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ELECTROLYTIC ETCHING: AN ALTERNATIVE METALLOGRAPHIC ETCHING METHOD WITH REDUCED CHEMICAL RISK

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Metallografisissa laboratorioissa näytteiden syövytys tehdään perinteisesti vahvoilla kemikaaleilla, jotka aiheuttavat riskejä työntekijöille ja ympäristölle. Tämän opinnäytetyön lähtökohtana oli kiinnostus ja halu parantaa turvallisuutta sekä kehittää menetelmiä, jotka vähentävät vaarallisten kemikaalien käyttöä. Työn tavoitteena oli selvittää, voidaanko elektrolyyttistä syövytystä käyttää vaihtoehtona kemialliselle syövytykselle, mitä muita tapoja voidaan käyttää parantamaan kemikaalien käsittelyä metallografisessa laboratoriossa sekä löytää kehityskohteita vaiheista ennen syövytystä.

Tutkimuksen teoreettinen viitekehys käsittelee metallografisen näytteenvalmistuksen vaiheita, elektrolyyttisen syövytyksen periaatteita ja hyötyjä sekä kemikaaliturvallisuutta ja jatkuvan parantamisen menetelmiä. Menetelminä käytettiin kokeellista testausta neljällä materiaalilla (kaksi terästä ja kaksi nikkelipohjaista superseosta) sekä työpaikkahavainnointia kemikaalien käsittelykäytännöistä. Elektrolyyttisessä syövytyksessä käytettiin 10 % oksaalihappoliuosta ja Struers LectroPol-5 -laitetta.

Tulokset osoittivat, että elektrolyyttinen syövytys voi parantaa turvallisuutta ja vähentää vaarallisten kemikaalien käyttöä, mutta se ei täysin korvaa perinteistä kemiallista syövytystä. Molempia menetelmiä tarvitaan parhaan mikrorakenteen näkyvyyden saavuttamiseksi. Lisäksi ehdotettiin parannuksia syövytteiden merkitsemiseen, neutralointiin ja syövytysprosessin dokumentointiin sekä kehitettiin uusi näytteenpidin, joka nopeuttaa venttiilinäytteiden valmistusta.

Avainsanat metallografia, kemikaaliturvallisuus, materiaalitekniikka, laboratorio, jatkuva parantaminen

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ABSTRACT

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In metallographic laboratories, etching of samples is traditionally performed using strong chemical solutions that pose risks to both personnel and the environment. This thesis was motivated by an interest and willingness to improve safety and develop methods that reduce the use of hazardous chemicals. The aim was to examine whether electrolytic etching can serve as an alternative to chemical etching, identify other ways to improve chemical handling in metallographic laboratories, and find development opportunities in the steps before etching.

The theoretical framework covers metallographic sample preparation stages, principles and benefits of electrolytic etching, chemical safety, and continuous improvement methods. The research methods included experimental testing on four materials (two steels and two nickel-based superalloys) and workplace observation of chemical handling practices. Electrolytic etching was carried out using a 10% oxalic acid solution and a Struers LectroPol-5 unit.

The results showed that electrolytic etching can improve safety and reduce the use of hazardous chemicals, but it cannot completely replace traditional chemical etching. Both methods are needed to achieve optimal microstructural visibility. Additional improvements were proposed for labeling etchants, neutralization procedures, and documentation of the etching process. Furthermore, a new sample holder was developed to speed up the preparation of valve samples.

Keywords metallography, chemical safety, materials engineering, laboratory, continuous improvement

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ABBREVIATIONS AND TERMS

ASTM	American Society for Testing and Materials, international standards organization
CLP	Classification, Labelling and Packaging of Substances and Mixtures, EU chemical classification regulation
EDS	Energy Dispersive Spectroscopy, method for elemental analysis
HV10	Vickers hardness test using 10 kg load
ISO	International Organization for Standardization, global standardization body
LMD	Laser Metal Deposition, additive manufacturing process using laser
Nital	mixture of nitric acid and ethanol, used for metallographic etching
OES	Optical Emission Spectroscopy, method for elemental analysis
PPE	Personal Protective Equipment, safety gear for laboratory work
REACH	Registration, Evaluation, Authorization and Restriction of Chemicals, EU chemical regulation
SEM	Scanning Electron Microscope, high-resolution imaging instrument
WAAM	Wire Arc Additive Manufacturing, additive manufacturing process using electric arc

1 INTRODUCTION

1.1 Background and motivation

In metallographic laboratories, samples of metals and alloys are prepared so that the microstructure can be examined under a microscope. One important step in this process is etching, which makes the grain structure visible (Vander Voort, 1999). Normally this is done with strong chemical solutions that can be dangerous for both people and the environment if not handled accordingly (Sigma-Aldrich, 2022a; Vander Voort, 1999). Because safety rules and environmental demands are only becoming stricter now and -in the future, therefore it is important to look for safer alternatives for chemical handling in the laboratory (European Chemicals Agency, 2023).

1.2 Chemical safety in metallographic laboratories

Traditional etching methods often use acids such as nitric acid or hydrochloric acid, or mixtures like Nital. These substances are corrosive and can cause harm if they are not handled correctly (Sigma-Aldrich, 2022a). Disposal of these chemicals is also a problem, since they count as hazardous waste. Because of that, laboratories must make sure that employees work safely and that all chemical handling and waste disposal follow environmental laws and regulations (European Chemicals Agency, 2023). One possible alternative is to swap chemical etching for electrolytic etching, which uses electricity together with milder solutions. This method could improve safety and reduce the need for hazardous chemicals (Struers, 2025a).

1.3 Role of Wärtsilä and description of the commissioning project

This thesis is done for Wärtsilä. Wärtsilä is a large international company that provides technology and services for the marine and energy industries. The company focuses strongly on innovation, efficiency, and environmental responsibility (Wärtsilä, 2025).

The thesis project takes place in the materials laboratory at Wärtsilä's Sustainable Technology Hub in Vaasa. In this laboratory, safer and more efficient ways of handling chemicals could improve daily work and hence reduce risks. The aim is to test whether electrolytic etching could be used instead of traditional chemical etching, and to explore other possible improvements in the sample preparation steps before etching. In addition, the current methods for chemical handling and disposal will be examined, and safer and more efficient alternatives will be suggested where possible.

1.4 Scope and limitations of the research

This thesis focuses on metallographic sample preparation, with particular attention to etching and chemical handling. Other parts of metallography, such as microscopic analysis, are used for comparing and evaluating results. Special focus is placed on improving laboratory safety and identifying practical methods that could reduce the use of hazardous chemicals.

The research is done in Wärtsilä's material laboratory, and its main goal is to evaluate the practical suitability of electrolytic etching and how it can make the work safer and more sustainable. The study does not cover all aspects of metallography but concentrates on the processes where chemical safety and process efficiency can be improved.

1.5 Use of artificial intelligence in this thesis

In this thesis, I have used Microsoft Copilot and Wärtsilä GPT for ideation, information retrieval, and proofreading. I have edited the text multiple times to make it more understandable and pleasant to read. Figure 2 in chapter 3.2.1 is created with Copilot.

If AI has produced new ideas for the text, I have verified them using original sources and referred to them properly. All the references I have marked are actual sources that I have used and not AI produced. On this chapter 1.5, I have not used AI.

