongest effect on the oxide scale during the pickling step while the HCl solution does very little to the oxides. The bulk material is found to be mostly nickel mixed with chromium and iron. There are two different kinds of oxides found on the surface. The first one is mostly aluminum oxides with a little titanium oxide while the other one is aluminum and titanium oxides with a niobium rich phase.

Keywords: Additive Manufacturing, Hot Isostatic Pressing, Etching, LPBF Alloy 718, oxide scales, HCl, HF/HNO₃, Metex M-629, SEM-EDS

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Gustav Ekman, Trollhättan, May 2026

List of Acronyms

Below is the list of acronyms that have been used throughout this thesis, listed in alphabetical order:

AM	Additive Manufacturing
EDS	Energy Dispersive X-ray Spectroscopy
HIP	Hot Isostatic Pressing
LPBF	Laser Powder Bed Fusion
SEM	Scanning Electron Microscopy

Contents

List of Acronyms	ix
List of Figures	xiii
List of Tables	xvii
1 Introduction	1
1.1 Background	1
1.2 Aim	2
1.3 Limitations	2
2 Theory	3
2.1 Manufacturing process	3
2.1.1 Additive Manufacturing (AM)	3
2.1.1.1 Laser Powder Bed Fusion (LPBF)	3
2.1.2 Hot Isostatic Pressing (HIP)	4
2.1.3 Etching	4
2.2 LPBF Alloy 718	6
2.2.1 Secondary Phases Formed in LPBF Alloy 718	6
2.3 Analytical Methods	7
2.3.1 Laser Microscope	7
2.3.1.1 Surface Roughness	7
2.3.2 Scanning Electron Microscopy (SEM)	8
2.3.2.1 Energy-Dispersive X-ray Spectroscopy (EDS)	10
3 Material and Methods	11
3.1 Literature study	11
3.2 Material	11
3.3 Etching Method	12
3.4 Analyzing Methods	13
3.4.1 Sample Preparation	14
	xi

4	Results and Discussion	15
4.1	Analysis in Optical Microscope	16
4.2	Analysis in Scanning Electron Microscope	18
4.3	Chemical Analysis	20
4.4	Surface Roughness	27
5	Conclusion	31
5.1	Future Work	31
	References	33
A	Hot Isostatic Pressing Chamber	I
B	Laser Microscope Images	III
C	Sample Treatment	VII
D	Optical Microscope Images	IX
E	Energy Dispersive X-ray Spectroscopy	XIII
F	Surface Roughness	XIX
G	Societal, Ethical and Ecological Aspects	XXI

List of Figures

1.1	Flow chart of the process for manufacturing a part using additive manufacturing, hot isostatic pressing and etching.	2
2.1	Flow chart of the additive manufacturing process laser powder bed fusion.	4
2.2	Schematic representation of a scanning electron microscope [27].	9
2.3	Schematic image showing the interaction volume and where the different signal originates from in a sample analyzed in SEM.	9
3.1	Laser microscope images of the front side of a typical sample a) before HIP and b) After HIP. In b) the arrows show the colored and the black oxide regions.	12
4.1	Laser microscope images of samples pickled in a-b) HCl, c-d) HF/HNO ₃ and e-f) Metex M-629. The left side of the sample has been pickled and the right side has both been pickled and etched.	15
4.2	Optical microscope images of the cross section at the a) colored oxide and b) black oxide for the reference sample that have only been HIP-treated. .	16
4.3	Optical microscope images of the cross sections at the colored oxide regions (top) and black oxide regions (bottom) for samples pickled in a) HCl, b) HF/HNO ₃ and c) Metex M-629.	17
4.4	Optical microscope images of the cross sections at the colored oxide regions (top) and black oxide regions (bottom) for samples pickled in a) HCl, b) HF/HNO ₃ and c) Metex M-629 and then etched.	17
4.5	Scanning electron microscope images of the cross section at the a) colored oxide and b) black oxide for the reference sample that have only been HIP-treated.	18
4.6	Scanning electron microscope images of the cross sections at the colored oxide regions (top) and black oxide regions (bottom) for samples pickled in a) HCl, b) HF/HNO ₃ and c) Metex M-629.	19

4.7	Scanning electron microscope images of the cross sections at the colored oxide regions (top) and black oxide regions (bottom) for samples pickled in a) HCl, b) HF/HNO ₃ and c) Metex M-629 and then etched.	19
4.8	SEM-EDS image and spectra of the colored oxide regions of a reference sample.	21
4.9	SEM-EDS image and spectra of the black oxide regions of a reference sample.	22
4.10	SEM-EDS image and spectra of the colored oxide regions of a sample pickled in HCl.	22
4.11	SEM-EDS image and spectra of the black oxide regions of a sample pickled in HCl.	23
4.12	SEM-EDS image and spectra of the black oxide regions of a sample pickled in HF/HNO ₃	24
4.13	SEM-EDS image and spectra of the colored oxide regions of a sample pickled in HF/HNO ₃ and then etched.	25
4.14	SEM-EDS image and spectra of the black oxide regions of a sample pickled in HCl and then etched.	26
4.15	SEM-EDS image and spectra of the black oxide regions of a sample pickled in HF/HNO ₃ and then etched.	26
4.16	SEM-EDS image and spectra of a sample that has been HIP-treated in an optimal environment.	27
4.17	The mean and standard deviation of surface roughness (R_a) on a) the colored oxide regions and b) the black oxide for the samples pickled in HCl, HF/HNO ₃ or Metex M-629 and then etched.	29
4.18	The mean and standard deviation of surface roughness (R_z) on a) the colored oxide regions and b) the black oxide for the samples pickled in HCl, HF/HNO ₃ or Metex M-629 and then etched.	29
A.1	Images showing how the samples was placed in the hot isostatic pressing. The samples used look yellow/ gold in the pictures before HIP and black/ gray after HIP.	I
B.1	Images of the front side of the samples after they have been HIP-treated. The images were taken using a laser microscope	III

B.2	Images of samples A90-8, A90-110 and A90-111 where the left side has been pickled in HCl and the right side has been pickled in HCl and etched.	IV
B.3	Images of samples A90-27, A90-80 and A90-99 where the left side has been pickled in HF/HNO ₃ and the right side has been pickled in HF/HNO ₃ and etched.	IV
B.4	Picture of sample A90-77, A90-82 and A90-93 where the left side has been pickled in Metex M-629 and the right side has been pickled in Metex M-629 and etched.	V
D.1	Optical microscope images of the cross section at the colored oxide regions (top) and black oxide regions (bottom) of reference samples (only HIP-treated).	IX
D.2	Optical microscope images of the cross section at the colored oxide regions (top) and black oxide regions (bottom) of samples pickled in HCl.	IX
D.3	Optical microscope images of the cross section at the colored oxide regions (top) and black oxide regions (bottom) of samples pickled in HCl and then etched.	X
D.4	Optical microscope images of the cross section at the colored oxide regions (top) and black oxide regions (bottom) of samples pickled in HF/HNO ₃	X
D.5	Optical microscope images of the cross section at the colored oxide regions (top) and black oxide regions (bottom) of samples pickled in HF/HNO ₃ and then etched.	XI
D.6	Optical microscope images of the cross section at the colored oxide regions (top) and black oxide regions (bottom) of samples pickled in Metex M-629.	XI
D.7	Optical microscope images of the cross section at the colored oxide regions (top) and black oxide regions (bottom) of samples pickled in Metex M-629 and then etched.	XII
E.1	SEM-EDS image and spectra of the colored oxide regions of a reference sample.	XIII
E.2	SEM-EDS image and spectra of the black oxide regions of a reference sample.	XIV
E.3	SEM-EDS image and spectra of the black oxide regions of a sample pickled in Metex M-629.	XIV

E.4	SEM-EDS image and spectra of the colored oxide regions of a sample pickled in HCl.	XV
E.5	SEM-EDS image and spectra of the colored oxide regions of a sample pickled in HCl and then etched.	XV
E.6	SEM-EDS image and spectra of the black oxide regions of a sample pickled in HF/HNO ₃ and then etched.	XVI
E.7	SEM-EDS image and spectra of the colored oxide regions of a sample pickled in Metex M-629 and then etched.	XVI
E.8	SEM-EDS image and spectra of the black oxide regions of a sample pickled in Metex M-629 and then etched.	XVII
E.9	SEM-EDS image and spectra on the black oxide regions of a sample pickled in Metex M-629 and etched.	XVII

List of Tables

2.1	Elemental composition for additive manufactured LPBF Alloy 718 from ASTM [21].	6
3.1	The parameters used for the pickling solutions. For the Metex M-629 solution a current density of 6 A/dm ² is applied.	13
C.1	Breakdown of the treatment on the samples used in this project. All samples that have been pickled have also been HIP-treated. The E56 sample has been HIP-treated in a contaminant free environment.	VII
F.1	Measured surface roughness R_a on both the colored oxides and the black oxides on the samples.	XIX
F.2	Measured surface roughness R_z on both the colored oxides and the black oxides on the samples.	XX

1. Introduction

Materials used in aerospace industries often require exceptional performance, heat resistance or strength at the expense of cost or conventional manufacturing [1]. Some materials are used because of their long term reliability. These materials are usually metal alloys but could also include some polymeric based materials. Because of the strict safety requirements within the area of aerospace materials. The use of such materials is therefore governed by international standards ASTM (American Society for Testing and Materials), AMS (Aerospace Material Standard) or company specifications.

By using additive manufacturing (also known as AM) techniques parts can be made with specific constraints that would be impossible to meet with other techniques [2]. However parts manufactured with AM contain a large number of pores/defects [3]. These pores negatively affect mechanical performance by reducing the yield strength [4]. When the part is manufactured via the AM technique laser powder bed fusion (also known as LPBF) these pores can be reduced. The pores close to the surface are removed in elevated temperatures and high pressures in an inert environment in a process called hot isostatic pressing (also known as HIP) [3]. Although the environment should be inert, oxide scales still form on the surfaces of the parts [5]. As these oxide scales are unwanted they need to be removed. This can be done using an etching process. The process from additive manufacturing to the finished product can be seen in figure 1.1.

The etching utilizes the chemical energy of the etching solution which binds to the oxides that are then released from the workpiece [6]. Via diffusion the oxide remains are then transferred away from the surface allowing new etchants to reach the surface.

1.1 Background

At GKN LPBF is used to print components with complex shapes. However these materials have an unfavorable microstructure when finished. In order to obtain a better structure HIP is used. HIP reduces near surface defects and reduces residual stresses. Even though it is performed in a controlled environment oxidation can occur at the surface which complicates the etching procedures that are performed after the HIP.

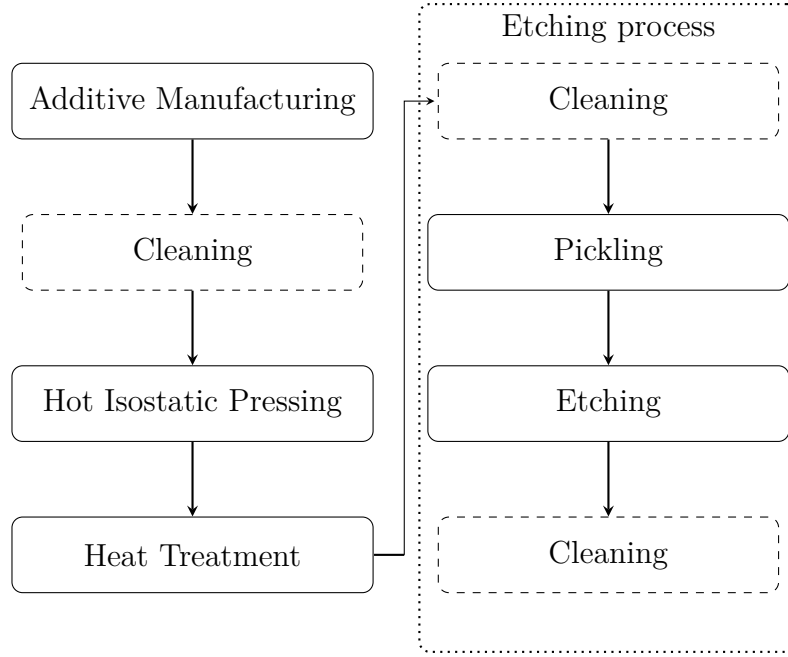


Figure 1.1: Flow chart of the process for manufacturing a part using additive manufacturing, hot isostatic pressing and etching.

1.2 Aim

The aim of the project is to investigate the effect of different pickling solutions on the removal of oxide scales on the LPBF alloy 718 after additive manufacturing and hot isostatic pressing. This is done to develop a better understanding of how the pickling step in the etching process effects the final product. The chemical composition of the oxides found on the samples will also be determined.

1.3 Limitations

The project is limited to focus only on the LPBF alloy 718. The project will also only focus on the pickling step in the etching process (as seen in figure 1.1) and the only chemicals that will be used for pickling are HCl, a mixture of HF and HNO₃ and Metex M-629 with electrolysis.

2. Theory

In this chapter the theory of the most important manufacturing steps, the material used and the methods used for analyzing the results are presented.

2.1 Manufacturing process

In this project the manufacturing of the sample plays a big role in how their properties turn out. In this section the theory of the most critical steps, additive manufacturing, hot isostatic pressing and etching, are briefly explained.

2.1.1 Additive Manufacturing (AM)

Additive manufacturing is a way to manufacture parts that have specific geometries or other restrictions that make them difficult to manufacture with normal methods [2]. Another factor is that cast parts become increasingly expensive as there are few manufacturers. In additive manufacturing the parts are built layer by layer from a computer model which leads to the parts not being limited by casting dies or traditional machining limits. Because of the way that the parts are modeled and manufactured the weight to strength ratio can be optimized. This is done by computer programs that remove unnecessary material without compromising the mechanical performance.

2.1.1.1 Laser Powder Bed Fusion (LPBF)

Laser powder bed fusion is a type of AM technique [7]. The manufacturing works by fusion of powdered metal according to a CAD (computer aided design) model. First metal powder is deposited onto the building plate and then a high concentration laser focuses on the selected area, defined in the CAD model, fusing the metal powder. The remaining powder is then removed, the building plate is lowered and new metal powder is deposited. This is repeated until the part is finished. The process is schematically shown in figure 2.1. The layers are usually between 20 μm and 100 μm thick [7]. This is because larger particles tend to result insufficient fusion while smaller particles tend to

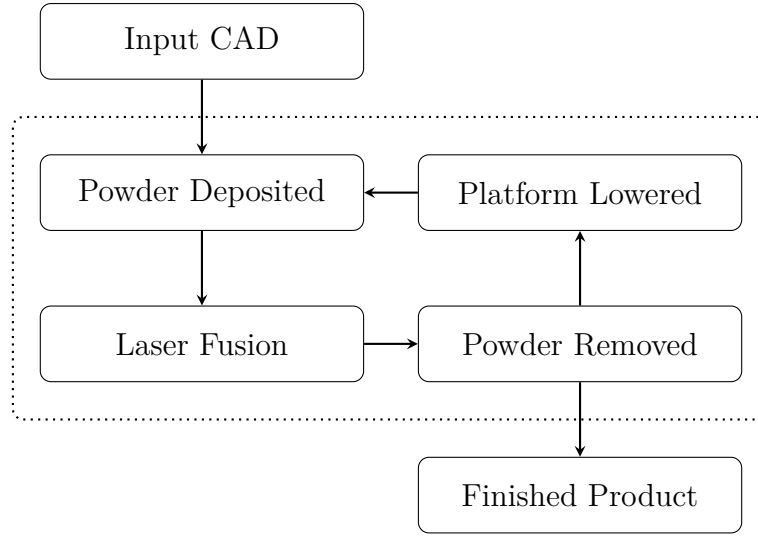


Figure 2.1: Flow chart of the additive manufacturing process laser powder bed fusion.

group together causing reduced particle flowability and consequently reduced deposition rate.

2.1.2 Hot Isostatic Pressing (HIP)

To reduce the pores that form in the part during additive manufacturing, HIP is used [8]. HIP is a process that involves the simultaneous application of heat and gas pressure that closes internal pores and improves homogeneity [9].

The environment in the HIP used for LPBF Alloy 718 is typically 1120 to 1200°C and 100 to 150 MPa with argon as the environment [5]. Due to trace amounts of oxygen in the argon environment, in the pores of the material and from the surfaces of the powder used in the printing process, surface oxide layers can form in the HIP.

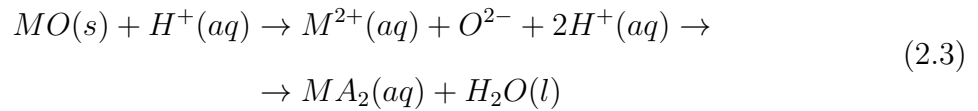
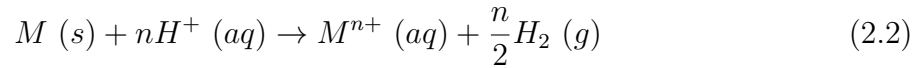
2.1.3 Etching

Chemical etching is a method to perform controlled dissolution of a part with the use of strong chemicals [10]. The etching solution is usually an alkaline solution or an acid [6]. The workpieces that are to be etched is sprayed or immersed in the etching solution where the chemical energy of the etchants removes unwanted oxides from the workpiece. To prevent etching on parts of the workpiece that are to be left untouched it can be masked. Masking prevents the chemicals from reaching the material and therefore hinders the removal.

Ferric Chloride (FeCl_3) is a commonly used etching agent [11]. It utilizes the Fe^{3+} -ions oxidation properties to perform redox reactions with metals as seen in reaction 2.1. The reaction reduces the iron-ions to produce metal cations. These metal cations are then soluble in water and can therefore be removed from the surface.



Strong acids are also used as etching agents to remove both solid metal and metal oxides [12]. For the reaction of metal with acid, the hydrogen ions take electrons from the metal forming hydrogen gas and metal ions which react with the ions from the acid to form a metal salt as seen in reaction 2.2 [13]. When acid reacts with metal oxides the hydrogen ion instead bonds to the oxygen in the oxide to form water. This leaves the metal ions which bond with the ions from the acid to form a metal salt as seen in reaction 2.3. The dissolved metal salts can then be transported away from the surface. Note that the equations change depending on if the metal forms M^+ , M^{2+} or M^{3+} .



Nitric acid (HNO_3) is especially good to use in etching as the nitrate ion has a strong oxidizing power [14]. This comes from the +5 oxidizing state of the nitrogen. The nitrate reacts with the metals forming soluble metal nitrates which are then easily removed [15]. However the oxidizing power is diminished below concentrations of 2 M. The oxidation power can be increased by adding chlorate, permanganate, hydrogen peroxide or bromine.

The mixture of HF/HNO_3 has shown good qualities in etching titanium and titanium oxides [16]. The HF reacts with TiO and Ti to form $[\text{TiF}_6]^{2-}$ that are soluble in water. When HF reacts with Ti hydrogen gas is produced which can weaken the material through hydrogen absorption. In order to limit hydrogen production HNO_3 is used to oxidize the hydrogen. This reaction instead produces water and NO_x .

Table 2.1: Elemental composition for additive manufactured LPBF Alloy 718 from ASTM [21].

Element	C	Mn	Si	P	S
wt%	≤ 0.08	≤ 0.35	≤ 0.35	≤ 0.015	≤ 0.015
Element	Cr	Co	Mo	Nb & Ta	Ti
wt%	17.0-21.0	≤ 1.0	2.80-3.30	4.75-5.50	0.65-1.15
Element	Al	Fe	Cu	Ni	B
wt%	0.20-0.8	Bal.	≤ 0.3	50.00-55.00	≤ 0.006

2.2 LPBF Alloy 718

LPBF Alloy 718 (also known as Inconel 718 or In718) is a nickel-chromium superalloy that is designed to exhibit high strength, good ductility, weldability, creep rupture properties and significant yields up to 700°C [17]. The alloy is commonly used in turbine discs, casings and afterburner components and fuel and hydraulic systems for modern turbine engines (both commercial and military) and rocket propulsion systems [18]. The alloy is popular to use because it has a good combination of mechanical properties, price and processability [19].

The alloy consists mainly of γ , γ' , γ'' and δ phases where γ is a solid solution strengthened FCC matrix phase with γ' ($\text{Ni}_3(\text{Al,Ti})$) and γ'' (Ni_3Nb) as the strengthening phases. The δ comes from the aging of the γ'' phase which is why there is a temperature restriction [20]. The chemical composition of LPBF Alloy 718 can be seen in table 2.1. The different elements in the alloy have different roles. Nickel is the main matrix and is used instead of iron as it is more corrosive resistant. Chromium is used for its excellent corrosion resistance as it forms a protective oxide on the surface. Iron and molybdenum influences solid solution strengthening, niobium is a constituent of the γ'' strengthening phase and titanium and aluminum are in the γ' phase [5]. Traces of carbon, silicon and manganese form carbides that act as a glue that holds the grains together.

2.2.1 Secondary Phases Formed in LPBF Alloy 718

One secondary phase formed in LPBF Alloy 718 that is of interest in this project is oxides. Oxides that can form include chromia (Cr_2O_3), NiO, spinels ($(\text{Ni,Fe})(\text{Cr,Fe})_2\text{O}_4$), Nb-rich

oxides (Nb_2O_5) and titanium and aluminum oxides (TiO_2 , Al_2O_3) [5]. The oxides that form on LPBF Alloy 718 are highly dependent on the diffusion of oxygen, chromium, titanium and manganese through the surface [22]. At high temperatures the oxides on LPBF Alloy 718 can be seen as three layers with the innermost composed of Ni_3Nb enriched with Al, Ti and Nb oxides, the outermost composed of TiO_2 - MnCr_2O_4 spinels and the layer between being Cr_2O_3 .

Another phase that can form is Niobium carbides [23]. These carbides have a very high melting temperature and are not soluble in the bulk material. Niobium carbides are a common phase in the material and are present just after solidification [24]. Studies have shown that the rapid cooling in LPBF has suppressed the formation of niobium carbides. Although they are not formed during the printing process they can still form during the subsequent heat treatment.

2.3 Analytical Methods

In this section the theory on the methods used to analyze the oxides on the surface of the samples is presented.

2.3.1 Laser Microscope

Laser microscopes use two different light sources, a white light for color information and a laser source for determining height differences [25]. The microscope uses the confocal principle to detect the height differences in a sample. The laser is scanned in X and Y direction over the sample surface. During the scanning the microscope automatically detects the focus point at each point on the sample surface by analyzing the intensity of the light that reaches the detector. The height differences can then be determined by analyzing how much the microscope needs to move in the Z direction to find the focus point.

2.3.1.1 Surface Roughness

Surface roughness is a measurement of how smooth a surface is [26]. There are two main parameters that are used to explain surface roughness. The first one is average roughness

R_a which is primarily used in the US and the other one is mean roughness depth R_z which is the international standard.

R_a is calculated using equation 2.4 [26].

$$R_a = \frac{1}{L} \int |y(x)| dx \quad (2.4)$$

Where L is the sampling length, x is position along the sampling length and $y(x)$ is the deviation from the mean line. R_z is calculated by dividing the surface profile into five equal sections. In each of the five sections the difference between the highest peak and the lowest valley is calculated. R_z is then defined as the mean of these five differences. For both R_a and R_z a lower value means a smoother surface.

2.3.2 Scanning Electron Microscopy (SEM)

Scanning electron microscopy is a type of microscope that uses a focused electron beam to scan the surface of a sample to generate images with resolutions greater than those of optical microscopes [27]. The technique is used to obtain images with a resolution from less than 1 nm up to several nm and information about the surface of the samples.

The SEM works by an electron gun generating electrons that are accelerated through a column at a specified accelerating voltage [27]. A schematic representation of a SEM can be seen in figure 2.2. The generated electrons are lined up with a positively charged anode. Condenser lenses and apertures reduce the diameter of the beam and focus it on the surface of the sample. Scancoils are then used to scan the beam over the sample surface. These scancoils are located before the last lens. The sample is mounted on a stage in the chamber area. Both the chamber and the column are kept under vacuum. When the electrons hit the sample surface a variety of signals can be analyzed with the appropriate detectors. The most commonly analyzed are backscattered electrons (BSE), secondary electrons (SE) and characteristic X-rays. Figure 2.3 shows a schematic image of the interaction volume and indicates where the different signals originates from.

Each signal gives different information [27]. The BSE are primary electrons that interact with the nucleus of an atom in the sample. The nucleus causes a deflection that makes it possible for the electron to be remitted from the surface. The yield of BSE depends on the elemental composition of the surface. Heavier nuclei possessing a higher positive charge

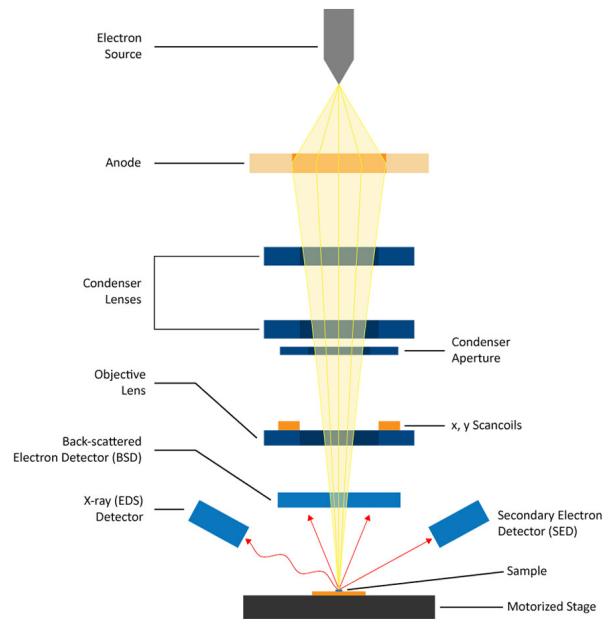


Figure 2.2: Schematic representation of a scanning electron microscope [27].

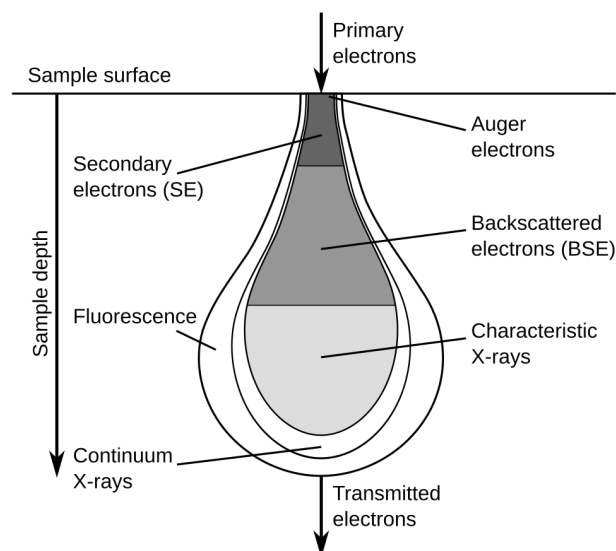


Figure 2.3: Schematic image showing the interaction volume and where the different signal originates from in a sample analyzed in SEM.

results in a higher yield, making regions of heavy elements brighter in the BSE image. SE are emitted by inelastic scattering or when it takes up energy from a primary electron. The images produced with SE have the best surface resolution because of the relatively small interaction volume. This also results in edges generating more SE and holes generating fewer SE resulting in brighter and darker regions in images. Characteristic x-rays are emitted when an ionized atom relaxes. This happens when an excited electron falls down to the ground state. The void in the ground state is produced when an incoming electron knocks out an electron in the lower state. The x-rays energy is equivalent to the difference in energy between the two electron states. Due to these energies being specific for each element the element can be known by analyzing the x-rays.

2.3.2.1 Energy-Dispersive X-ray Spectroscopy (EDS)

Energy-Dispersive X-ray Spectroscopy (EDS) is a technique that is often used together with SEM to determine the elements present on a sample's surface [28]. An x-ray spectrometer is used to detect x-rays emitted from the surface of a sample when it is irradiated with an electron beam. The detected x-rays can then be analyzed to form a spectrum and determine the relative elemental abundance at different points on the surface of the sample. This information can then be used to form elemental distribution maps for the different elements on the surface. The resolution of the EDS is around 2 μm at 15 keV which therefore also is the uncertainty of the EDS measurement. This is due to the width of the interaction volume at the characteristic X-rays (as seen in figure 2.3). However most of the signal obtained comes from the middle of the interaction volume and should therefore not affect the results and conclusions in this work.

3. Material and Methods

This chapter will cover the materials and methods used in this project.

3.1 Literature study

A literature study has been conducted using Chalmers Libraries database resources with primary use of ScienceDirect. The literature study also used H. Appelqvists Master thesis "A Literature Study of the Etching Process on Nickel- and Titanium- Based Superalloys" [29] and S. Goels PhD Thesis "Thermal post-treatment of alloy 718 produced by electron beam melting" [30] as starting points for sources. Key words for the literature study are additive manufacturing, laser powder bed fusion, hot isostatic pressing (HIP), LPBF alloy 718 (Inconel 718), etching (chemical milling) and oxide scales. The goal of the search is to gather information for a deeper understanding of additive manufacturing, laser powder-bed fusion (LPBF), LPBF alloy 718 and its different phases and etching.

3.2 Material

The test samples used in this project are LPBF Alloy 718 pieces with dimensions 40 mm x 25 mm x 5 mm. The samples have been additively manufactured using LPBF at GKN Aerospace in Trollhättan and HIP-treated at Bodycote in Surahammar. During the HIP of the samples there were multiple other samples present of unknown materials and the environment was recycled with no measurements of the ingoing and outgoing gas. This introduced a risk of contamination from both the environment and from other samples. The HIP was also executed with the samples lying on top of each other. Images of this can be seen in appendix A. This led to the samples having discolorations where they had been covered by another sample. This discoloration can be seen in figure 3.1 together with a sample that has not been HIP-treated. Images of all samples used in this project can be found in appendix B. The discolored part will be referred to as colored oxide regions and the other parts as black oxide regions.

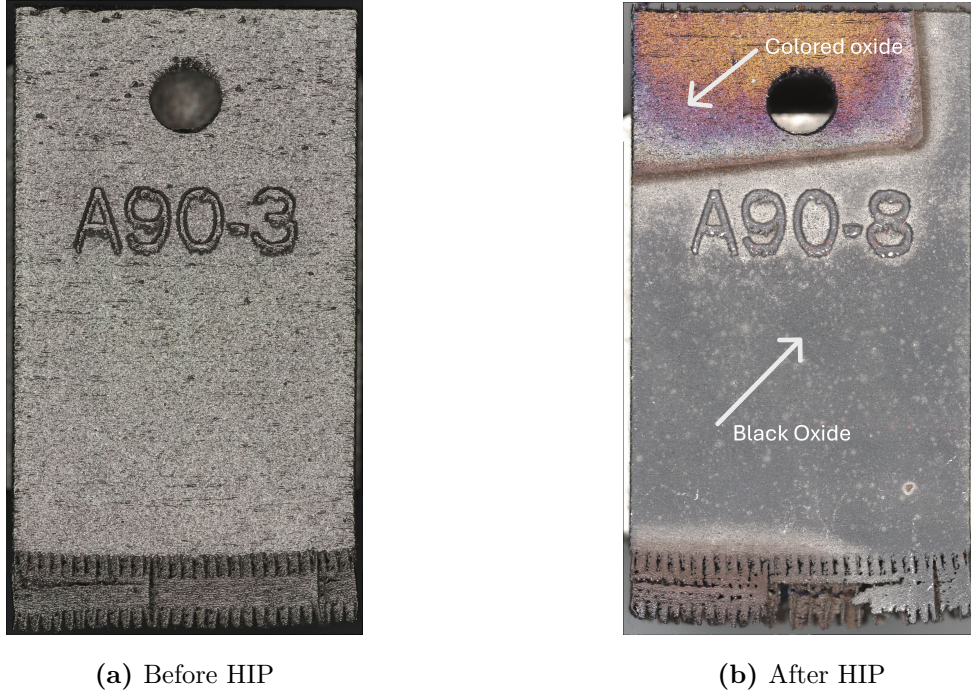


Figure 3.1: Laser microscope images of the front side of a typical sample a) before HIP and b) After HIP. In b) the arrows show the colored and the black oxide regions.

3.3 Etching Method

For the pickling and etching the samples are cut into two equal parts. One part is only pickled while the other part goes through the whole etching process (figure 1.1). This is done to minimize the variations that come from the AM process and to see the differences between the whole etching process and the pickling. The process is repeated three times for each of the three pickling solutions HCl, a mixture of HF and HNO₃ and Metex M-629 (used with electrolysis) for a total of 18 samples. The exact concentrations and exposure times used for the pickling solutions can be found in table 3.1. The etching solution is an acid-based solution that has been developed to etch nickel-based materials. All the samples were etched in the etching solution for 10 minutes. Three samples that have not undergone any etching treatments are used as references to see if the etching process affects the oxides. An additional sample that has been HIP-treated in a contamination free environment is also used as a reference. A full breakdown of how the samples were treated can be found in appendix C.

As seen in figure 1.1 the parts are cleaned both before and after pickling and etching.

Table 3.1: The parameters used for the pickling solutions. For the Metex M-629 solution a current density of 6 A/dm² is applied.

Solution	Time (min)	Concentration (g/l)
HCl	10	212
HF/HNO ₃	10	14 (HF), 355 (HNO ₃)
Metex M-629	5	110

Before pickling the cleaning is done with CEE BEE SB300 LFG (Alkaline solution). A water break test is conducted to ensure that the metal is sufficiently clean. The cleaning after etching is done with water in several steps to ensure that all acids are removed. The samples are dipped in water between the pickling and etching to reduce the spillage of chemicals. This is not necessary for full scale production as the pickling and etching will be placed next to each other.

3.4 Analyzing Methods

The samples in this project were analyzed using three different analytical methods. A laser microscope was used to obtain images of the surfaces and measure the surface roughness. To get a better measurement of the surface roughness the measurements are repeated on five parallel lines separated by 4 pixels where the final value is the mean of the five separate measurements.

An optical microscope was used to examine the cross section of the surfaces. In the optical microscope enhancements of up to 1000x were accomplished. The optical microscope was used as it is faster and easier to use than SEM. This made it easy to see differences at a large scale before SEM was used for more detailed analysis. In the SEM magnifications of up to 20,000x were used to obtain precise images of the surface. SEM-EDS was used to obtain point, line-scans and area analyses of the cross section of the samples. To make the analysis easier in the optical microscope and SEM the samples were cut up, cast in PolyFast and polished. This is done so that the cross section surface is as level as possible. When the samples were cut up both colored and black oxide regions were ensured to be on the pieces cast in PolyFast.

3.4.1 Sample Preparation

To get the samples ready for optical microscope and SEM the samples were first cut up using a Struers Secotom 60 with Struers cut-off wheel 50A20, at 3000 rpm and a cutting speed of 0.05 to 0.3 mm/s. The samples were then ultrasonically washed in ethanol before they were mounted in 20 ml of PolyFast. After the mounting the samples were ground and polished in a Struers Tegramin using the grinding/ polishing plates MD-Piano 120/220/500, MD-Largo, MD-Dac and MD-Chem (in that order). MD-Piano is used with water, MD-Largo with 9 μm diamond suspension, MD-Dac with 3 μm diamond suspension and MD-Chem with a silica suspension. Between each plate and after the MD-Chem the samples were washed in a Struers Lavamin which only uses water and ultrasound. Just before the samples were put into the SEM the samples were again washed in ethanol.

4. Results and Discussion

This chapter will cover the results obtained and the discussion surrounding these. Figure 4.1 shows laser microscope images of the surface investigated for one sample of each pickling solution after pickling and after etching. Images of all the samples can be found in appendix B.

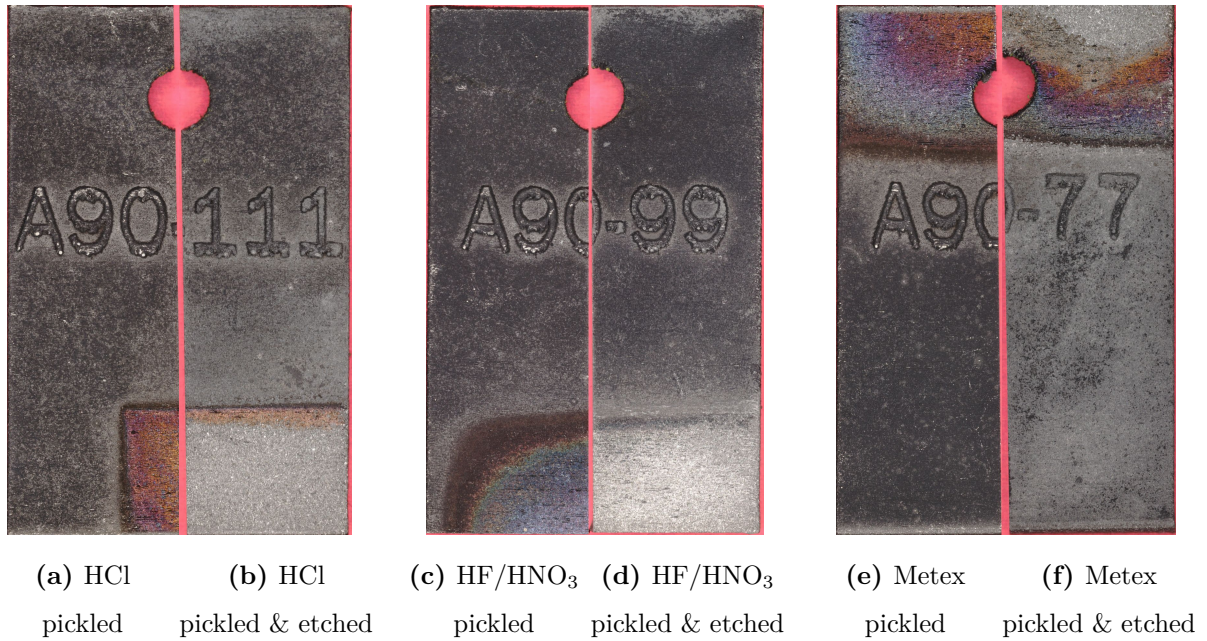


Figure 4.1: Laser microscope images of samples pickled in a-b) HCl, c-d) HF/HNO₃ and e-f) Metex M-629. The left side of the sample has been pickled and the right side has both been pickled and etched.