2 OBJECTIVES AND RESEARCH QUESTIONS

2.1 Main and sub-objectives

The main goal of this thesis is to find out if electrolytic etching can be used as a safer and more sustainable alternative to traditional chemical etching in metallographic laboratories.

The sub-objectives are:

- To compare electrolytic etching to traditional chemical etching in terms of safety, usability, and quality of results.
- To look at the current ways of handling and disposing chemicals and suggest safer and more practical methods.
- To find possible improvements in the sample preparation steps that happen before etching, for example mounting, grinding and polishing

As a result, the thesis will produce a documented comparison of the two etching methods, proposals for safer chemical handling and disposal, and recommendations for improving pre-etching sample preparation processes to enhance safety and efficiency.

2.2 Research questions

This thesis will try to answer the following questions:

- How does electrolytic etching compare to conventional chemical etching in terms of safety, quality, and practicality?
- What improvements can be made in chemical handling and disposal processes within the laboratory?
- How can earlier steps in the sample preparation chain be optimized to support safer and more consistent results?

3 THEORETICAL FRAMEWORK

3.1 Metallographic sample preparation steps

The preparation of metallographic samples includes several main stages: cutting, mounting, grinding, polishing, and etching. The purpose of cutting is to obtain a representative piece of the material without causing changes to its microstructure (Vander Voort, 1999). Mounting is used to make the sample easier to handle during later stages. This can be done either by hot mounting with thermosetting resins or by cold mounting with polymer resins, sometimes under vacuum so that pores and cavities are filled (ASTM International, 2011; Struers, 2025b).

Grinding is used to remove surface damage from cutting and to bring the sample to a flat surface. Polishing is then carried out using polishing cloths with suspension to achieve a mirror-like surface that is free of scratches. Each cloth is designed to work with a specific type of suspension, usually containing fine abrasive particles such as alumina or diamond. The right combination ensures efficient material removal while avoiding new scratches or surface deformation (Vander Voort, 1999). Figure 1 shows 2 of our sample preparation machines, on the left Citopress-20 for hot mounting and on the right Tegrapol-31 for grinding and polishing.

Figure 1 Citopress-20 and Tegrapol-31.

Only after polishing the sample is ready for etching, which reveals the microstructure under a microscope. Each step must be carried out carefully, because mistakes in early preparation usually cannot be corrected later. Proper preparation ensures accurate microstructural analysis and reliable results (Vander Voort, 1999).

3.2 Principles and benefits of electrolytic etching

3.2.1 Principles of electrolytic etching

Electrolytic etching is an electrochemical process where a direct current (DC) is applied to selectively dissolve a thin surface layer of a metal sample, revealing grain boundaries and phases (Vander Voort, 1999). The specimen acts as the anode (+), while an inert cathode (-) such as stainless steel or platinum is placed in the same electrolyte solution. When current flows through the electrolyte, metal atoms at the anode oxidize and dissolve into the solution in a controlled manner, exposing structural details without excessive material removal, while reduction reactions occur at the cathode (Struers, 2025a).

The main components of the setup are (Struers, 2025a):

- **Power supply**: provides constant or adjustable DC voltage.

- **Anode (sample):** the specimen to be etched.
- **Cathode:** a counter electrode that completes the circuit.
- **Electrolyte bath:** a chemical solution that conducts electricity.

Figure 2 illustrates the basic configuration of the electrolytic etching process. Oxalic acid is an example of electrolyte.

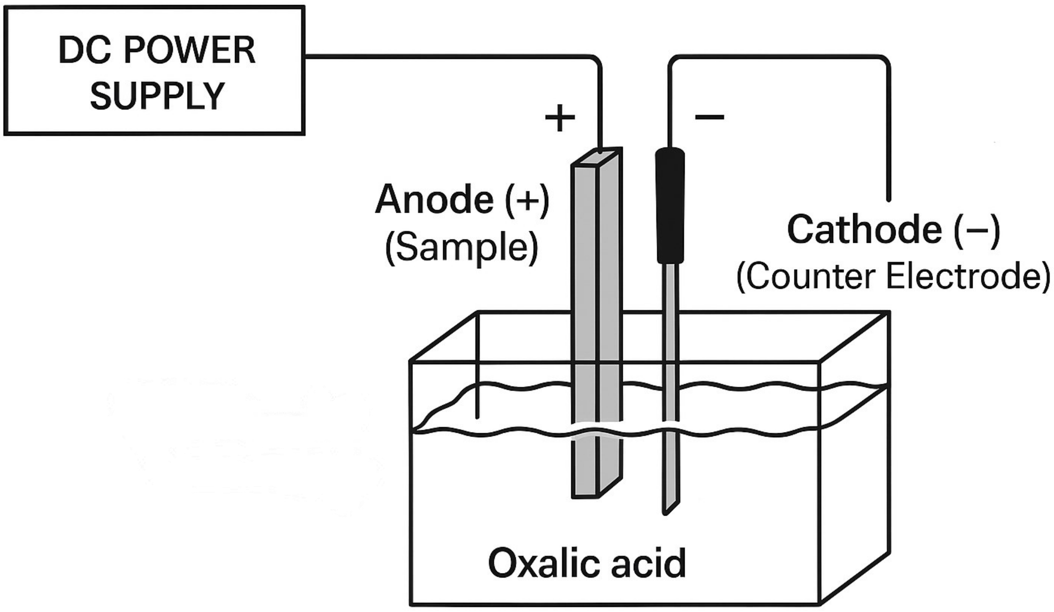

Figure 2 Basic setup for electrolytic etching process (Image created with Microsoft Copilot, 2025).

Electrochemical reactions:

Electrochemical reactions during electrolytic etching follow standard oxidation and reduction principles (Vander Voort, 1999; Bard & Faulkner, 2001):

- **At the anode (sample):**

$$\boldsymbol{M \rightarrow M^{n+} + ne^{-}} \quad (1)$$

Explanation:

M = metal atom

M^{n+} = metal cation (positive ion, n=charge)

e^- = electron

This is the oxidation reaction where metal atoms lose electrons and dissolve into the electrolyte.

- **At the cathode (counter electrode):**
  Option 1-Hydrogen evolution
  (common in acidic electrolytes)

$$2H^+ + 2e^- \rightarrow H_2 \uparrow \qquad (2)$$

Explanation

H^+ = hydrogen ion (proton)

e^- =electron

H_2 = hydrogen gas (released as bubbles)

↑ = gas formation

This reduction reaction produces hydrogen gas at the cathode. In weak acids such as oxalic acid, hydrogen evolution at the cathode is possible but limited due to low H^+ concentration.

Option 2 – Metal deposition (if metal ions are present)

$$M^n + ne^- \rightarrow M \qquad (3)$$

Explanation

M^{n+} = metal cation

e^- = electron

M = metal atom (deposited on cathode)

This reduction reaction occurs when metal ions in the electrolyte are reduced and deposit on the cathode.