At the macroscopic scale the visual appearance after pickling appears similar for all three solutions as seen in figure 4.1. Both the colored oxide regions and the black oxide regions are largely unchanged compared with the reference sample. After the etching however there are more differences between the different pickling solutions. On the sample that was pickled in Metex M-629 (figure 4.1f) the black oxide regions have been partially removed showing the metallic bulk material underneath. On the same sample some of the colored oxide regions have been removed but most are still visible. The sample pickled in the HF/HNO₃ solution (figure 4.1d) shows almost no removal of the black oxide regions while the colored oxide regions are almost completely gone. The sample pickled in HCl (figure 4.1b) is somewhere between the other two solutions, some black oxide regions are removed

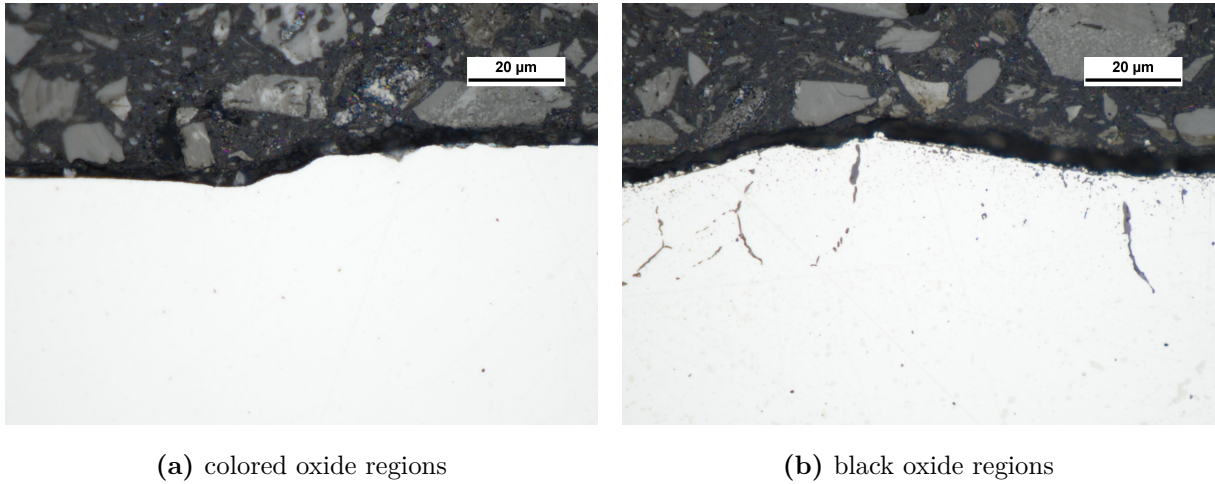


Figure 4.2: Optical microscope images of the cross section at the a) colored oxide and b) black oxide for the reference sample that have only been HIP-treated.

but not as much as from the Metex M-629 solution and much of the colored oxide regions are removed but not as much as from the HF/HNO₃ solution.

4.1 Analysis in Optical Microscope

When the cross section of the reference samples were investigated using an optical microscope it is clear that the surface and the near surface region at the colored and black oxide regions differ in appearance (figure 4.2). At the colored oxide regions (figure 4.2a) there are almost no near surface defects and the surface oxides are so thin that they are hard to detect with an optical microscope. At the black oxide regions (figure 4.2b) oxide intrusions extend approximately 30-40 μm into the sample at the grain boundaries as well as a clearly visible oxide layer at the surface. The reason for these differences is related to the way that the samples were put in to the HIP. As explained in section 3.2 when the samples were HIP-treated some parts of the samples were covered by other samples leading to the colored oxide. This coverage has led to the colored oxide regions of the samples not being exposed to the contamination in the environment in the same way as the black oxide regions. This coverage has been favorable as the near surface defects and surface oxides are unwanted.

After the samples have been pickled and etched the differences in the areas can still be seen. After pickling both surfaces are the same as before the pickling and there is no

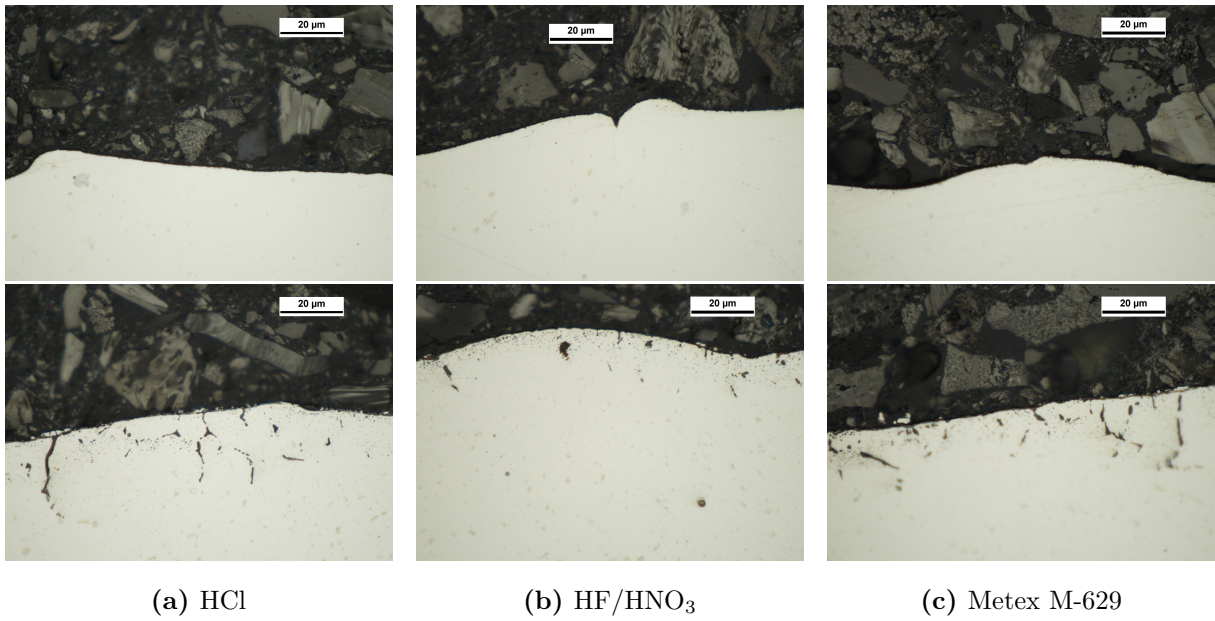


Figure 4.3: Optical microscope images of the cross sections at the colored oxide regions (top) and black oxide regions (bottom) for samples pickled in a) HCl, b) HF/HNO₃ and c) Metex M-629.

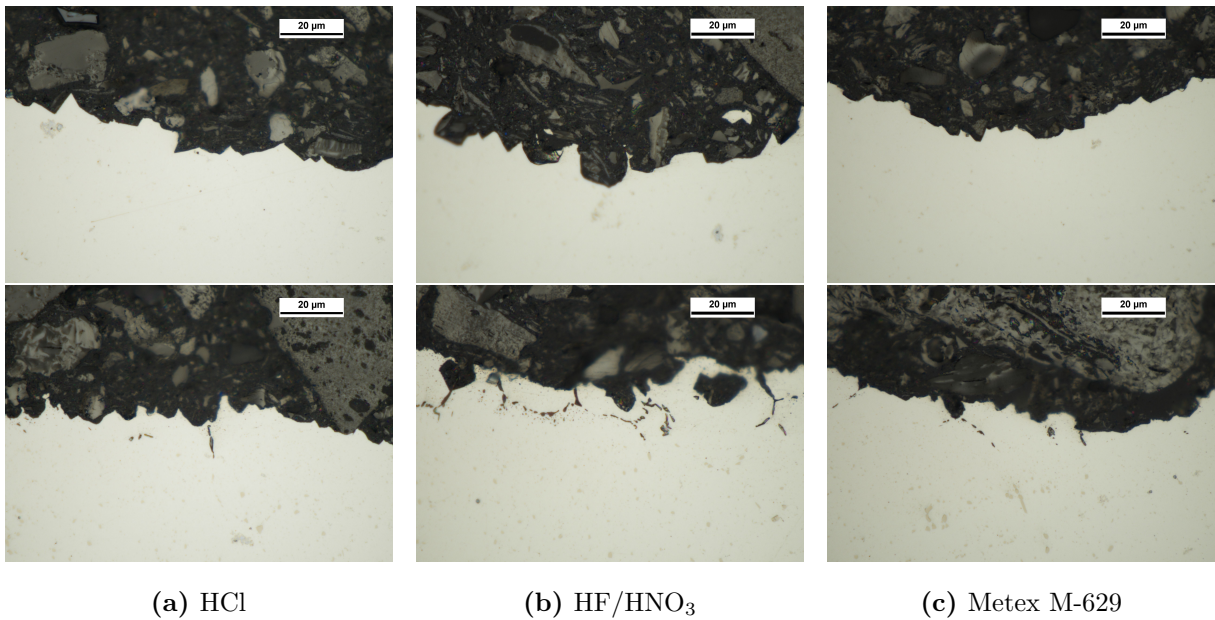


Figure 4.4: Optical microscope images of the cross sections at the colored oxide regions (top) and black oxide regions (bottom) for samples pickled in a) HCl, b) HF/HNO₃ and c) Metex M-629 and then etched.

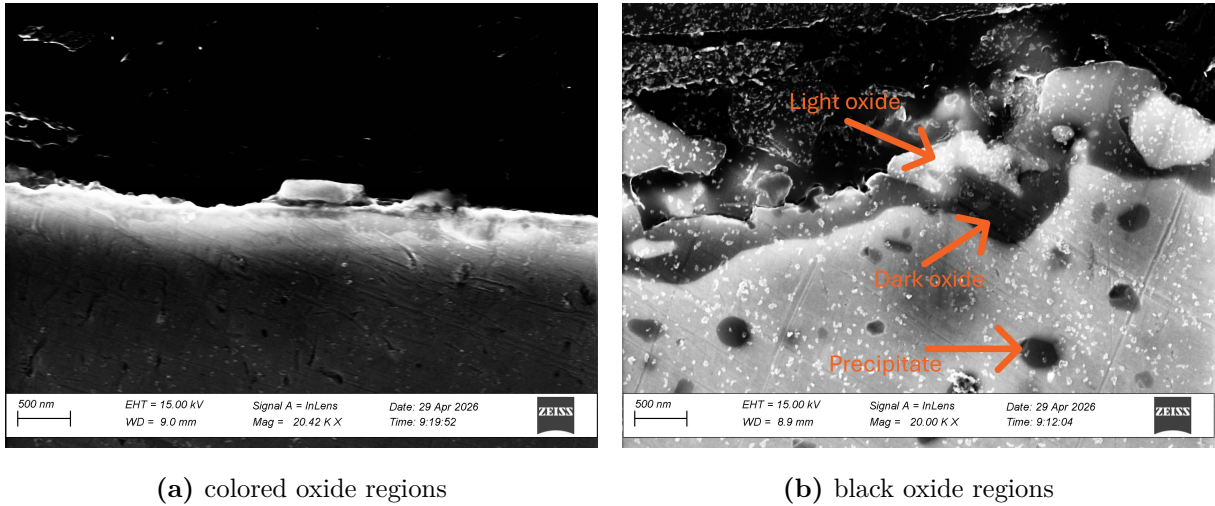


Figure 4.5: Scanning electron microscope images of the cross section at the a) colored oxide and b) black oxide for the reference sample that have only been HIP-treated.

difference between the pickling solutions that have been used. This can be seen in figure 4.3. However after the etching it is clear that material has been removed from the surface, this is seen in figure 4.4. Almost all of the oxide layer at the surface has been removed in both the colored and the black oxide regions for all of the pickling solutions. After etching the surfaces also look more ragged for all the pickling solutions. Microscope images of all of the samples can be found in appendix D.

4.2 Analysis in Scanning Electron Microscope

When analyzing the samples in a SEM the differences between the colored oxide regions and the black oxide regions become clearer. The colored oxide regions on the reference sample (figure 4.5a) appear as one solid layer integrated into the bulk material with small oxides scattered at the surface, most likely detached parts from the oxide layer. While the black oxide regions (figure 4.5b) appear to be a two-layer oxide on top of the bulk material. When analyzing the black oxide regions they appear to consist of a darker solid oxide underneath a lighter porous oxide. In addition to the surface oxides the near-surface region at the black oxide regions contains many precipitates embedded in the bulk material.

Images of the cross section after they have been pickled can be seen in figure 4.6. These images show that after the samples have been pickled the appearance of both the colored

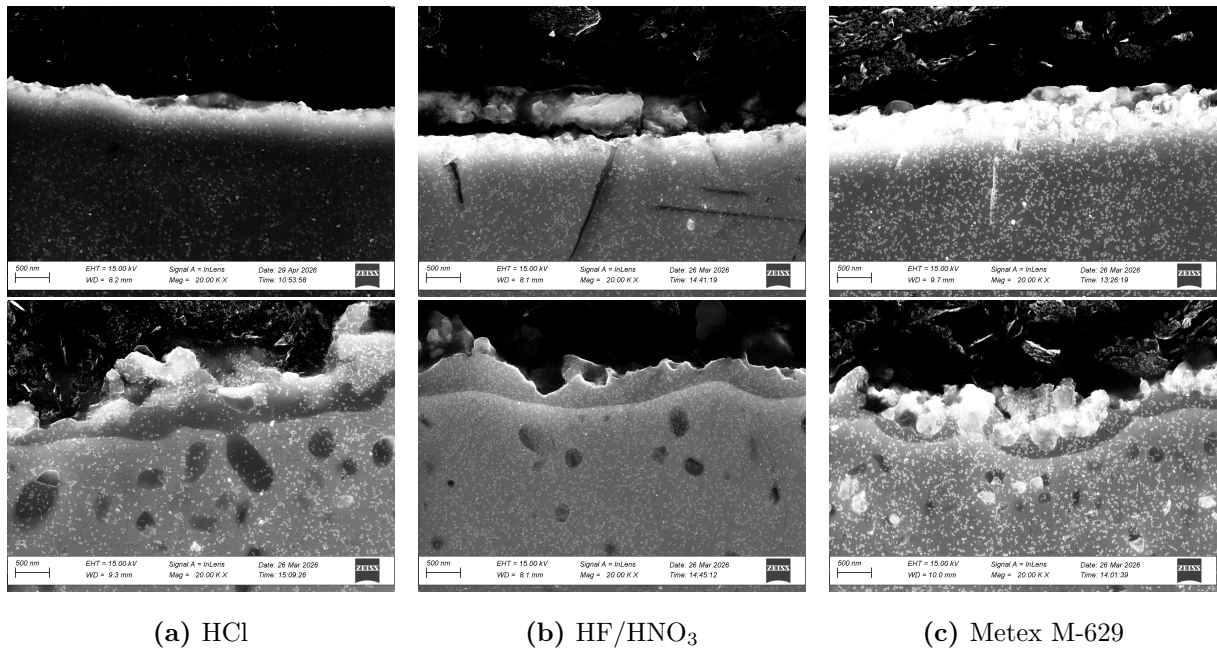


Figure 4.6: Scanning electron microscope images of the cross sections at the colored oxide regions (top) and black oxide regions (bottom) for samples pickled in a) HCl, b) HF/HNO₃ and c) Metex M-629.

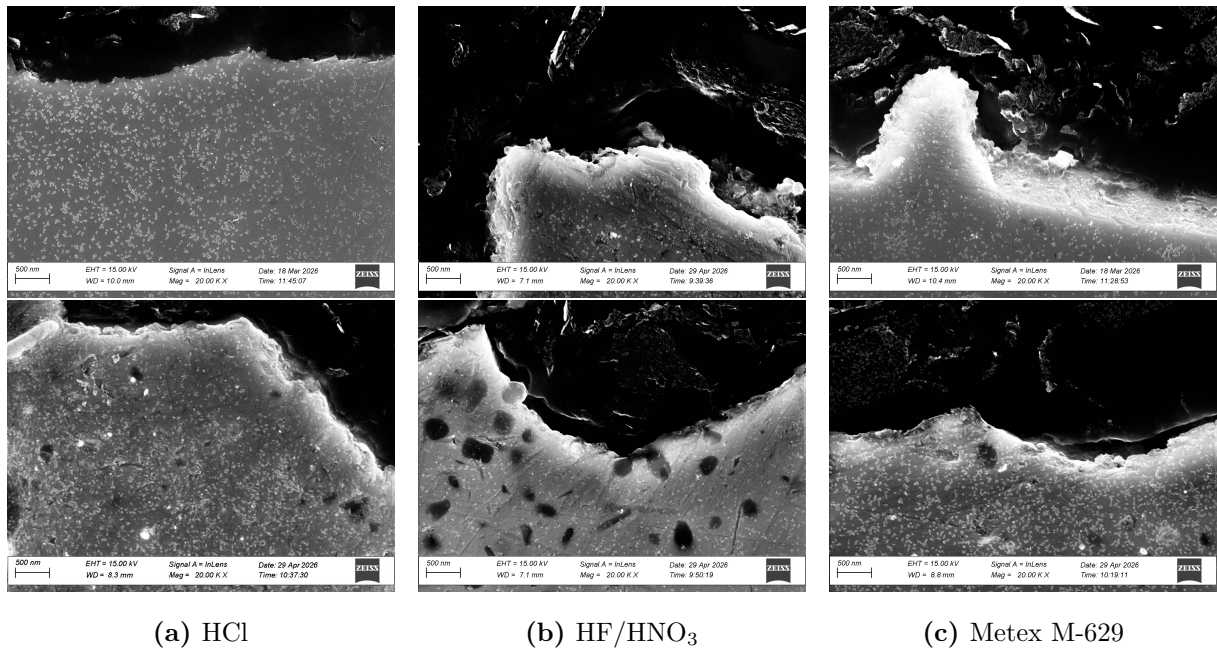


Figure 4.7: Scanning electron microscope images of the cross sections at the colored oxide regions (top) and black oxide regions (bottom) for samples pickled in a) HCl, b) HF/HNO₃ and c) Metex M-629 and then etched.

and the black oxide regions have changed. In the HF/HNO₃ solution (figure 4.6b) and the Metex M-629 solution (figure 4.6c) the colored oxide layer has become more detached from the bulk material and looks more like a separate phase. This is not the case for the HCl solution (figure 4.6a) as this seems to have no effect on the oxides. On the black oxide regions both the HCl and the Metex M-629 solutions seem to have no effect while the HF/HNO₃ removes the top layer of the oxides leaving only the darker oxides on the surface.

That the pickling does not remove any material is to be expected as it is not aimed at removing any material. Instead this step is supposed to loosen the oxides making them easier to remove in the subsequent etching. By comparing figure 4.5 and figure 4.6 at least the HF/HNO₃ and the Metex M-629 solutions change the appearance of the oxides and thus at least partially fulfills their purpose.

The results of both pickling and etching of the material can be seen in figure 4.7. Here the surface oxides at both the colored and the black oxide regions appear to have been completely removed and no difference can be seen between the different pickling solutions. The aggressiveness of the etching solution may reduce the importance of the pickling step under the tested conditions. This however has not been investigated and cannot be confirmed.

4.3 Chemical Analysis

To analyze the chemical composition of the oxides SEM-EDS has been used. The results of this can be seen in figures 4.8-4.15. The EDS results indicates that most of the oxides are aluminum containing oxides mixed with titanium containing oxides. The EDS also confirms that the bulk material for the most part consists of nickel, chromium and iron which is consistent with what is presented in table 2.1. Additional spectra for the samples not shown in this section can be found in appendix E.

Figure 4.8 shows that the colored oxide regions on the reference sample are mostly aluminum containing oxides and titanium containing oxides. These oxides do not seem to be mixed as peaks in one elemental signal coincide with minima in the other. The oxide also shows a high carbon content. This carbon content should not be there and can come

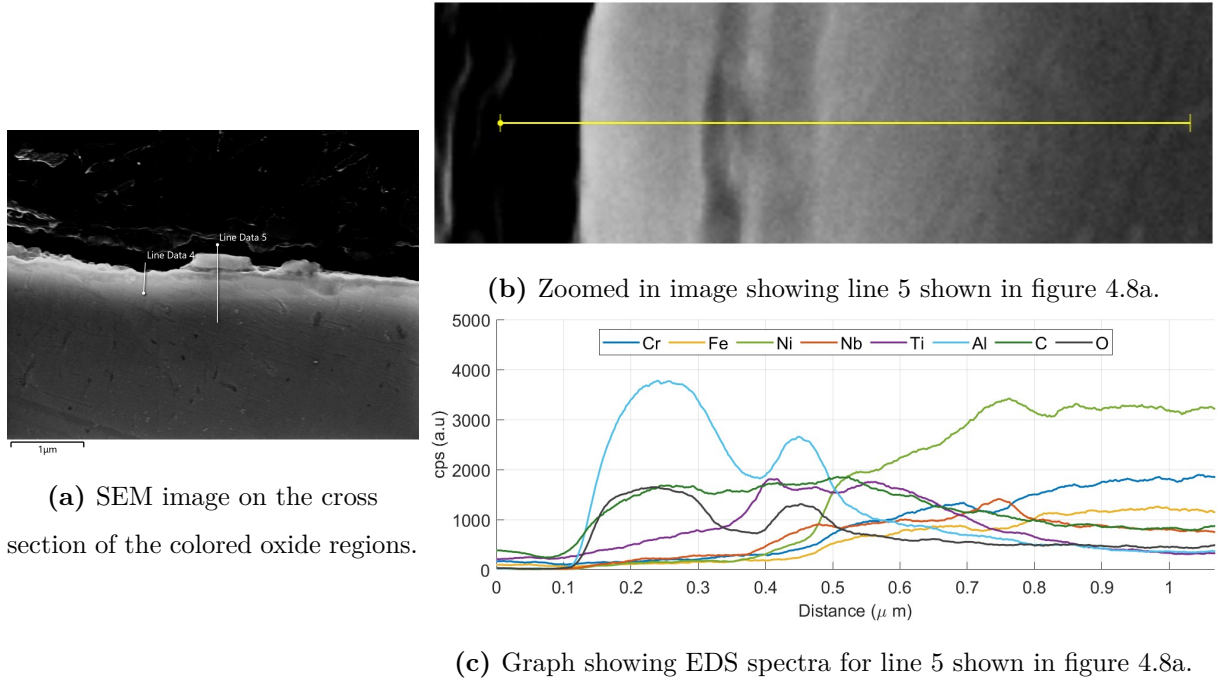


Figure 4.8: SEM-EDS image and spectra of the colored oxide regions of a reference sample.

from the Polyfix that the sample is mounted in or be contamination from hydrocarbons. This is however unlikely to be the whole explanation as the content should be even higher in the Polyfix (outside the sample). More likely this carbon comes from the environment in the HIP where carbon dioxide could have been present in the process or from the air when the sample has been waiting to get analyzed. This oxide also shows a low content of titanium.

As seen in figure 4.9 the black oxide regions also consist of aluminum and titanium containing oxides but here with clearer spikes in the titanium. These oxides also have a clear presence of niobium which is not seen in the colored oxide regions.

The pickled samples show chemistry similar to that of the reference samples. In figures 4.10-4.12 EDS analysis of the oxides are shown. There are two significant differences between the reference samples and the pickling samples. The colored oxide regions have very little titanium in them and the niobium layer on the black oxide regions is removed from the surface of the sample pickled in HF/HNO_3 . Line 16 in figure 4.11 supports that the aluminum, titanium and niobium are in separate phases, just as in the reference sample.

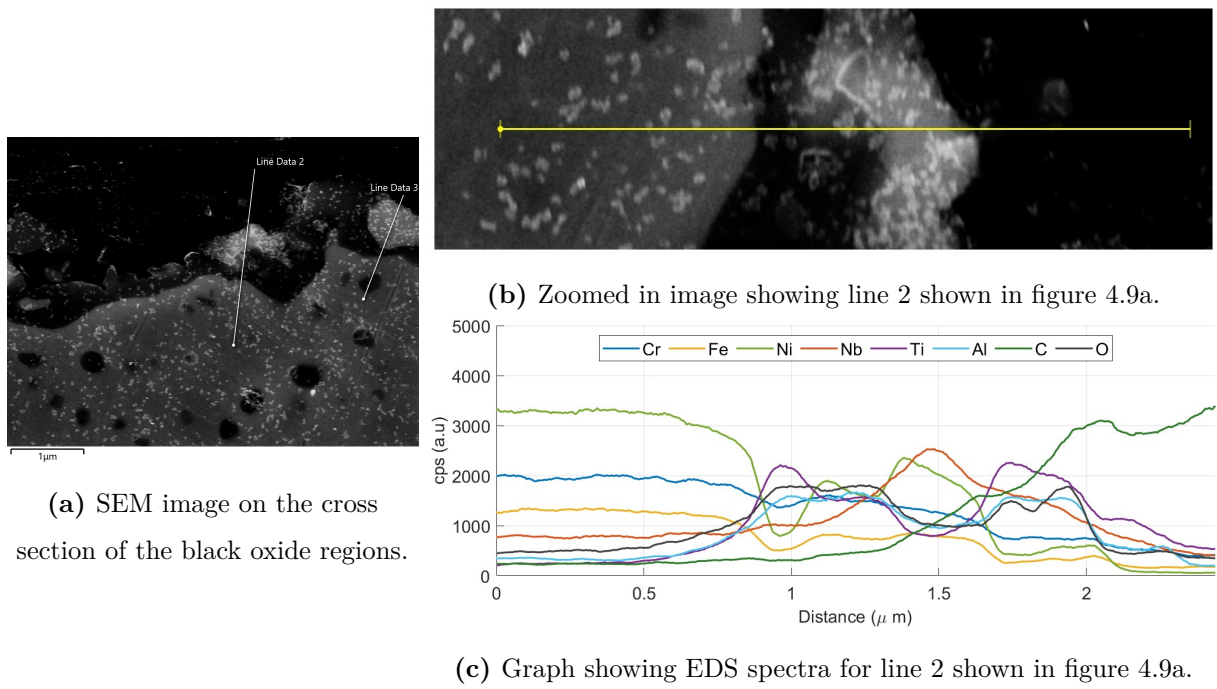


Figure 4.9: SEM-EDS image and spectra of the black oxide regions of a reference sample.

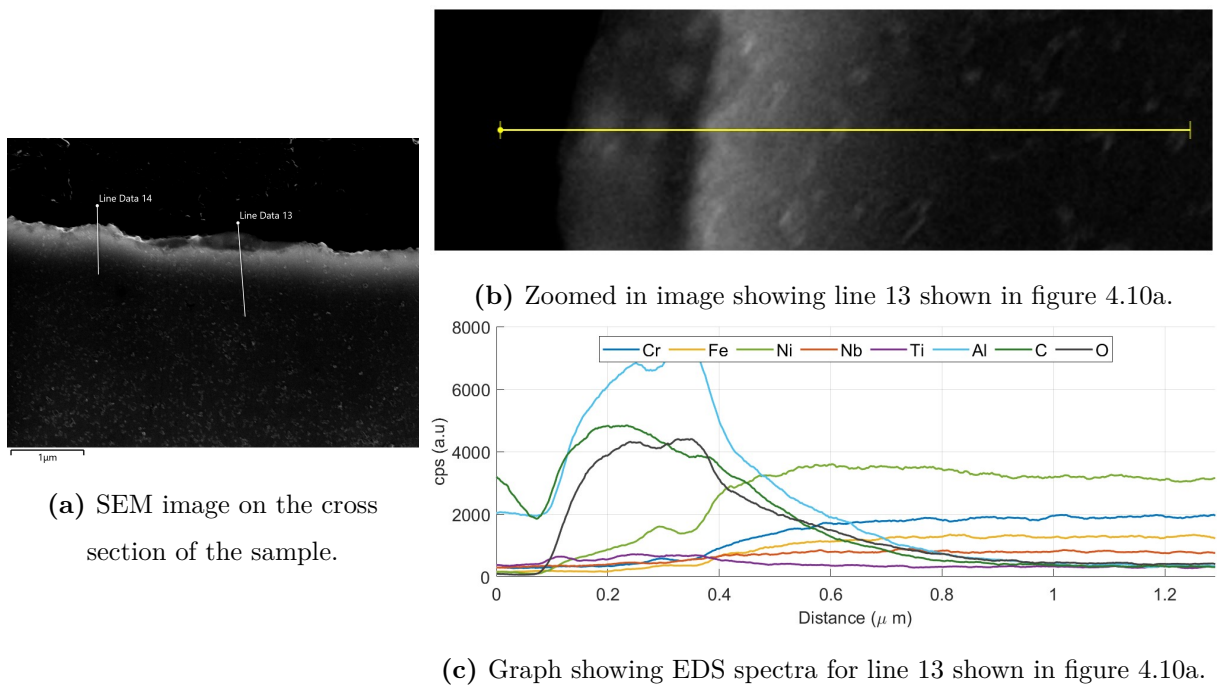
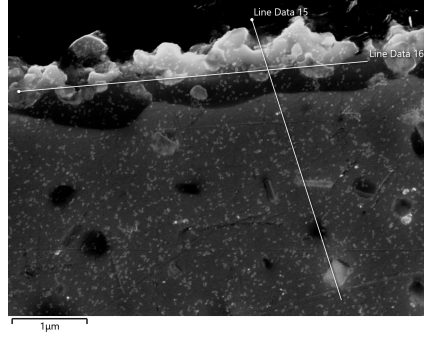
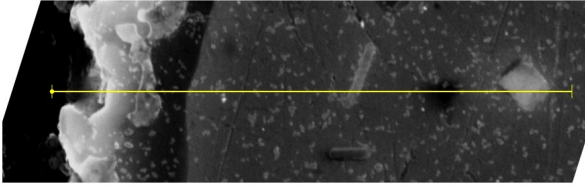


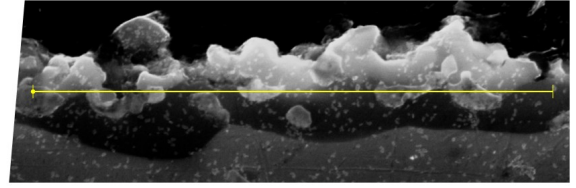
Figure 4.10: SEM-EDS image and spectra of the colored oxide regions of a sample pickled in HCl.



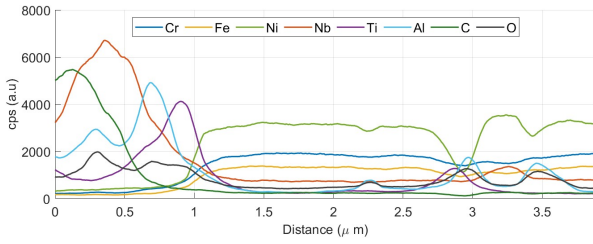
(a) SEM image on the cross section of the sample.



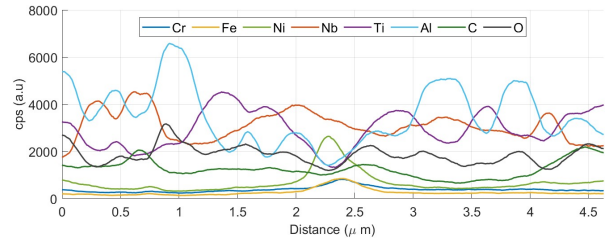
(b) Zoomed in image showing line 15 shown in figure 4.11a.



(c) Zoomed in image showing line 16 shown in figure 4.11a.



(d) Graph showing EDS spectra for line 15 shown in figure 4.11a.



(e) Graph showing EDS spectra for line 16 shown in figure 4.11a.

Figure 4.11: SEM-EDS image and spectra of the black oxide regions of a sample pickled in HCl.

When the samples have been etched more differences can be seen compared to the reference sample. The EDS of the etched samples can be seen in figures 4.13-4.15. The surface oxides are removed from all of the samples and the only oxides that are not removed are the oxides that are embedded beneath the black surface oxide regions.

The EDS for oxides embedded in the bulk material can be seen at around 2.9 μm in figure 4.11d, 1.5 μm in figure 4.12c and 0.7 μm in figure 4.14c. These all show that these oxides mostly consist of aluminum oxide and a little titanium containing oxide. As these oxides do not penetrate deep into the sample (as viewed from the cross section image in figure

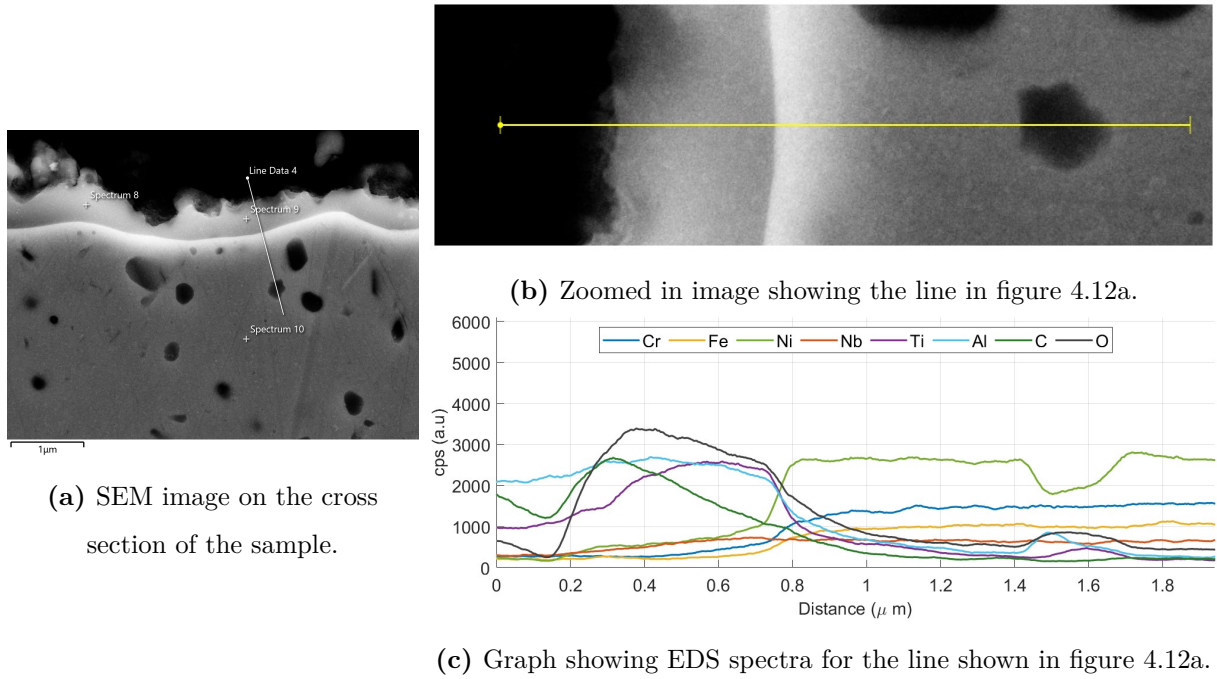


Figure 4.12: SEM-EDS image and spectra of the black oxide regions of a sample pickled in HF/HNO₃.

4.12) the material underneath it also gives of a signal. This is the reason why some of the oxides also show a high contents of nickel, chromium and iron.

As seen in table 2.1 the material should have a very low content of aluminum and titanium. This leads to two possible explanations for the presence of these oxides. One is that most of the aluminum and titanium diffuse to the surface during HIP and another is that there were other samples with high aluminum and titanium contents present during the HIP which could contaminate the environment. For this reason an additional sample that has been HIP-treated in a controlled environment without contaminants was tested. The EDS for this sample can be seen in figure 4.16. Since this sample also shows high aluminum and titanium contents, diffusion is the most likely explanation.