This selective dissolution occurs at different rates depending on grain orientation and phase composition, making the microstructure visible under a microscope. Etching depth and quality depend on parameters such as voltage, current density, time, and temperature. By adjusting these variables, the operator can achieve precise control over the process and reproducible results. Once the desired surface structure is visible, the current is switched off, and the sample is rinsed and dried. Electrolytic etching can also be combined with polishing, making it a versatile method for metallographic preparation. Sample should be ground to at least 600 grit if electrolytic polishing is applied (Vander Voort, 1999).

3.2.2 Benefits and applications

Compared to conventional chemical etching, electrolytic etching offers several advantages. It allows the use of less aggressive electrolytes, such as oxalic or phosphoric acid, instead of strong acids like nitric or hydrochloric acid, improving safety and reducing hazardous waste (Struers, 2025a; Vander Voort, 1999). The process can be stopped instantly by switching off the current, minimizing the risk of over-etching or under-etching. In addition, it provides better reproducibility, ensuring more consistent results between samples. Sustainability is also improved through reduced chemical waste and easier disposal of used electrolytes (Struers, 2025a; Vander Voort, 1999).

Typical electrolytes used in electrolytic etching include (Vander Voort, 1999):

- Aqueous solutions of perchloric acid in ethanol (commonly used for steels and alloys).
- Oxalic acid solutions (used for stainless steels to reveal grain boundaries).
- Phosphoric acid (applied to aluminum and some steels).

Electrolytic etching is widely applied to stainless steels, nickel-based alloys, and aluminum alloys, where chemical etching may give less reliable results or involve more hazardous substances. It can also reveal certain features, such as carbide distributions, more clearly than chemical methods.

3.3 Chemical safety and environmental legislation

Working with chemicals in laboratories is tightly regulated to protect both workers and the environment. In the European Union, two of the most important regulations are the CLP Regulation (Classification, Labelling and Packaging of Substances and Mixtures) and the REACH Regulation (Registration, Evaluation, Authorization and Restriction of Chemicals) (European Chemicals Agency, 2023a; European Chemicals Agency, 2023b).

The CLP Regulation defines how chemicals must be labelled and packaged before they are used in workplaces. For example, bottles and containers must carry clear hazard pictograms, signal words, and safety phrases that tell users about risks such as "corrosive," "toxic," or "flammable." This ensures that laboratory staff can immediately recognize the type of danger and take the correct precautions (European Chemicals Agency, 2023b).

The REACH Regulation focuses on controlling which chemicals can be used in the EU and under what conditions. Companies are required to register the chemicals they import or manufacture, and they must provide safety information in the form of Safety Data Sheets (SDS). In practice, this means that laboratories always have access to detailed instructions on how to handle each substance safely and how to deal with spills or accidents (European Chemicals Agency, 2023a).

In Finland, the Waste Act and related national regulations define how chemical waste must be collected, stored, and treated. Hazardous waste must never be disposed of through regular sewage or mixed with normal trash. Instead, acids, solvents, and heavy metal residues are collected separately in labelled waste containers. These are then delivered to specialized waste treatment facilities (Finnish Waste Act, 2011).

Neutralization is commonly used to make acidic solutions safer before disposal. Strong acids, such as hydrochloric or sulfuric acid, should be neutralized gradually by adding an alkaline agent like sodium hydroxide (NaOH) or sodium carbonate (Na_2CO_3). During the process, pH and temperature must be monitored to avoid rapid reactions. Once neutralization is complete, the solution should be checked to confirm that the pH is close to neutral (around 6–8) and that no harmful residues remain before final disposal (National Research Council, 2011; North Industrial, 2025; Lab Alley, 2025).

For a company like Wärtsilä, following these rules is not only a legal requirement but also part of its sustainability goals. Proper chemical safety and waste management reduce environmental impact, protect employees, and help the company meet both national and international standards (Wärtsilä, 2025).

3.4 Occupational safety guidelines

Occupational safety in laboratories is guided both by legislation and by workplace-level practices. In Finland, the Finnish Institute of Occupational Health (TTL) provides recommendations for safe laboratory work. These guidelines cover, for example, the correct use of personal protective equipment (PPE) such as lab coats, safety goggles, gloves, and sometimes face shields when handling corrosive chemicals. (TTL, 2023; Valtioneuvoston asetus 427/2021).

Another important safety measure is the use of technical protective equipment. Fume hoods are essential when working with volatile or hazardous substances, since they prevent harmful vapors from spreading into the laboratory. Emergency showers and eye washing stations must also be available in case of accidents. Proper ventilation, fire safety systems, and safe chemical storage cabinets are part of the overall safety infrastructure (Työturvallisuuskeskus, 2023).

Workplace safety culture is equally important. Staff are expected to receive regular training on chemical risks, safe handling procedures, and emergency response. Written instructions and risk assessments should be available for all chemicals and processes in use. In addition, laboratories should carry out safety inspections to ensure that equipment, protective gear, and facilities are in good condition (Työturvallisuuskeskus, 2023).

At company level, internal policies often go beyond the legal minimum. Large companies like Wärtsilä usually have their own safety management systems, which include systematic risk assessments, continuous monitoring, and reporting of near misses or unsafe situations. This helps create an environment where safety is seen as a shared responsibility and not only as an individual task (Wärtsilä, 2025).

3.5 Continuous improvement approaches

Continuous improvement means developing work processes step by step to make them safer, more efficient, and more sustainable. Instead of large changes at once, the idea is to find small improvements that together create significant benefits over time.

In industrial environments, continuous improvement is often linked with methods such as Lean, 5S, and Kaizen. (Imai, 1986)

Lean thinking focuses on removing unnecessary steps and waste from processes (Liker, 2004). In a laboratory, this can mean organizing sample preparation so that time and resources are not wasted.

5S (sort, stabilize, shine, standardize, sustain) is a practical method for keeping the workplace tidy and organized (Liker, 2004). Applying 5S in a metallographic lab helps reduce accidents, makes tools easier to find, and ensures that chemicals are stored safely.

Kaizen emphasizes the idea that everyone can contribute to improvements. For laboratory work this can mean encouraging staff to suggest better ways to handle chemicals, maintain equipment, or prepare samples. (Imai, 1986)

For this thesis, continuous improvement is an important framework. It is not only about replacing chemical etching with electrolytic etching, but also about looking at related processes that could be made safer or more efficient. Examples are the new sample holder for valve grain flow samples and the possibility of cold mounting under vacuum. These are not big changes, but good examples of small steps in continuous improvement.

4 METHODOLOGY

4.1 Experimental study: comparison of conventional and electrolytic etching

4.1.1 Procedure

The primary research method in this thesis is experimental testing. Previously obtained microscopic images of the same materials etched by conventional chemical methods were used as references. If suitable reference images were not available, chemical etching was also performed for comparison. The results were compared based on clarity of grain boundaries and general microstructure. Also, safety and practicality were evaluated.

In this study, oxalic acid is selected as the electrolytic etchant because it is commonly used for these material types and is considered one of the safest among widely applied electrolytic etchants (Vander Voort, 1999; ASTM International, 2023). For comparison, phosphoric acid another commonly used electrolytic etchant is primarily applied to copper-based materials and poses a higher risk due to its greater toxicity upon skin contact (Vander Voort, 1999; ASTM International, 2023) . Since the focus of this thesis is on improving safety, using highly toxic chemicals would not align with the objectives. However, oxalic acid is not without hazards: it is poisonous if ingested and can irritate the upper respiratory tract and digestive system if inhaled. Skin contact produces a caustic effect and may discolor and embrittle fingernails, and it can cause serious eye damage (Vander Voort, 1999; Sigma-Aldrich, 2022b).