The main difference between the colored and black oxide regions is the niobium carbides that form the outermost layer of the surface at the black oxide regions. The only difference in where these black and colored oxide regions come from is how the samples were put in the HIP. The colored oxide regions were covered by other metallic specimens while the black oxide regions were completely exposed to the recycled gas. As the only possible carbon source is the environment in the HIP. The most probable explanation is that

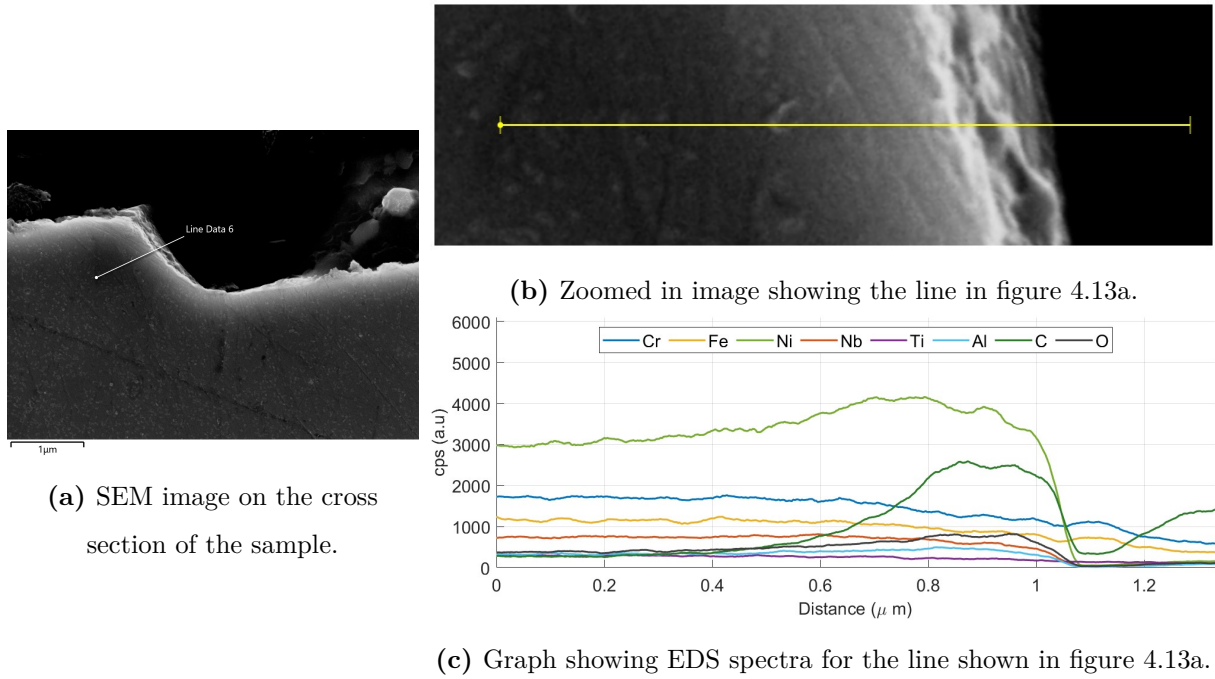


Figure 4.13: SEM-EDS image and spectra of the colored oxide regions of a sample pickled in HF/HNO₃ and then etched.

the niobium in the material has reacted with carbon in the environment under high temperature and pressure to form niobium carbides.

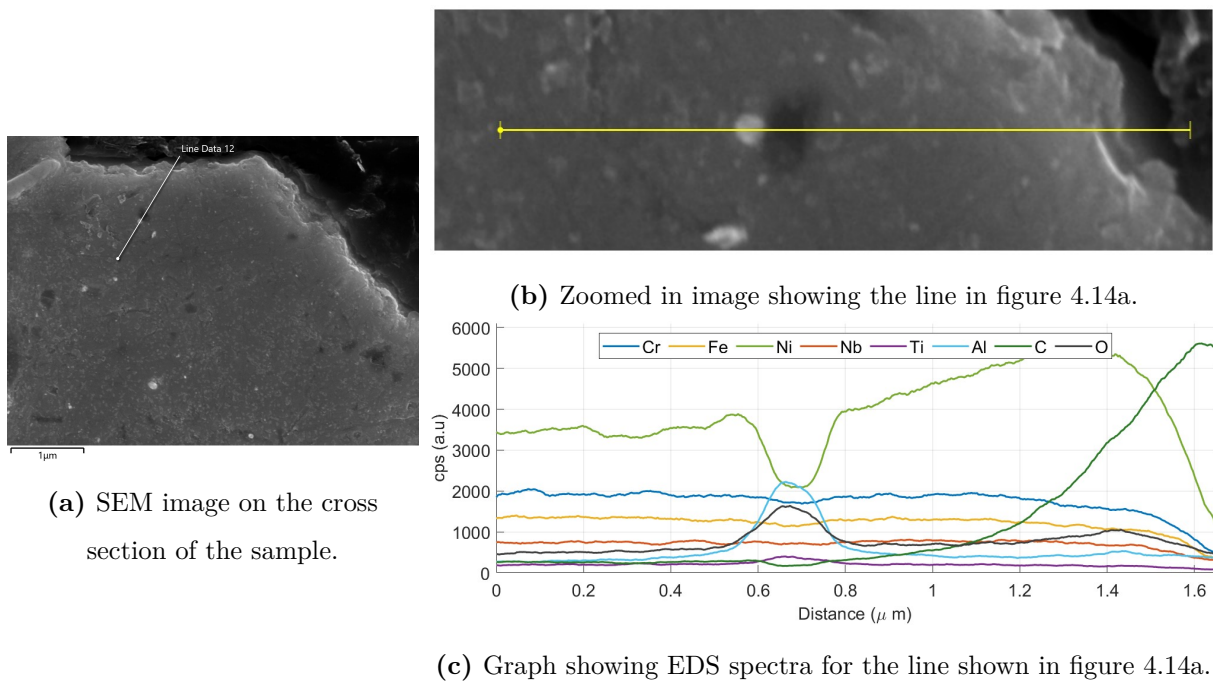


Figure 4.14: SEM-EDS image and spectra of the black oxide regions of a sample pickled in HCl and then etched.

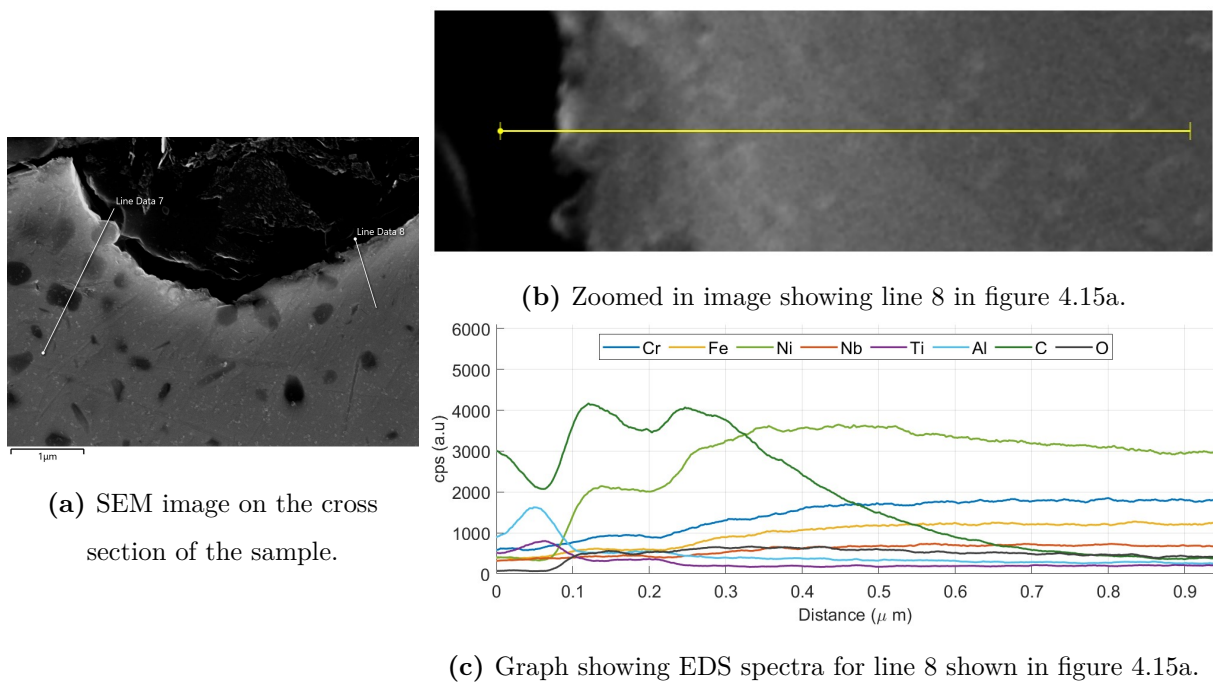


Figure 4.15: SEM-EDS image and spectra of the black oxide regions of a sample pickled in HF/HNO₃ and then etched.

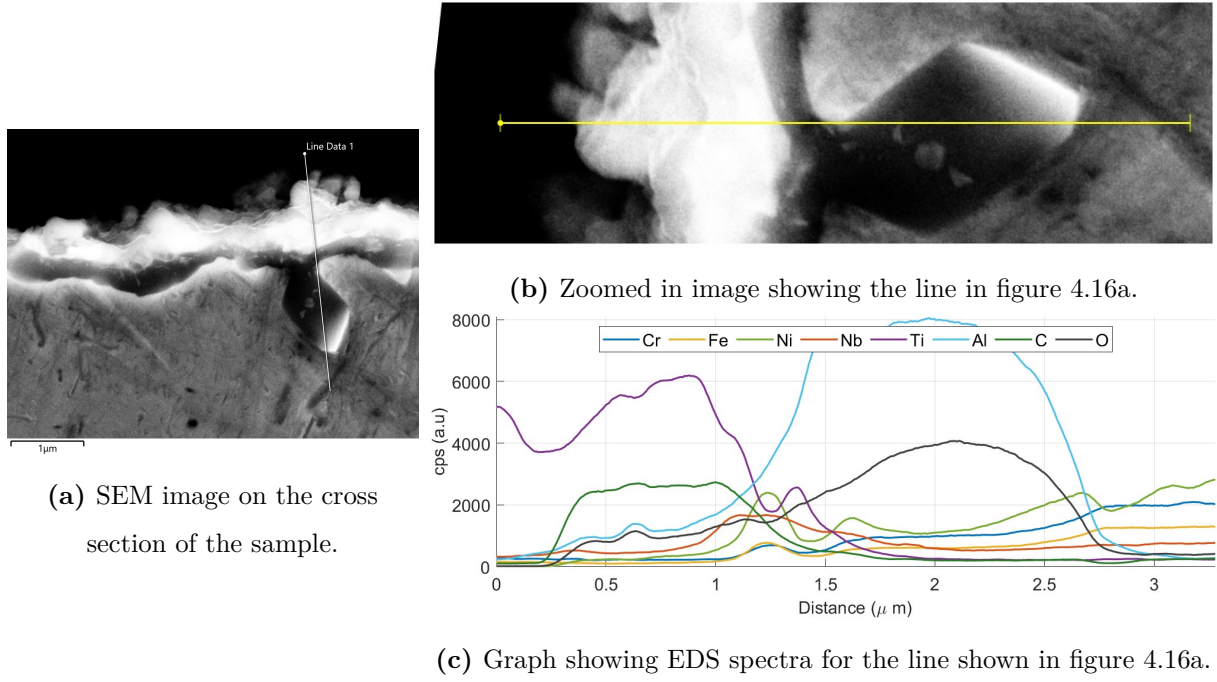


Figure 4.16: SEM-EDS image and spectra of a sample that has been HIP-treated in an optimal environment.

4.4 Surface Roughness

The surface roughness R_a and R_z for the samples in the different steps of the etching process can be seen in figures 4.17 and 4.18. The values used to make the graphs can be found in appendix F. The figures show that there is little to no variation in surface roughness between the different pickling solutions. The surface roughness tends to decrease during the pickling but for the etching some samples remain the same while others increase and some decrease. The surface roughness decreasing from pickling shows that at least some material is removed in this stage. This is important to note as it could not be seen in the microscope images.

One of the samples pickled in HCl shows a large increase in surface roughness in the pickling solution leading to a high mean and a large standard deviation, which then return to approximately the initial value after etching. The trend that the surface roughness decreases during pickling and then increases during etching could be due to the etching being inconsistent over the whole surface. In the pickling solution the high peaks are removed while in the etching the peaks are already removed and the change in surface

roughness could come from the abundance of near surface defects making some parts of the material easier to remove. Another cause for the uneven etching could be that the oxide layer is thinner at some places leading to it being easier to remove.

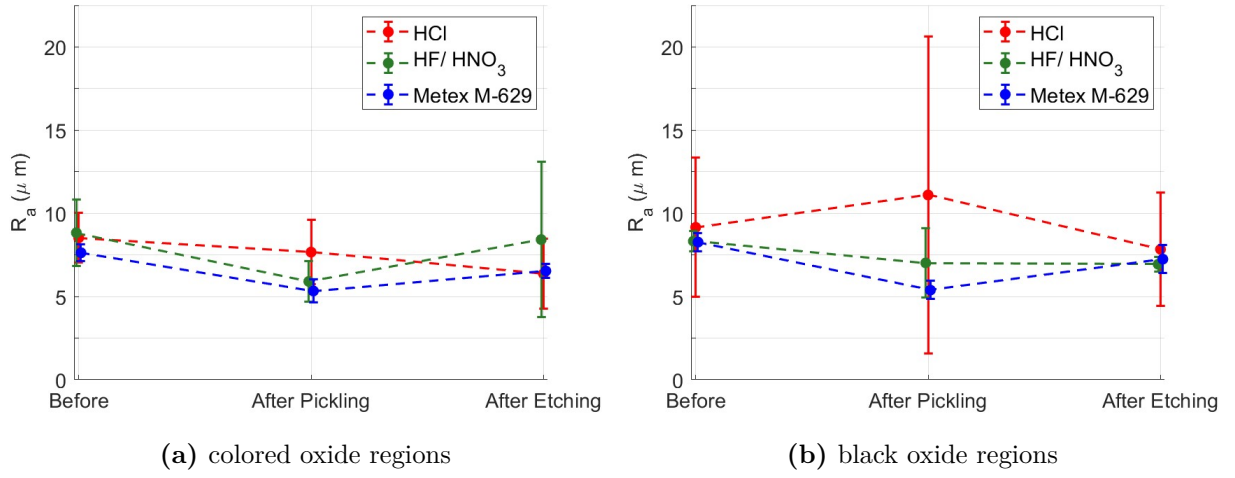


Figure 4.17: The mean and standard deviation of surface roughness (R_a) on a) the colored oxide regions and b) the black oxide for the samples pickled in HCl, HF/HNO₃ or Metex M-629 and then etched.

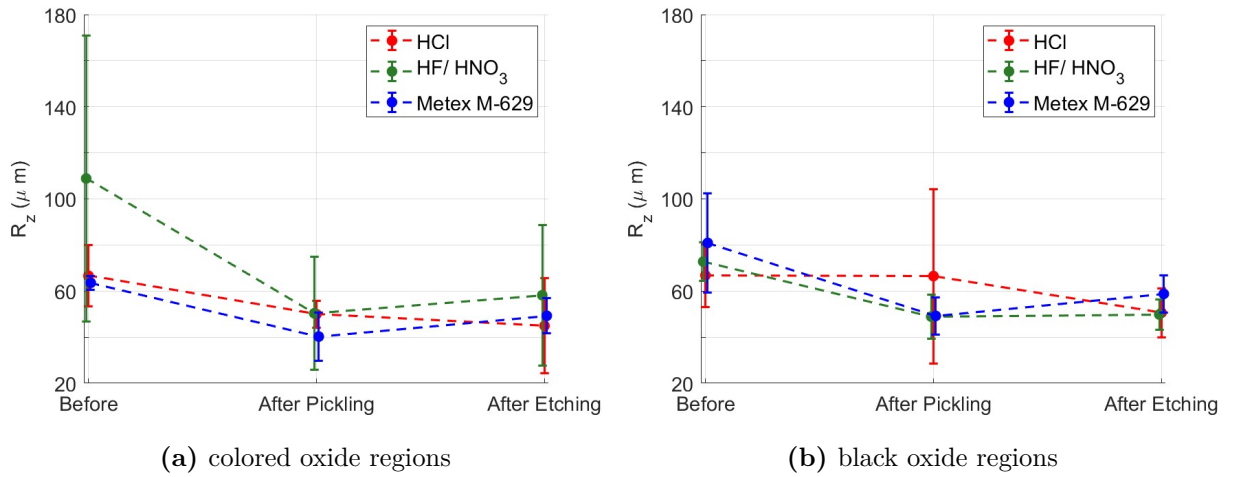


Figure 4.18: The mean and standard deviation of surface roughness (R_z) on a) the colored oxide regions and b) the black oxide for the samples pickled in HCl, HF/HNO₃ or Metex M-629 and then etched.

5. Conclusion

This work shows that there is no real difference in the end result when using the three different pickling solutions because the subsequent etching step effectively removes the surface oxides under the tested conditions. When looking at just the pickling the HF/HNO₃ solution performs the best as it removes the outermost layer of the black oxides and shows some effect on the colored oxide regions. The HCl solution does not show any noticeable effect on the surface oxides and appears less suitable under the tested conditions. The Metex M-629 solution shows some improvement on the colored oxides but has no noticeable effect on the black oxides.

The bulk material is confirmed to consist mostly of nickel, chromium and iron. The colored oxide regions are mostly aluminum containing oxides with a little titanium containing oxide while the black oxide regions are aluminum containing oxide with titanium containing oxides with a niobium rich layer on top.

Although all of the pickling solutions lead to complete surface oxide removal after the etching the surface roughness during the visual inspection suggests that the surface becomes rougher after etching. This is however not shown in the R_a and R_z measurements.

5.1 Future Work

To continue this work there are a number of different things that could be looked at. Other pickling solutions could be tested to see if there are more effective solutions for removing oxides. Another interesting parameter could be to increase the time in the pickling solution to see if the oxides are removed more easily when the time is increased. It would also be interesting to see what happens when a sample is only etched and not pickled.

To gain more knowledge about how the oxides on the sample surface are formed it would be good to look at the environment and other parameters in the hot isostatic pressing.

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A. Hot Isostatic Pressing Chamber

In this appendix there are images showing how the samples were placed in the hot isostatic pressing chamber. These images shows where the discolorations come from.



(a) Before HIP



(b) After HIP

Figure A.1: Images showing how the samples was placed in the hot isostatic pressing. The samples used look yellow/ gold in the pictures before HIP and black/ gray after HIP.

B. Laser Microscope Images

This appendix contains images of the samples front sides both before and after treatment. The images were taken using a laser microscope.