The procedure was identical for all four materials. For each material, five samples were prepared. Samples were mounted to facilitate grinding and polishing. Samples were mechanically polished to a final surface finish of 1 µm right before etching. Materials which were tested and their typical etchants can be seen in table 1. 2 steels and 2 nickel chromium-based alloys were used. Recipes of the etchants in Table 1 can be found from appendix 1.

Table 1 Test materials and typical chemical etchants.

Material	Material type	Typical etchant	Reason for selection
3d printed 316L	low-carbon stainless steel	Aqua regia	Widely known to etch well with oxalic acid.
X45CrSi9-3	martensitic stainless steel	Kalling's 1 or 2	Very common material for me to work with.
3d printed Inconel 718	nickel-based superalloy	Marble's	Somewhat known to etch with oxalic acid.
Nimonic 80a	nickel-based superalloy	Aqua regia, Kalling's 1 or 2	Common material for me to work with.

Etching was carried out in stages: all five samples were etched at the initial voltage setting, followed by microscopic evaluation. After evaluation, samples were reground, repolished and etched at the next voltage level. There was also possibility of stopping earlier if satisfactory results were obtained before completing all planned steps. In case a sample would been over etched already at the first voltage/time combination, the time or voltage could be decreased for subsequent tests. If samples show signs of over-etching before completing all planned voltage and time combinations, the test was stopped. Conversely, if the microstructure was not sufficiently revealed after the final etching step, voltage and time was increased. If no satisfactory results were obtained after completing all 20 tests and possible extra tests, the material was considered unsuitable for electrolytic etching under these conditions, and testing was proceeded to the next material. Initial testing voltages and times can be seen in table 2, current (A) is automatic and can’t be adjusted. During the etching two parameters were varied: time 15, 30, 45, 60 and 75s and voltage at 6, 8, 10 and 12 V.

Table 2 Test voltages and times.

Etchant	Time(s)	Voltage(V)	Etchant	Time(s)	Voltage(V)
$C_2H_2O_4$ (10%)	15	6	$C_2H_2O_4$ (10%)	15	10
$C_2H_2O_4$ (10%)	30	6	$C_2H_2O_4$ (10%)	30	10
$C_2H_2O_4$ (10%)	45	6	$C_2H_2O_4$ (10%)	45	10
$C_2H_2O_4$ (10%)	60	6	$C_2H_2O_4$ (10%)	60	10
$C_2H_2O_4$ (10%)	75	6	$C_2H_2O_4$ (10%)	75	10
Etchant	Time(s)	Voltage(V)	Etchant	Time(s)	Voltage(V)
$C_2H_2O_4$ (10%)	15	8	$C_2H_2O_4$ (10%)	15	12
$C_2H_2O_4$ (10%)	30	8	$C_2H_2O_4$ (10%)	30	12
$C_2H_2O_4$ (10%)	45	8	$C_2H_2O_4$ (10%)	45	12
$C_2H_2O_4$ (10%)	60	8	$C_2H_2O_4$ (10%)	60	12
$C_2H_2O_4$ (10%)	75	8	$C_2H_2O_4$ (10%)	75	12

Additionally, Optical Emission Spectroscopy (OES) analysis and hardness measurements were performed for each material to provide a clearer understanding of their properties. For the nickel-based materials OES analysis had to be swapped for Energy Dispersive Spectroscopy (EDS) analysis because I was obtaining inaccurate results.

4.1.2 Used equipment

The electrolytic etching tests were carried out using the Struers LectroPol-5 device (Figure 3), an automated unit designed for electrolytic polishing and etching in metallographic preparation. The system provides precise control of voltage and time, ensuring repeatable and reliable results (Struers, 2025c). In addition, LectroPol includes a scanning function for determining the optimal voltage and a method database for various materials (Struers, 2025c). However, in this thesis, these advanced features were not utilized; instead, the tests followed a predefined test table, and 10 % oxalic acid were used as the electrolyte. Although this machine was already available in our laboratory, it had been sitting in a cabinet unused for many years.

Figure 3 Struers Lectropol-5

Microscopic evaluation of the etched samples was performed using a Keyence VHX-X1 digital microscope (Figure 4). The system offers magnification up to 2500× and features high-resolution imaging with adjustable depth of field, enabling clear observation of microstructural details (Keyence, 2025). All pictures taken with the Keyence microscope in this thesis will include a scale bar and a magnification indicator in the lower right corner.

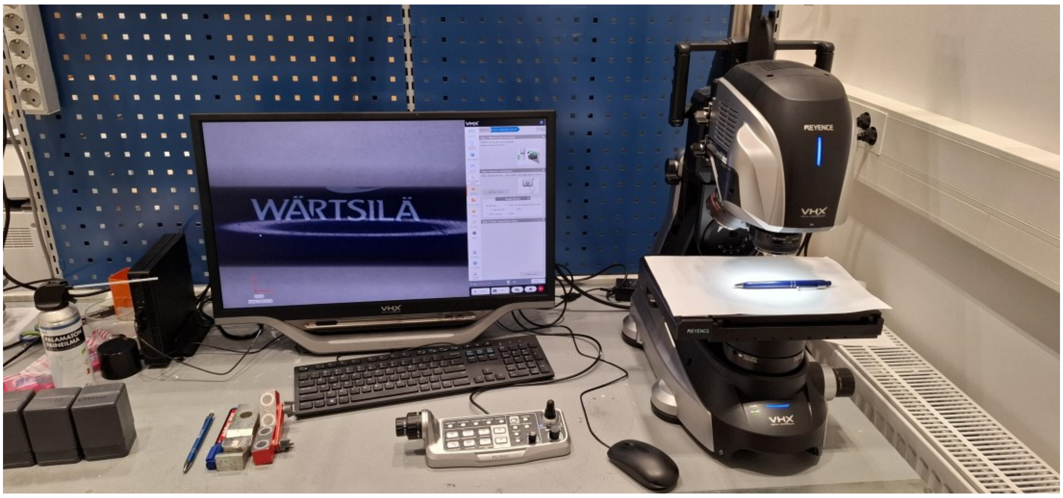

Figure 4 Keyence VHX-X1 digital microscope

Hardness measurements were conducted using the Struers DuraScan hardness tester (Figure 5). The available methods on this machine are Vickers and Knoop, with a load range of 0.01 kg to 10 kg. Five test points were measured using HV10 for each material.

Figure 5 Struers Durascan

Chemical composition analysis was performed using the Hitachi Foundry-Master Smart OES analyzer (Figure 6). This optical emission spectrometer provides rapid and precise elemental analysis of metallic samples, ensuring accurate identification of alloy composition before etching (FinFocus, 2025). Five successful test points were saved as a report.

Figure 6 OES analyzer.

4.2 Chemical etching

The only material for which I couldnt find suitable reference images of chemically etched microstructures was 316L stainless steel. Therefore, I performed chemical etching on this material to obtain an image. This was necessary to provide a reliable visual reference for comparison with electrolytic etching.

The chemical etching process begins by preparing the etchant, unless a ready solution is available. Also, some etchants cannot be stored for long periods. In this case, I used Kalling's 2 etchant, which was already prepared and stored in the cabinet.

For the etching method, I chose submersion rather than swabbing. A small Petri dish was used, and a thin layer of etchant was poured into the bottom. The sample was partially submerged so that only the top surface of the area to be examined was in contact with the etchant. After etching, the sample was rinsed thoroughly with water, followed by ethanol, and then dried.

4.3 Workplace observation: assessment of current chemical handling practices

The second method involved direct observation of current chemical handling practices in the laboratory. This included examining how chemicals are stored, labeled, and disposed of. In addition, the use of safety equipment such as fume hoods, storage cabinets, and personal protective gear was assessed during daily operations. These observations provided valuable insights into existing practices and helped to identify potential risks.

Based on this evaluation, suggestions for new or improved practices were developed. The goal was to enhance chemical safety without compromising efficiency. Proposed improvements focus on practical measures that can reduce hazards and streamline workflows.

4.4 Documentation

All findings were documented systematically. Laboratory notebooks were used to record procedures and results. Microscopy images were saved in digital format. Excel templates helped to organize the results and make comparisons between different methods easier. This documentation ensures that the research can be repeated and that the results are transparent.

Every file used in this thesis was stored in Wärtsilä Corporation’s OneDrive. If physical notes contained important information, they were digitized and saved to OneDrive as well. Later, it will be discussed whether there is a possibility or need to save some of the more important information also to Miles. Physical samples used in etching trials will most likely be scrapped due to lack of storage space.

5 RESULTS

5.1 Comparison of electrolytic and conventional etching

The results of the experimental tests will demonstrate how electrolytic etching compares with traditional chemical etching. The comparison will focus on the quality of the etched microstructures. Microscopy images will be presented to highlight differences in grain boundary visibility, clarity, and reproducibility. In addition, elemental analysis and hardness measurements will be included, along with basic information about the test materials. However, I will not go too deeply into microstructure analysis, as I do not have much expertise in that area. Only the best results regarding microstructure clarity are presented here. For each material 2 pictures from electrolytically etched surfaces are presented along with 1 picture of unetched surface and 1 picture of chemically etched surface.

5.1.1 316L

The first material in the etching trial was 3D-printed 316L. 316L is austenitic low carbon stainless steel (Alumeco, 2025). In this case it was manufactured using either Wire Arc Additive Manufacturing (WAAM) or Laser Metal Deposition (LMD). Unfortunately, I could not confirm which of these methods was used. Both WAAM and LMD are types of Directed Energy Deposition (DED) processes. LMD uses laser to melt powder or wire, while WAAM employs an electric arc to melt wire (RamLab, 2025). Essentially, both methods are like welding and do not produce the same microstructure as conventional 316L stainless steel.

The average hardness of the material was measured at 196 HV10. The OES analysis results appeared consistent with typical 316L; however, since the exact manufacturing method and material is unknown, a direct comparison cannot be made. The full OES analysis can be found in Appendix 3. Additionally, a microscope image of the entire sample, including the hardness testing points, is provided in appendix 2.

Electrolytic etching with oxalic acid was easy. Good results were already visible after 6 volts and 30 seconds, and the best results were achieved at 6 volts and 60 seconds (Figure 7, bottom pictures). Beyond that, there were no significant changes until 12 volts and 60 seconds, when the sample started to become over etched. Obtaining reference picture was more challenging. I could not find any old images to use as reference, so I had to etch one sample chemically. For this, I used Kallings 2 etchant, as my colleague recommended it based on good results with 3D-printed stainless steel. The chemical etching required several attempts because the sample tended to go from under etched to over etched very quickly. Eventually, I managed to get a good reference image (Figure 7, top right), but the process was much more difficult compared to electrolytic etching, which worked well with almost any voltage and time combination in my test matrix (Table 2). Figure 7, top left picture shows the surface before any etching was applied.

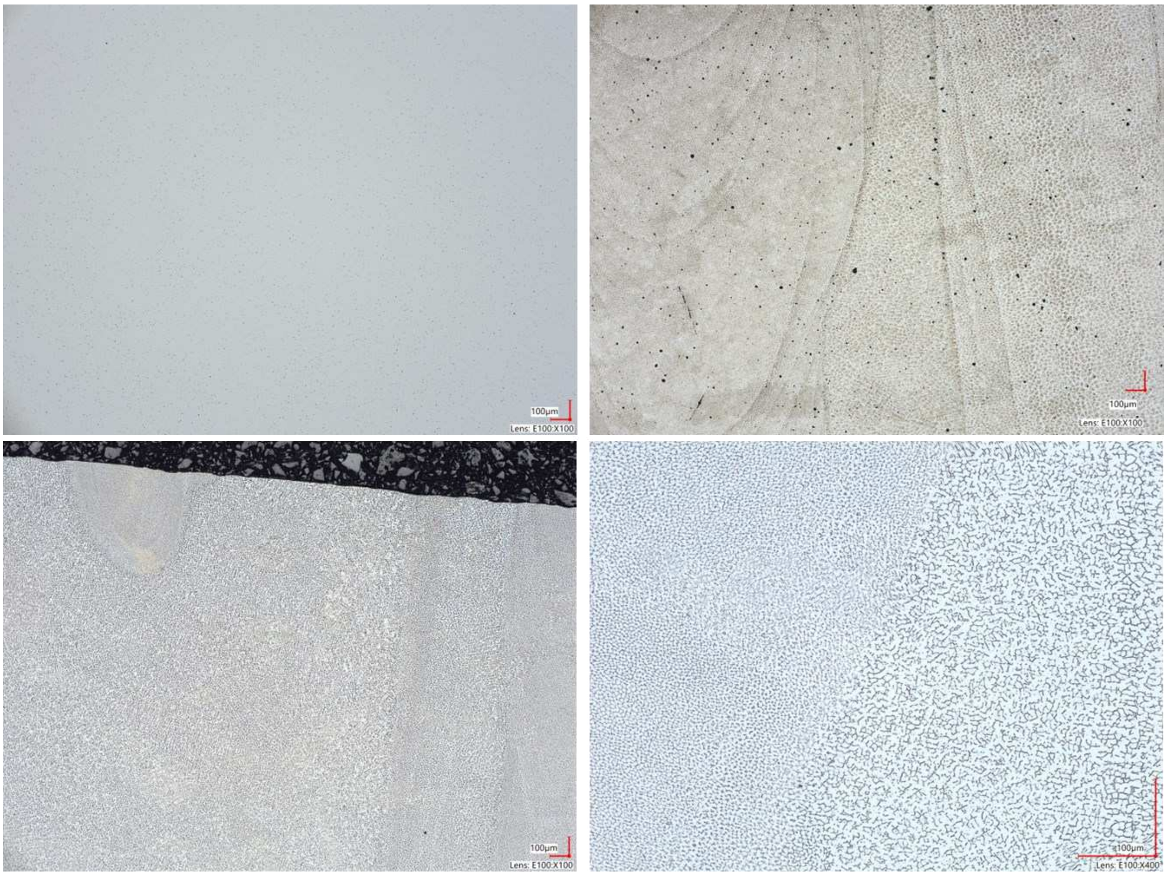

Figure 7 316L surface in different conditions.