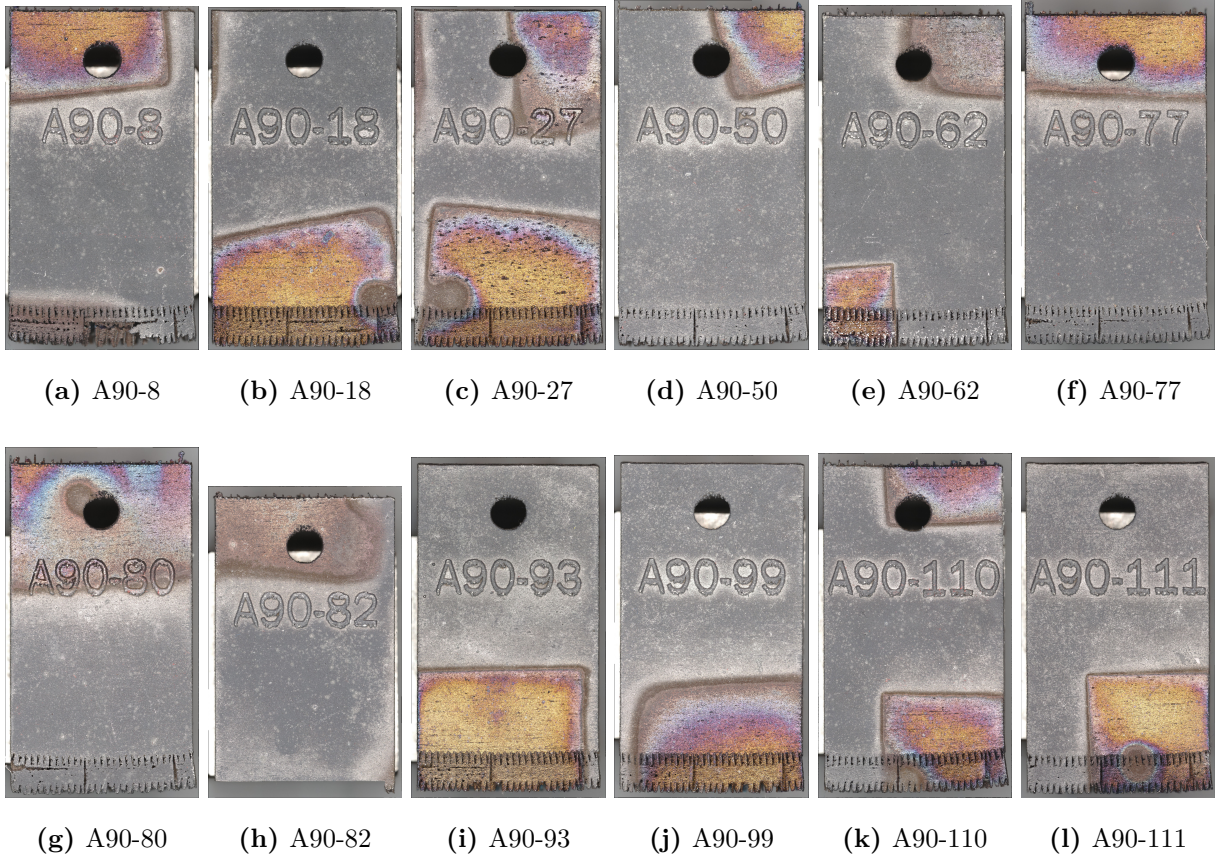


Figure B.1: Images of the front side of the samples after they have been HIP-treated. The images were taken using a laser microscope

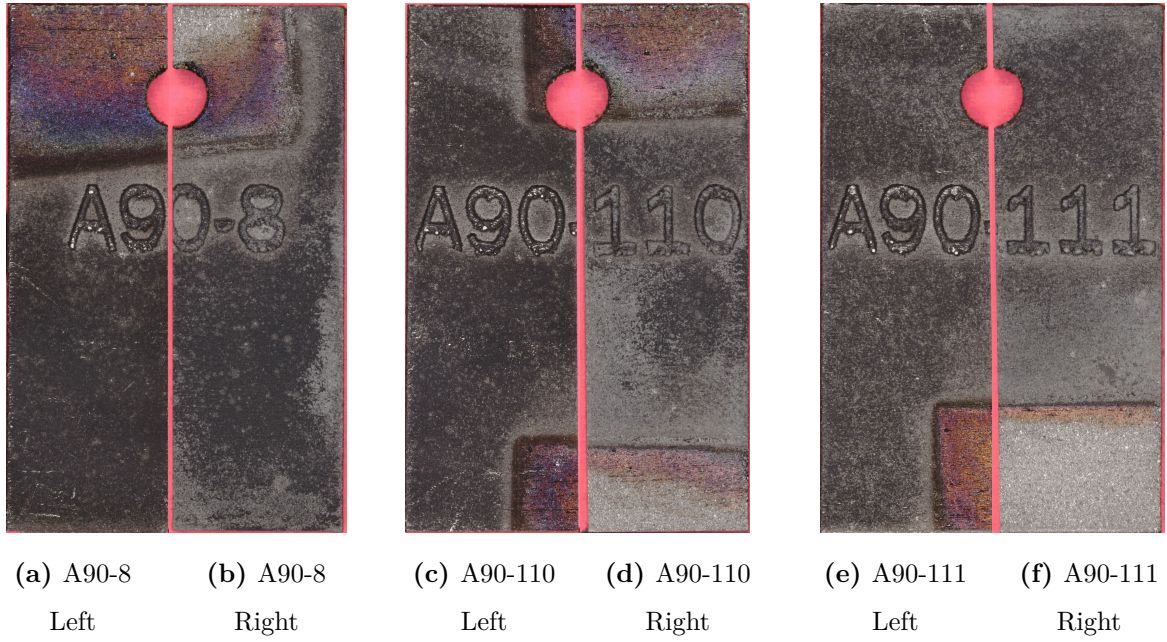


Figure B.2: Images of samples A90-8, A90-110 and A90-111 where the left side has been pickled in HCl and the right side has been pickled in HCl and etched.

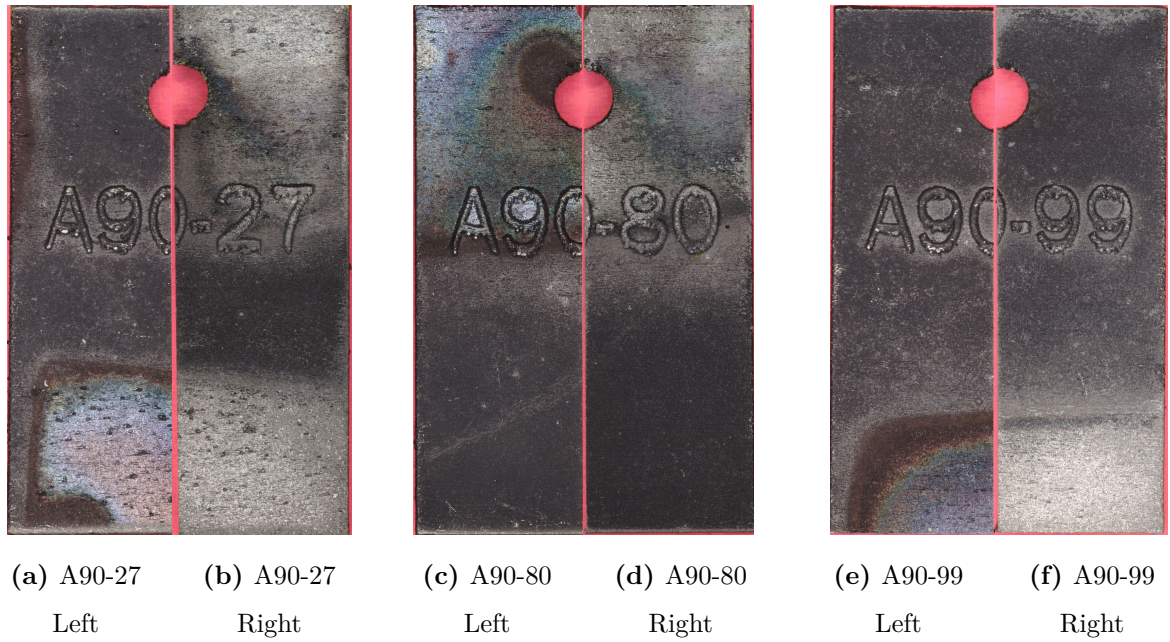


Figure B.3: Images of samples A90-27, A90-80 and A90-99 where the left side has been pickled in HF/HNO₃ and the right side has been pickled in HF/HNO₃ and etched.

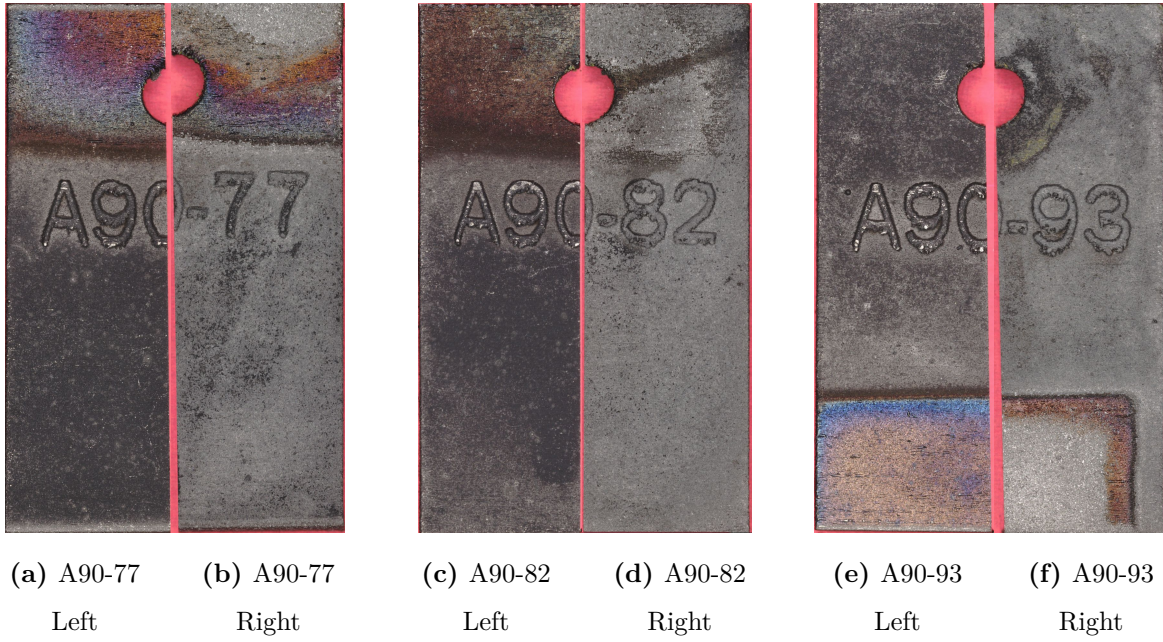


Figure B.4: Picture of sample A90-77, A90-82 and A90-93 where the left side has been pickled in Metex M-629 and the right side has been pickled in Metex M-629 and etched.

C. Sample Treatment

This appendix contains the breakdown of how the samples have been treated in this project.

Table C.1: Breakdown of the treatment on the samples used in this project. All samples that have been pickled have also been HIP-treated. The E56 sample has been HIP-treated in a contaminant free environment.

Pickling solution	Treatment	Sample name		
Non	HIP	A90-18	A90-50	A90-62
HCl	Pickled	A90-8 V	A90-110 V	A90-111 V
	Pickled & Etched	A90-8 H	A90-110 H	A90-111 H
HF/HNO ₃	Pickled	A90-27 V	A90-80 V	A90-99 V
	Pickled & Etched	A90-27 H	A90-80 H	A90-99 H
Metex	Pickled	A90-77 V	A90-82 V	A90-93 V
	Pickled & Etched	A90-77 H	A90-82 H	A90-93 H
Non	HIP	E56		

D. Optical Microscope Images

This appendix contains optical microscope images of all the samples used in this project.

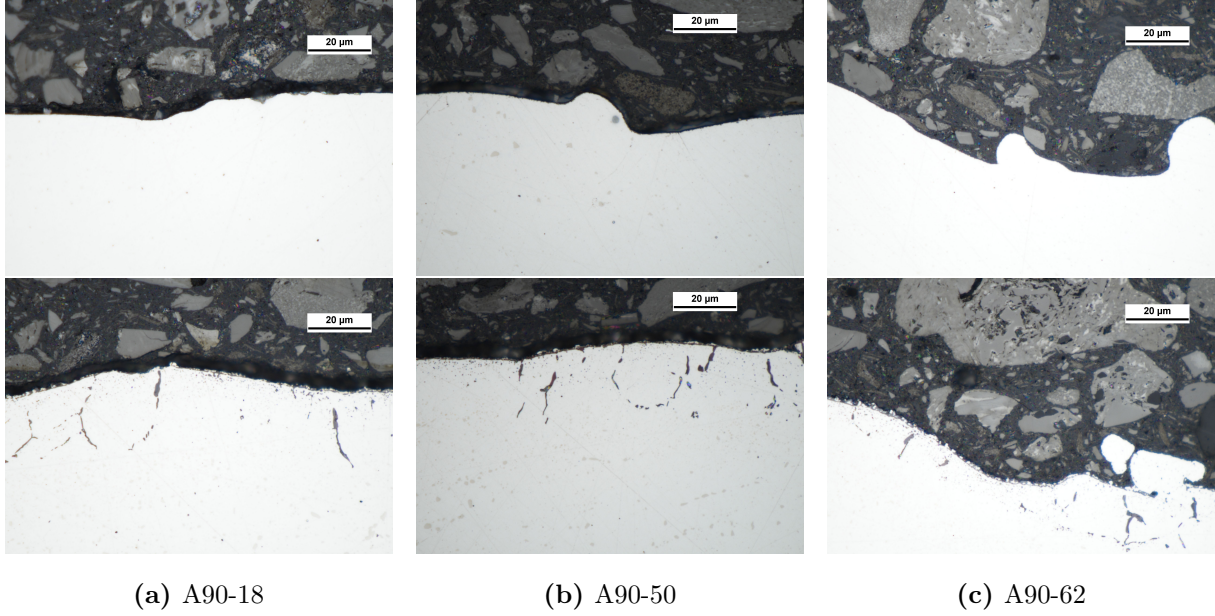


Figure D.1: Optical microscope images of the cross section at the colored oxide regions (top) and black oxide regions (bottom) of reference samples (only HIP-treated).

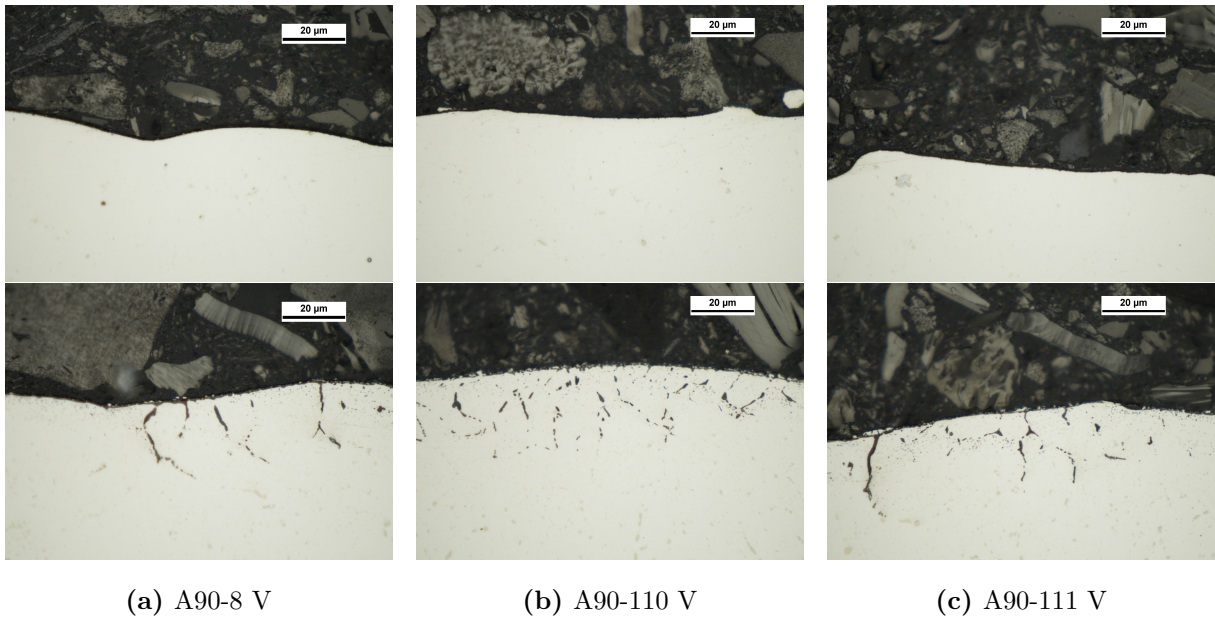
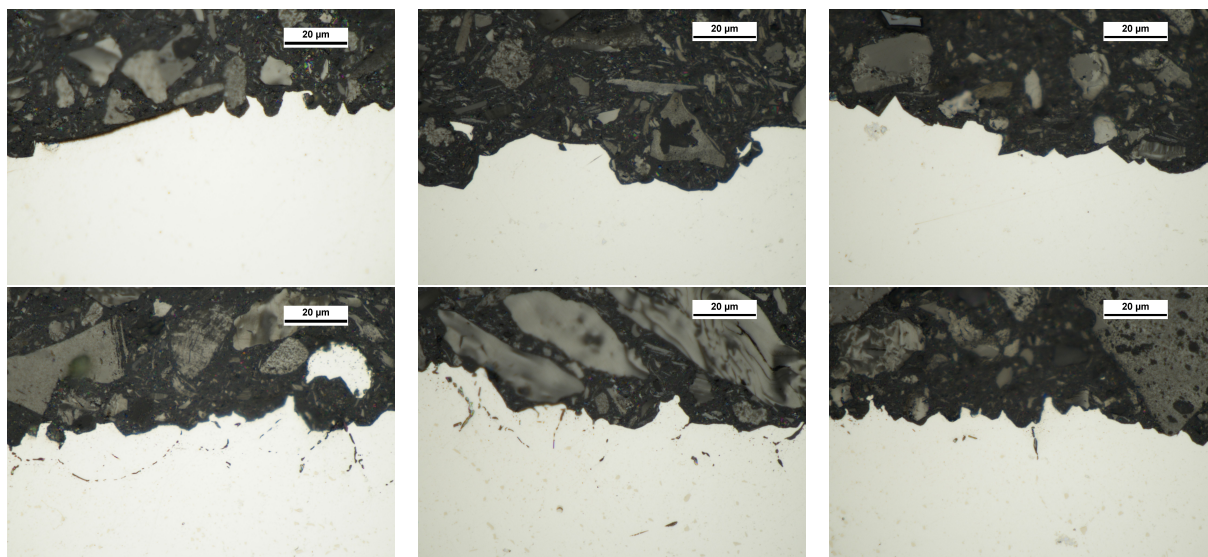


Figure D.2: Optical microscope images of the cross section at the colored oxide regions (top) and black oxide regions (bottom) of samples pickled in HCl.