5.1.2 X45CRSi9-3

The second material was X45CrSi9-3, a martensitic steel mainly used in valves (SteelNumber, 2025). These samples were cut from a valve stem. All five samples came from the same valve.

The average hardness of the material was measured at 292 HV10. OES analysis results are fully correlated with the reference from the Total Materia database (Total Materia, 2025). The complete OES analysis can be found in appendix 5. Microscope image of the full sample with hardness measurement points can be found in appendix 4.

Initial results appeared at 6 volts and 30 seconds of electrolytic etching. The images obtained at 6 volts and 45 seconds (Figure 8, bottom pictures) represent the best result achieved; beyond that, no significant changes occurred even after completing the entire test matrix. The reference image (Figure 8, top right picture) is from a colleague’s old case using the same material etched with Kallings 2, where the martensitic structure is visible, something that did not appear with oxalic acid. Revealing the martensitic structure requires a stronger acid due to its high corrosion resistance and complex microstructure. With oxalic acid, grain boundaries were somewhat visible, but that was the extent of it. To the naked eye, the sample looked like one etched with Nital for grain flow, so oxalic acid could be suitable for that purpose if the valve can fit into the setup. Figure 8, top left picture shows the surface before any etching was applied.

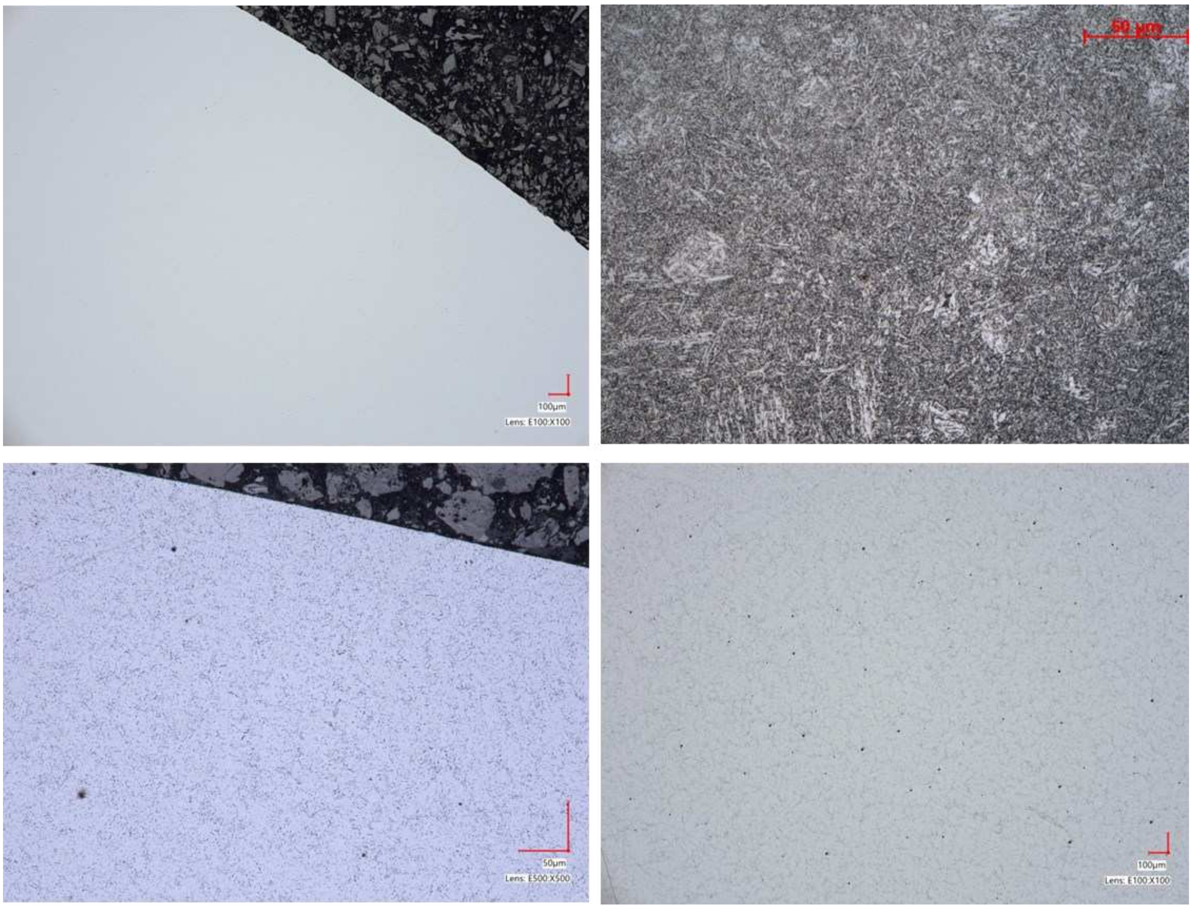

Figure 8 X45CrSi9-3 surface in different conditions.

5.1.3 Inconel 718

The third material in the etching trial was Inconel 718. Inconel 718 is a nickel-based superalloy where chromium is the second most present element after nickel (Nine Steels, 2025a). The specific Inconel used in this trial was manufactured using Laser Powder Bed Fusion (L-PBF) additive manufacturing technology.

The average hardness of the material was measured at 455 HV10, making it the hardest material in this experiment. I assume it has undergone some form of heat treatment, although I do not have exact information. Since I have experienced some inaccuracies when measuring nickel-based alloys with OES, I substituted the OES analysis with EDS analysis, as shown in appendix 7. EDS analysis is fully correlating with Inconel 718 reference in total material database (Total Materia, 2025). Microscope image of the full sample with hardness measurement points can be found in appendix 6.

Electrolytic etching of Inconel 718 was interesting because it colored the sample very strongly, as seen in Figure 9 bottom pictures. In Figure 9, bottom right picture grain boundaries are somewhat visible. The colors changed as the test continued, but the grain boundary visibility remained largely the same. At 12 volts, the sample became so over etched that no details could be seen. The reference image (Figure 9, top right picture) is from an earlier case etched with Marble's etchant. Figure 9, top left picture shows the surface before any etching was applied.