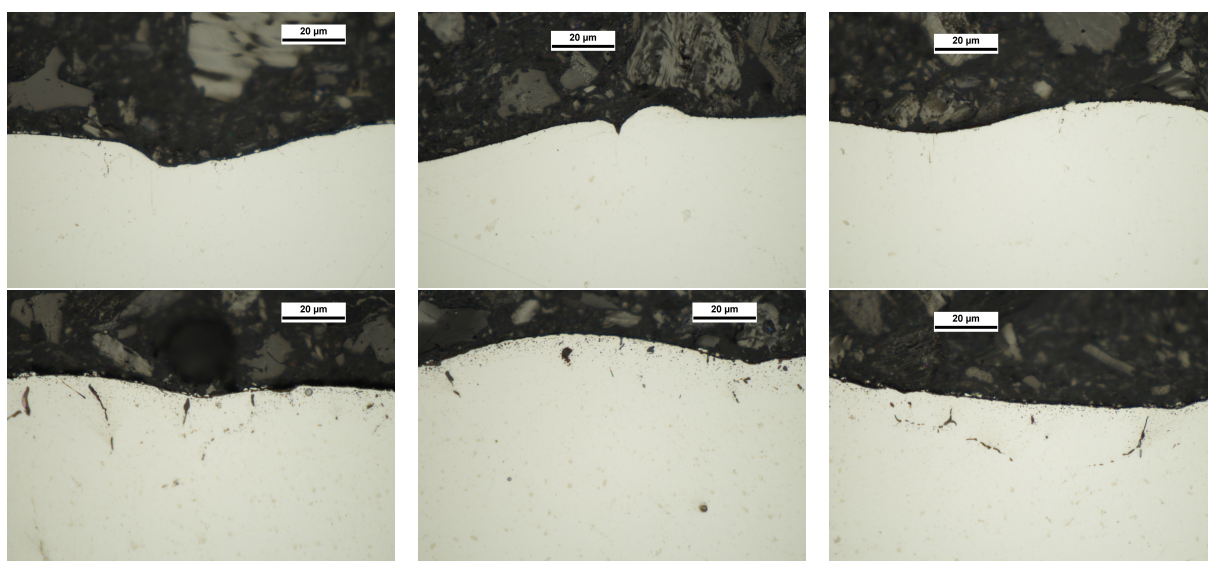


(a) A90-8 H

(b) A90-110 H

(c) A90-111 H

Figure D.3: Optical microscope images of the cross section at the colored oxide regions (top) and black oxide regions (bottom) of samples pickled in HCl and then etched.



(a) A90-27 V

(b) A90-80 V

(c) A90-99 V

Figure D.4: Optical microscope images of the cross section at the colored oxide regions (top) and black oxide regions (bottom) of samples pickled in HF/HNO₃.

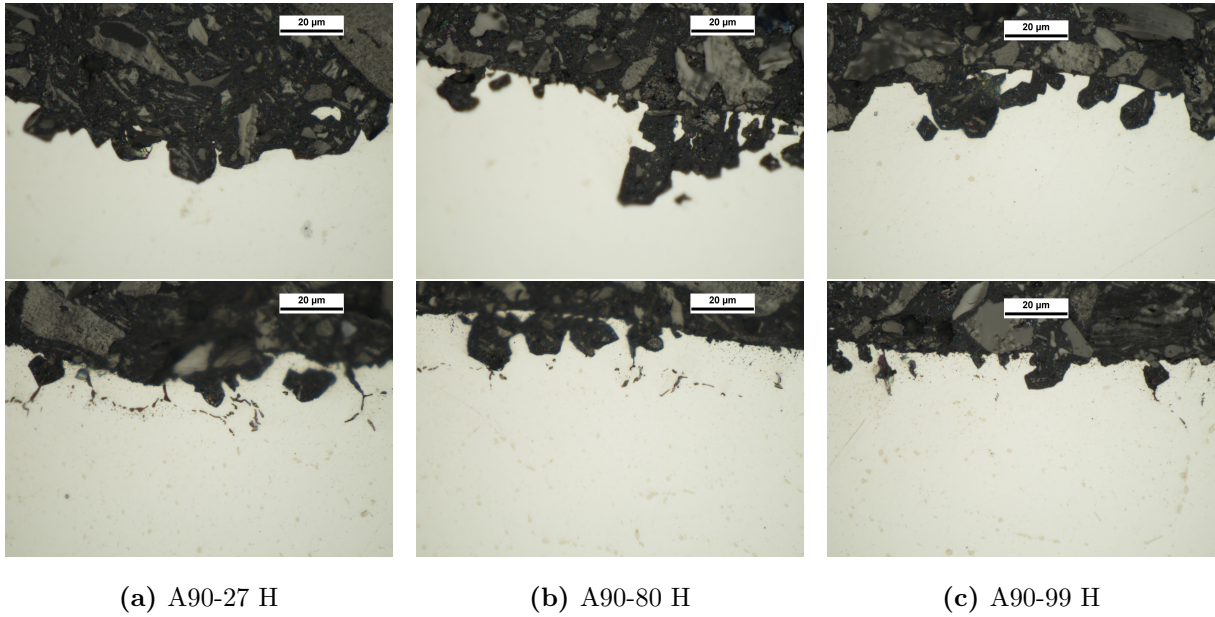


Figure D.5: Optical microscope images of the cross section at the colored oxide regions (top) and black oxide regions (bottom) of samples pickled in HF/HNO₃ and then etched.

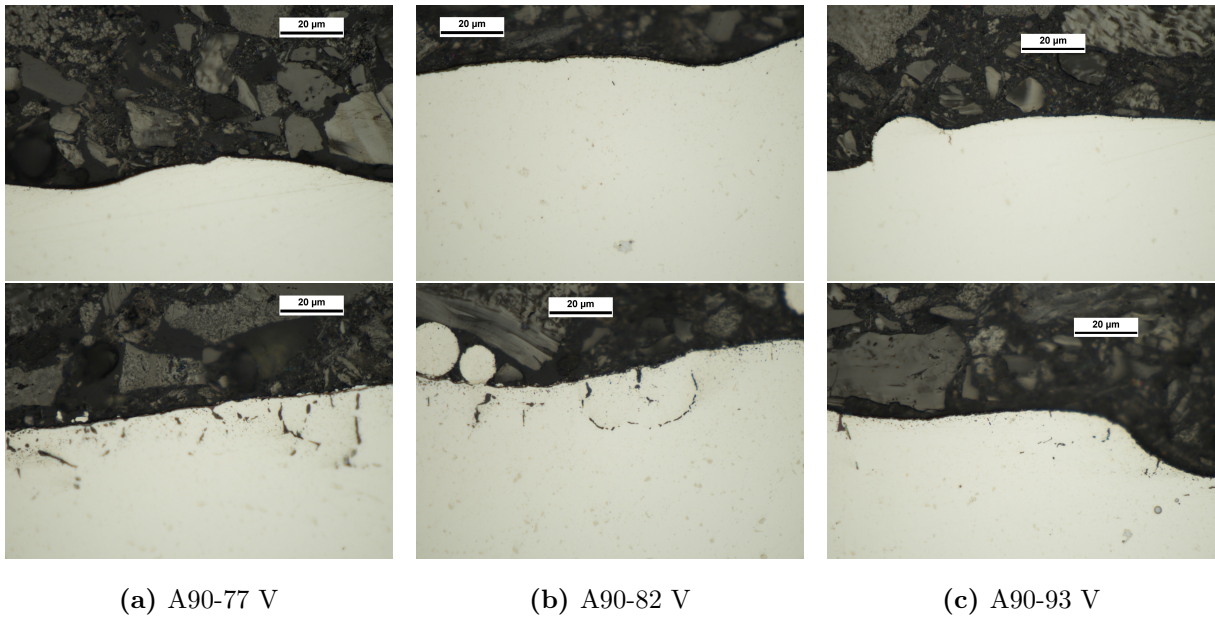
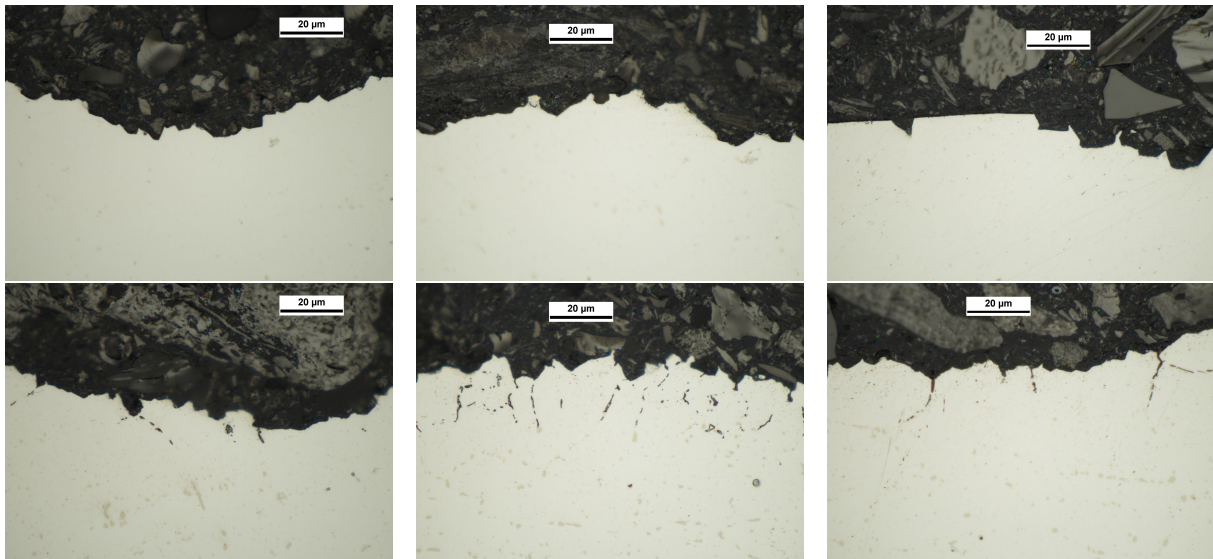


Figure D.6: Optical microscope images of the cross section at the colored oxide regions (top) and black oxide regions (bottom) of samples pickled in Metex M-629.



(a) A90-77 H

(b) A90-82 H

(c) A90-93 H

Figure D.7: Optical microscope images of the cross section at the colored oxide regions (top) and black oxide regions (bottom) of samples pickled in Metex M-629 and then etched.

E. Energy Dispersive X-ray Spectroscopy

This appendix contains more SEM-EDS analysis of the oxides on the testing material.

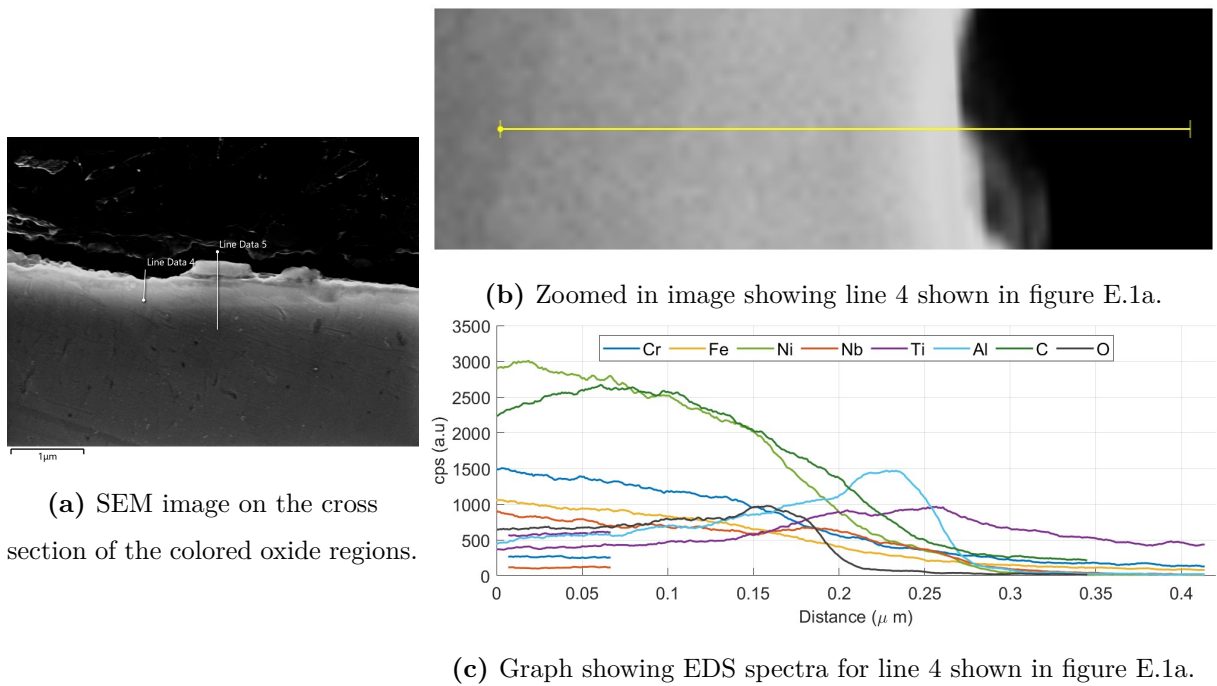


Figure E.1: SEM-EDS image and spectra of the colored oxide regions of a reference sample.

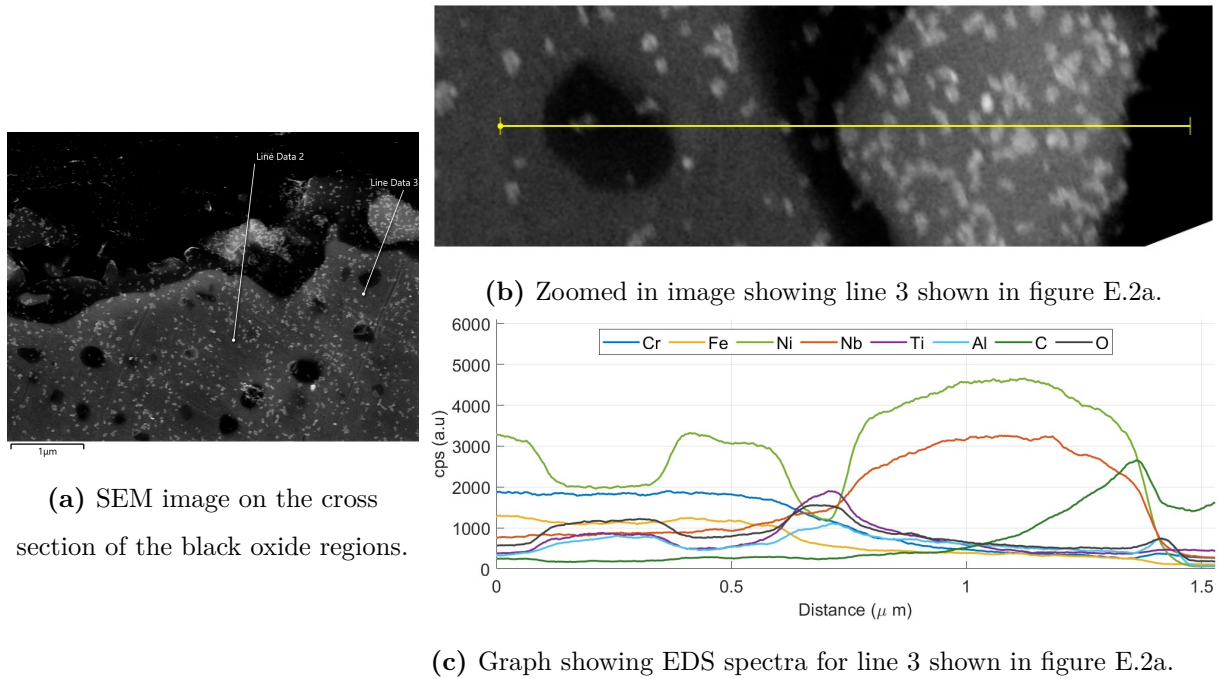


Figure E.2: SEM-EDS image and spectra of the black oxide regions of a reference sample.

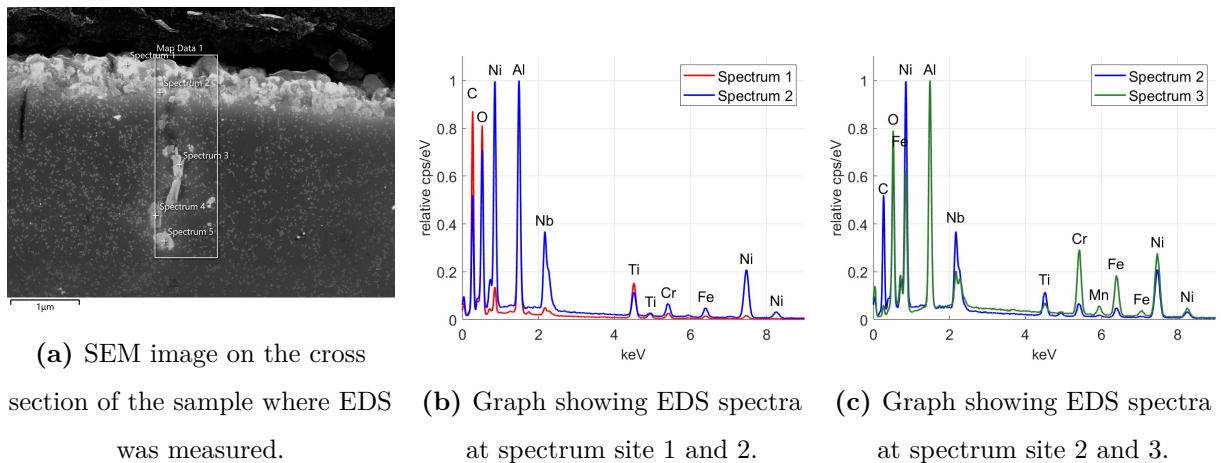


Figure E.3: SEM-EDS image and spectra of the black oxide regions of a sample pickled in Metex M-629.

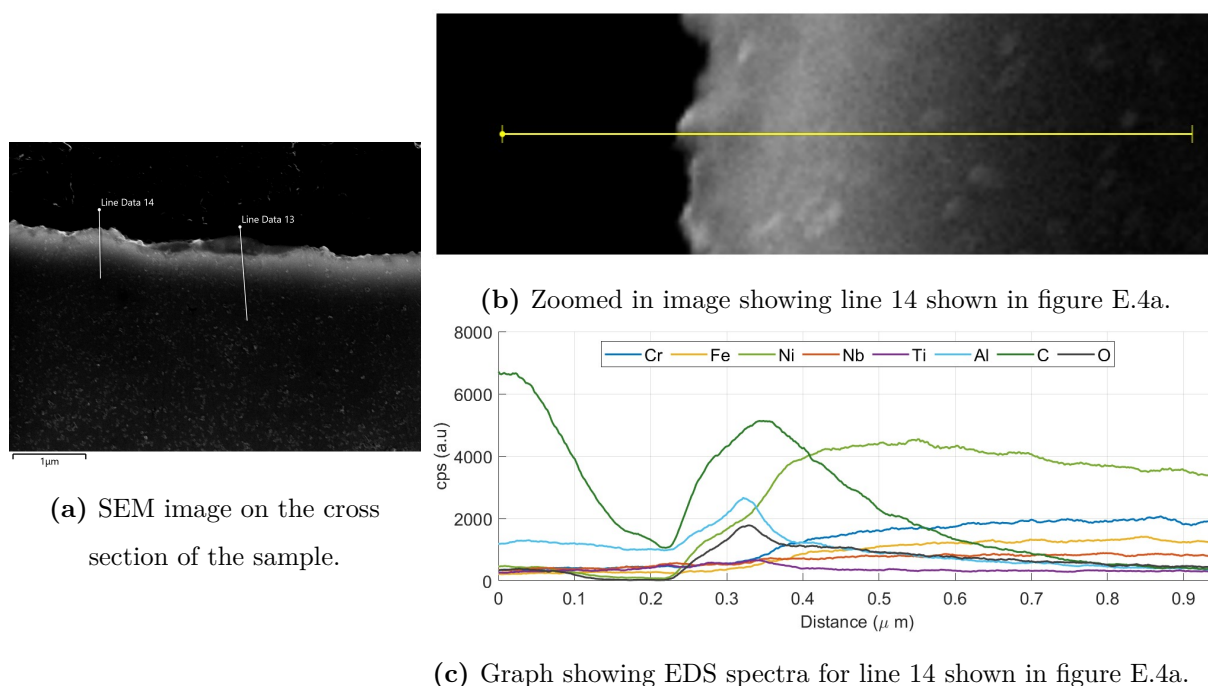


Figure E.4: SEM-EDS image and spectra of the colored oxide regions of a sample pickled in HCl.

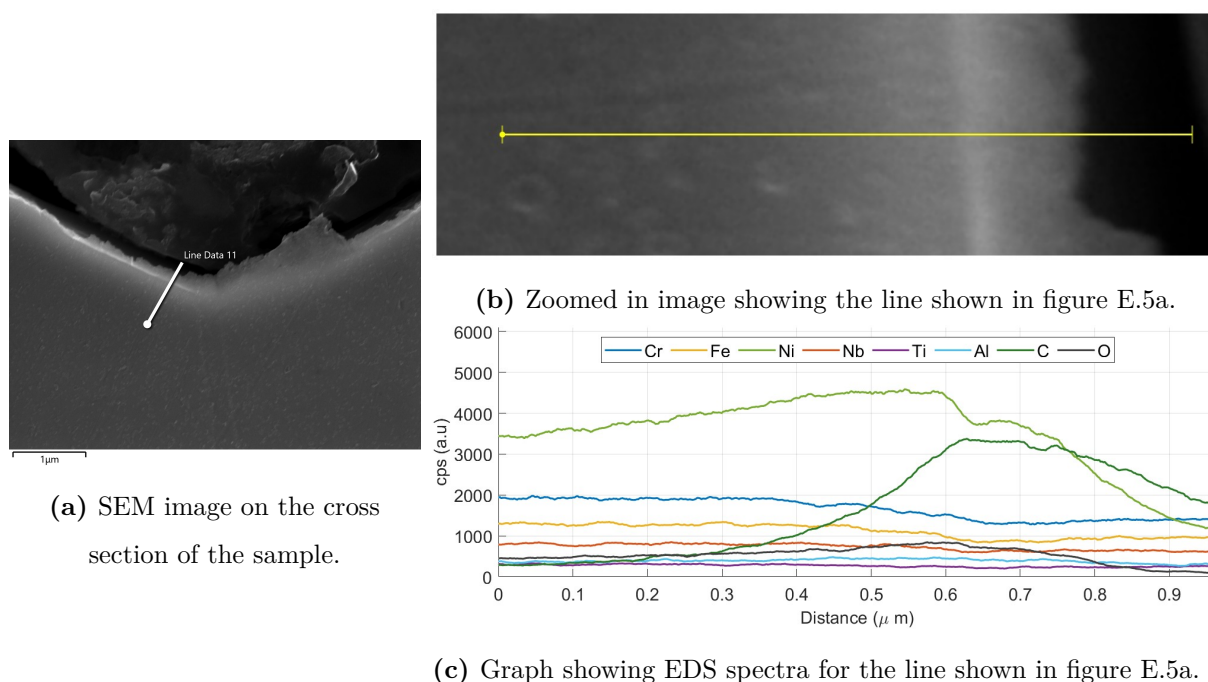


Figure E.5: SEM-EDS image and spectra of the colored oxide regions of a sample pickled in HCl and then etched.

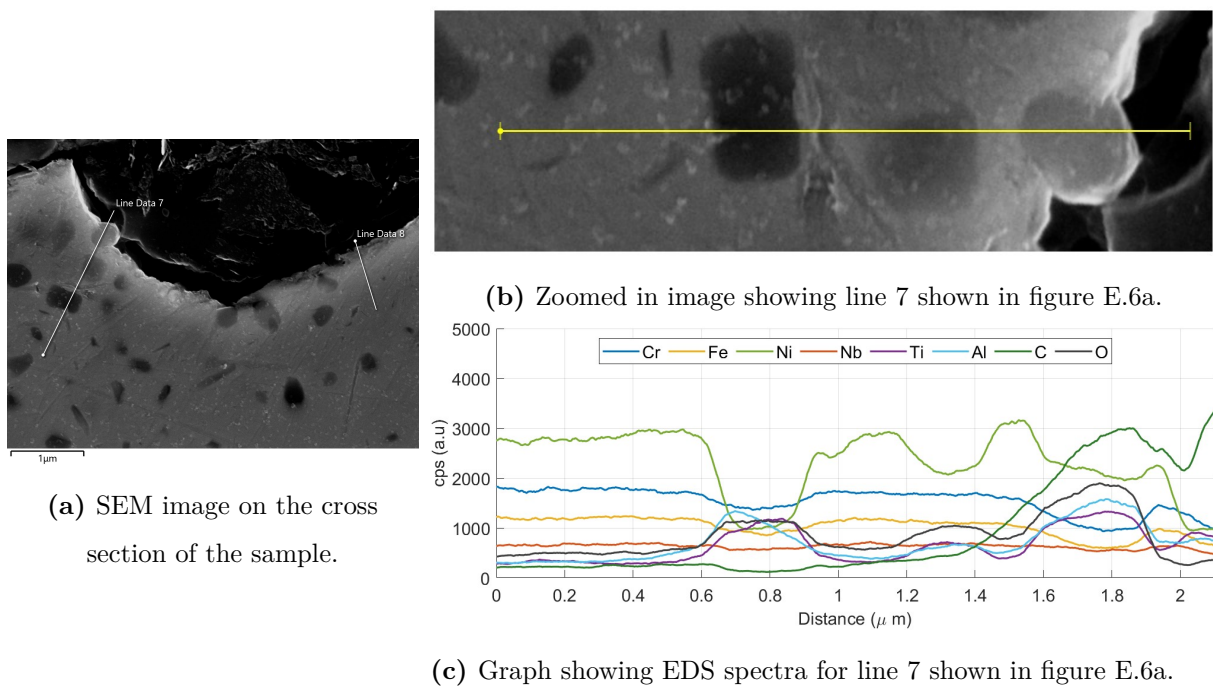


Figure E.6: SEM-EDS image and spectra of the black oxide regions of a sample pickled in HF/HNO₃ and then etched.

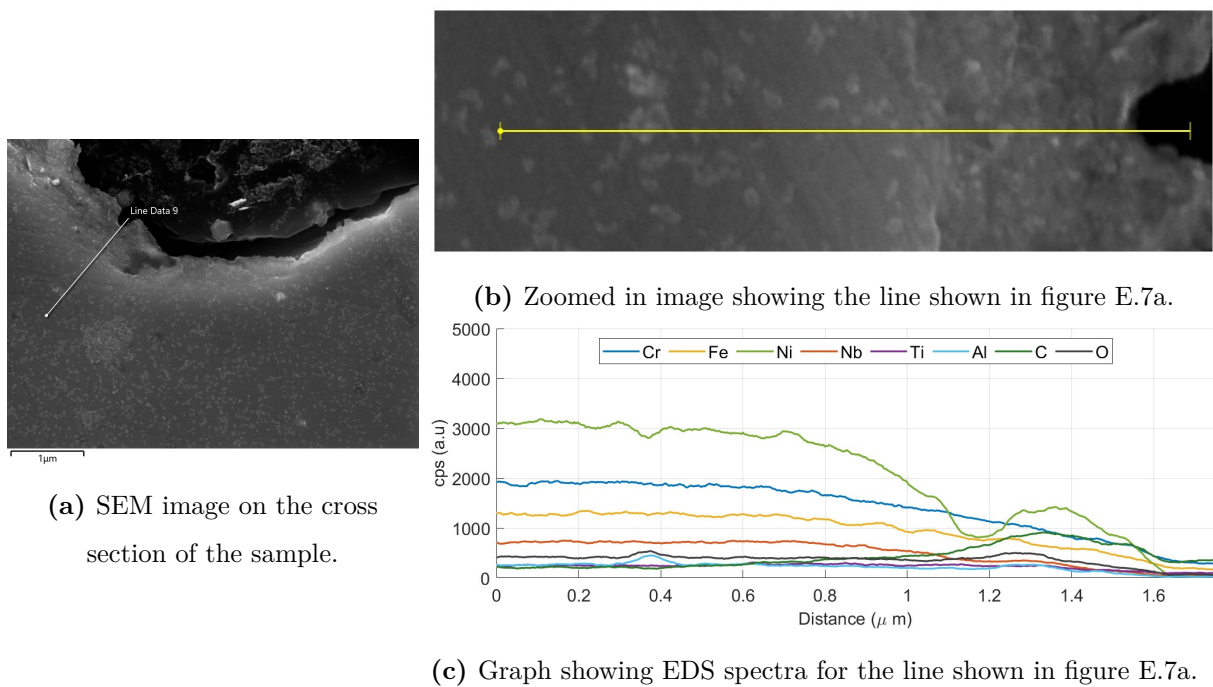


Figure E.7: SEM-EDS image and spectra of the colored oxide regions of a sample pickled in Metex M-629 and then etched.

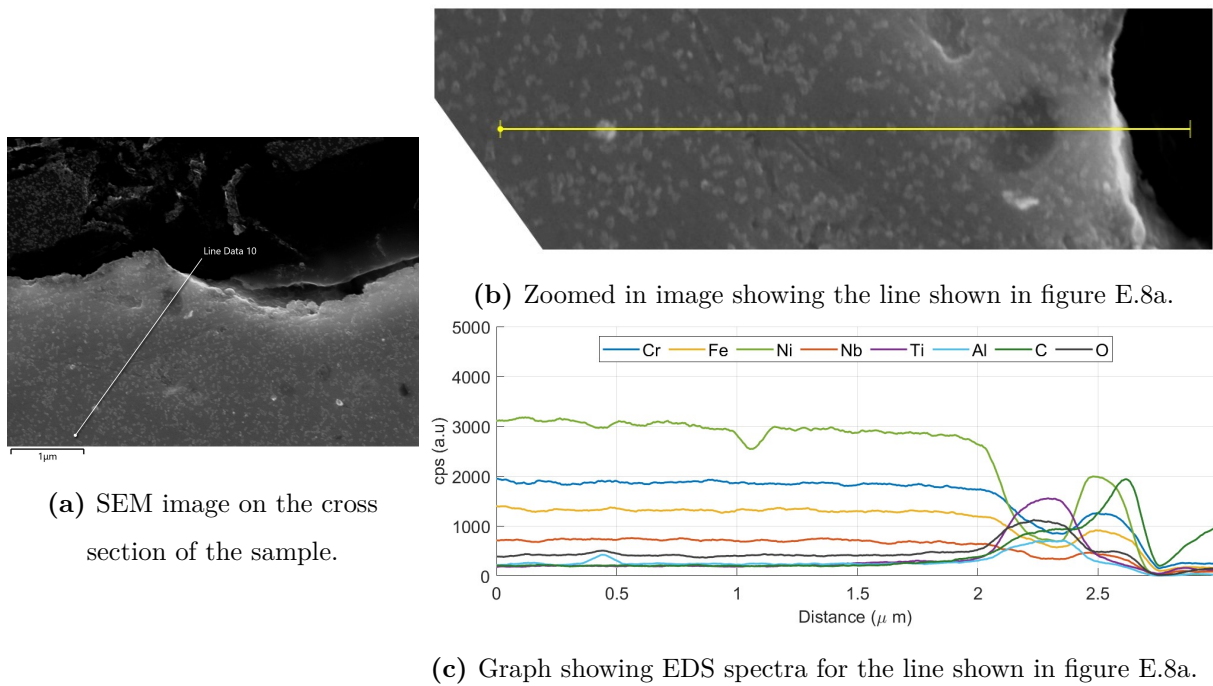


Figure E.8: SEM-EDS image and spectra of the black oxide regions of a sample pickled in Metex M-629 and then etched.

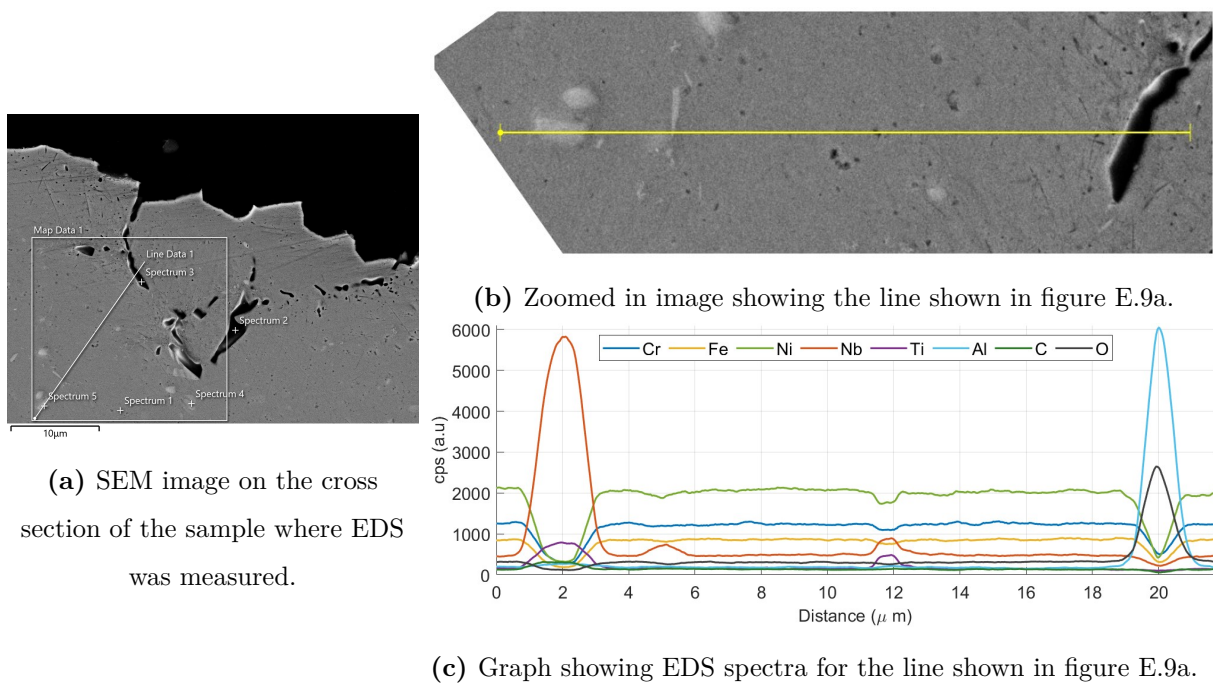


Figure E.9: SEM-EDS image and spectra on the black oxide regions of a sample pickled in Metex M-629 and etched.

F. Surface Roughness

This appendix contains the measured values for R_a and R_z on all the pickled and etched samples.

Table F.1: Measured surface roughness R_a on both the colored oxides and the black oxides on the samples.

Pickling Solution	Sample	Before		After Pickling		After Etching	
		Color	Black	Color	Black	Color	Black
HCl	A90-8	6.812	6.899	5.54	5.715	4.513	6.389
	A90-110	9.155	6.592	8.11	5.504	5.949	5.385
	A90-111	9.612	13.978	9.358	22.097	8.637	11.735
HF/ HNO ₃	A90-27	11.113	7.648	7.081	7.37	13.787	6.546
	A90-80	8.087	8.573	4.613	4.781	5.290	6.890
	A90-99	7.295	8.767	6.027	8.912	6.185	7.388
Metex M-629	A90-77	7.817	8.799	5.307	4.796	7.009	7.18
	A90-82	7.056	8.308	4.646	5.486	6.286	6.439
	A90-93	8.022	7.703	6.046	5.879	6.315	8.121

Table F.2: Measured surface roughness R_z on both the colored oxides and the black oxides on the samples.

Pickling Solution	Sample	Before		After Pickling		After Etching	
		Color	Black	Color	Black	Color	Black
HCl	A90-8	58.143	61.619	45.372	51.585	30.570	51.093
	A90-110	59.670	56.316	48.032	38.404	35.672	39.771
	A90-111	82.154	82.535	56.423	109.512	68.55	60.940
HF/ HNO ₃	A90-27	178.670	63.371	78.711	55.873	93.499	42.372
	A90-80	88.068	75.624	35.186	37.885	40.315	52.377
	A90-99	59.849	79.348	37.087	52.962	40.795	54.789
Metex M-629	A90-77	63.803	88.688	51.817	50.749	55.359	51.488
	A90-82	60.450	97.365	31.500	56.404	51.679	57.465
	A90-93	66.527	56.635	37.437	40.529	40.770	67.397

G. Societal, Ethical and Ecological Aspects

For this project the most important ethical aspect is that the results of this report could be used in the manufacturing of materials for military applications. There are also smaller societal and environmental aspects with regard to using strong acids.

The issue is not that military research is not beneficial, it's that the possibility that the results are being used by the wrong militaries may be too great. There will always be innovations in the field of military industries and even though the intended use of the results is not in the military industries, they could still be used there. This is however also true for most of the research conducted in the field of material science.

The processes currently used utilize cast and forged goods that are then milled and turned into the finished part. This produces a lot of waste material. The project aims to make changes to a manufacturing process to deliver manufactured parts that use less natural resources than the processes uses today. This would greatly benefit the world (from an ecological standpoint) as there is a problem with the overuse of the world's resources.

The method cannot be changed without changing the objective of the project. That the results can be used for military applications cannot be changed with the method and the main goal of the project is to investigate how different etchants affect the oxide scales. If the strong acids were exchanged for another chemical the project would lose its meaning and the problem with using chemicals still exists. The strong acids that are used have to be used as they are the only ones that can be utilized in etching on materials like LPBF. Special consideration will instead be given to safe handling of the chemical and minimizing chemical use.