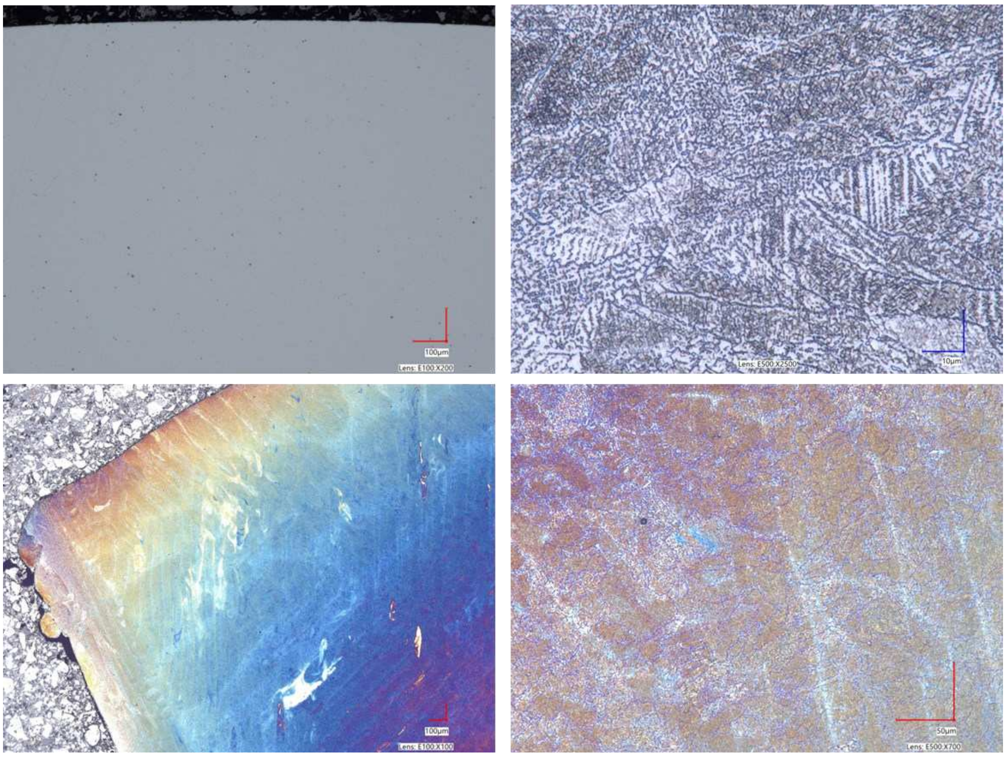

Figure 9 Inconel 718 surface in different conditions.

5.1.4 Nimonic 80a

The fourth and final material in the etching trial was Nimonic 80A. Nimonic 80A is also a nickel-based superalloy like Inconel 718, and it contains chromium as the second most abundant element after nickel (Nine Steels, 2025b). These samples were cut from a valve stem.

The average hardness of the material was 369 HV10. For the same reason as with Inconel 718, the OES analysis for this material was substituted with EDS analysis. The EDS analysis can be found in Appendix 9. EDS analysis is fully correlating with Nimonic 80a reference in total material database (Total Materia, 2025). Microscope image of the full sample with hardness measurement points can be found in appendix 8.

Chemically etching Nimonic 80A for grain boundaries is fairly easy, as shown in the reference image (Figure 10, top right picture). I still wanted to see if grain boundaries could also be revealed using oxalic acid. Figure 10 bottom right pictures show that it worked quite well. Similar to Inconel 718, the surface was heavily colored after etching. I noticed that washing the sample with dish soap after etching removes the colors while keeping the grain boundaries visible. After discovering this, I tried the same method with Inconel, but it did not work as effectively. Figure 10, top left picture shows the surface before any etching was applied.

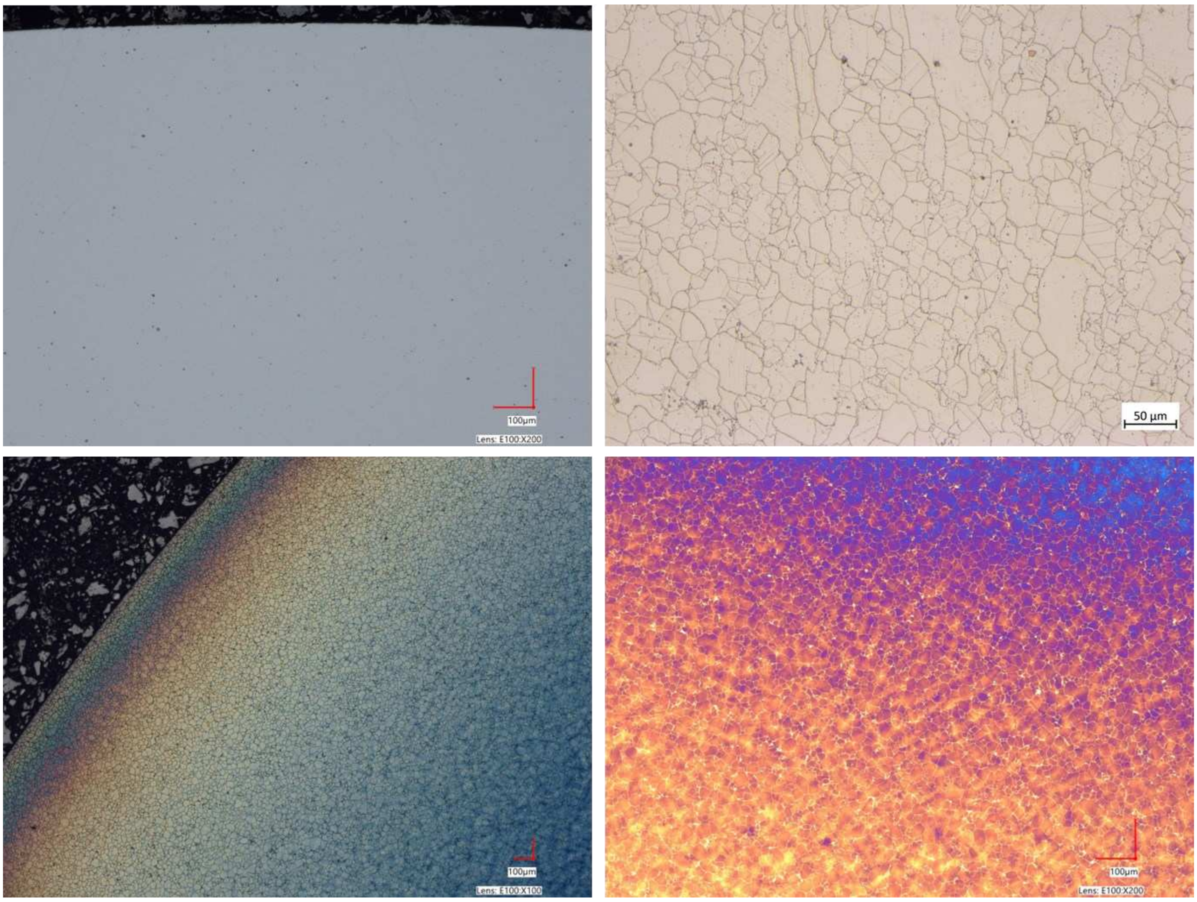

Figure 10 Nimonic 80a surface in different conditions.

5.2 Chemical handling and disposal

After observing the current chemical handling and disposal practices in the laboratory, it was concluded that the overall level of chemical safety is good. Staff have received proper training on how to handle chemicals safely during etching and when preparing etchants. Personal protective equipment and fume hoods are used appropriately, and general safety awareness is high. However, the observation also revealed three areas that could be improved to further enhance safety, efficiency, and documentation. These issues are related to labeling of premixed etchants, chemical disposal, and the completeness of the etching diary.

During the observation, it was noted that some premixed etchants stored in cabinets were either missing labels or had faded markings. This poses a risk of confusion and potential misuse. To correct this, all premixed etchants should be labeled with durable marking that clearly indicate the contents and preparation date. Additionally, a routine inspection schedule should be introduced to ensure that labels remain legible and up to date. This simple measure will improve traceability and reduce the risk of handling errors.

I was also asked to explore possibilities for neutralizing acids to support safer disposal practices. Below is a step-by-step example for neutralizing nitric acid, which is likely the most commonly used acid in our laboratory. The procedure is pretty simple and straightforward and has been collected from two sources (North Industrial, 2025; Lab Alley, 2025).

Required Equipment and PPE:

- Personal Protective Equipment (PPE): safety goggles or face shield, chemical-resistant gloves, lab coat
- Tools: pH paper or pH meter, stirring rod, glass or plastic neutralization vessel, and water

Choosing a Neutralizing Agent:

The following bases can be used:

- Sodium hydroxide ($NaOH$)
- Calcium hydroxide ($Ca(OH)_2$)
- Magnesium oxide (MgO)
- Ammonia (NH_3)
- Sodium bicarbonate also known as baking soda ($NaHCO_3$) – recommended for small-scale operations because it is inexpensive and easy to obtain

Prepare the neutralization vessel:

- Mix the chosen neutralizing agent with water.
- You can also dilute the nitric acid with water first to make the reaction more controlled. In our case, the acid is usually already diluted for example Nital etchant or Etchant 219.

Add the base solution to the acid slowly:

- Pour the neutralizing mixture into the nitric acid in small increments while stirring continuously.
- Add slowly to avoid excessive heat and splashing.

Check the pH:

- Use pH paper or a pH meter.
- Target pH: approximately 6–8 (neutral range).

Repeat if necessary:

- If the pH is not neutral, continue adding the base in small amounts and stir until the desired pH is reached.

Important Safety Notes:

- Always add base to acid, never the other way around, to prevent uncontrolled reactions.
- Perform the procedure under a fume hood or in a well-ventilated area.

Third topic I want to address is the etching diary. The Current etching diary in excel is useful but lacks complete recipes and sufficient reference images. To improve its functionality, the diary should include recipes taken directly from the ASTM E407 standard wherever applicable, along with reference images for each etchant. These reference images should be easily accessible, rather than requiring users to search through M-Files for a report. Regular updates and contributions from the team should be encouraged to keep the diary accurate and comprehensive. Implementing these improvements will make the diary a reliable resource for both routine work and future investigations.

6 IMPROVEMENTS IN PRE-ETCHING STAGES

6.1 Grain flow sample preparation problem

One of the common tasks in valve validation investigations is examining the grain flow in the valve head. Traditionally, this has been done by cutting the valve head in half and manually grinding and polishing the surface. Once the surface is completely polished, it must be etched to reveal the grain flow, which shows how the grains are oriented in the forged component.

Typically, other samples analyzed in our laboratory are small enough to be mounted in resin. If the valve is very small, it can also be mounted and processed using standard sample holders. Alternatively, half of the valve head can be mounted if the valve is larger. However, mounting only half of the valve head is not ideal, as it is better to have the entire cross-sectional surface available.

Manual grinding of valves can be difficult and time-consuming, especially for those without experience. First, the surface must be made flat and then scratch-free, both of which take considerable time. This led my colleague Lwin Phyo and me to consider whether the process could be made more efficient and easier.

6.2 Grain flow sample holder

We contacted Wärtsilä’s additive manufacturing team and began brainstorming ideas. It was quickly decided to design a holder similar to those used for mounted samples (Figure 11). In the Struers Tegrapol-31 machine, mentioned earlier in Chapter 3.1, there are five pistons that press the sample down during grinding and polishing. For the grain flow samples, these same pistons needed to press down on the sample while it was secured in the holder. The pistons align with the holes shown in Figure 11.

Figure 11 Sample holder for 30mm sample.

Initially, there was some miscommunication, and the first prototype we received was a black block (Figure 12) intended to assist with manual grinding. After clarifying our requirements, new prototypes were produced, but there were measurement issues, the pistons were contacting the wrong areas. Once we provided the standard sample holder (Figure 11) as a reference, functional prototypes were delivered immediately.

Figure 12 First prototype of grain flow sample holder.

Now, six months later, I have prepared approximately ten grain flow samples using these holders (Figure 13) without any major issues. There are several holder sizes available for different valve dimensions. The finished samples are not perfect but are generally of good quality. Minor defects such as rounded corners, scratches (Figure 14), or unground areas on the top (Figure 15) still occur, but these were also common with manual grinding and polishing. While a highly skilled operator might achieve a slightly better finish manually, there is also a risk of excessive corner rounding if sample is grounded or polished for too long.

This improvement has significantly reduced the time required for grain flow sample preparation. The fastest time from cutting the piece to polishing, etching, and photographing has been 1 hour and 30 minutes. Previously, when I first started working here, the same process could take me days. Following this successful collaboration with the additive manufacturing team, we have continued to request additional small prints, and they have consistently delivered satisfactory results.

Figure 13 Different sizes of grain flow sample holders.

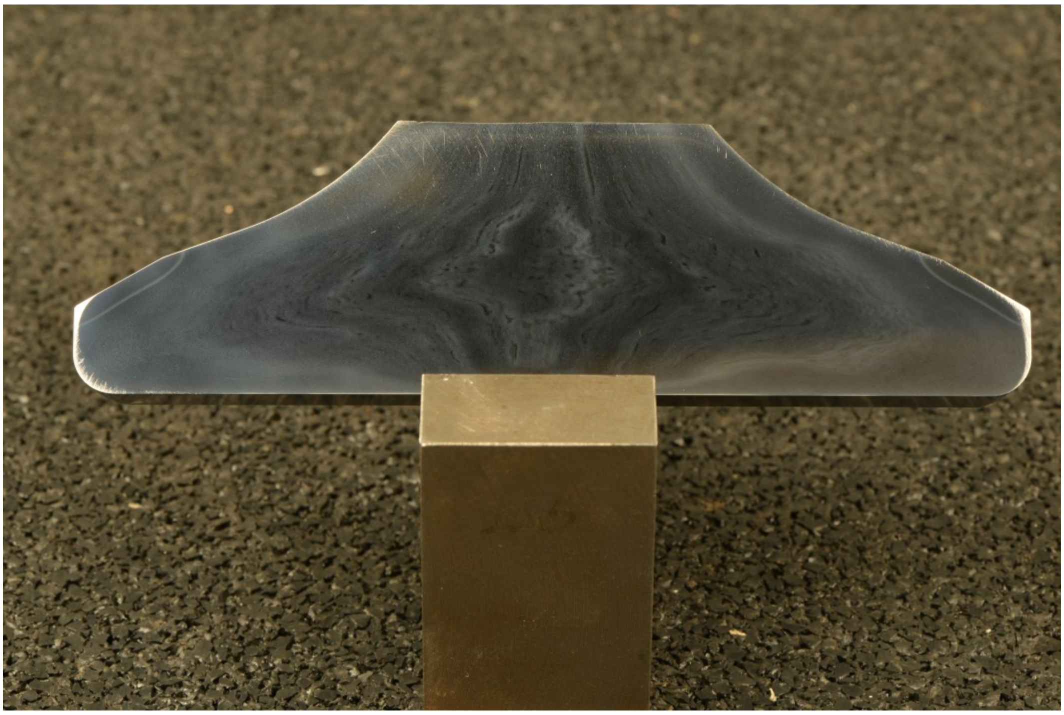

Figure 14 Polished grain flow sample etched with nital (1).

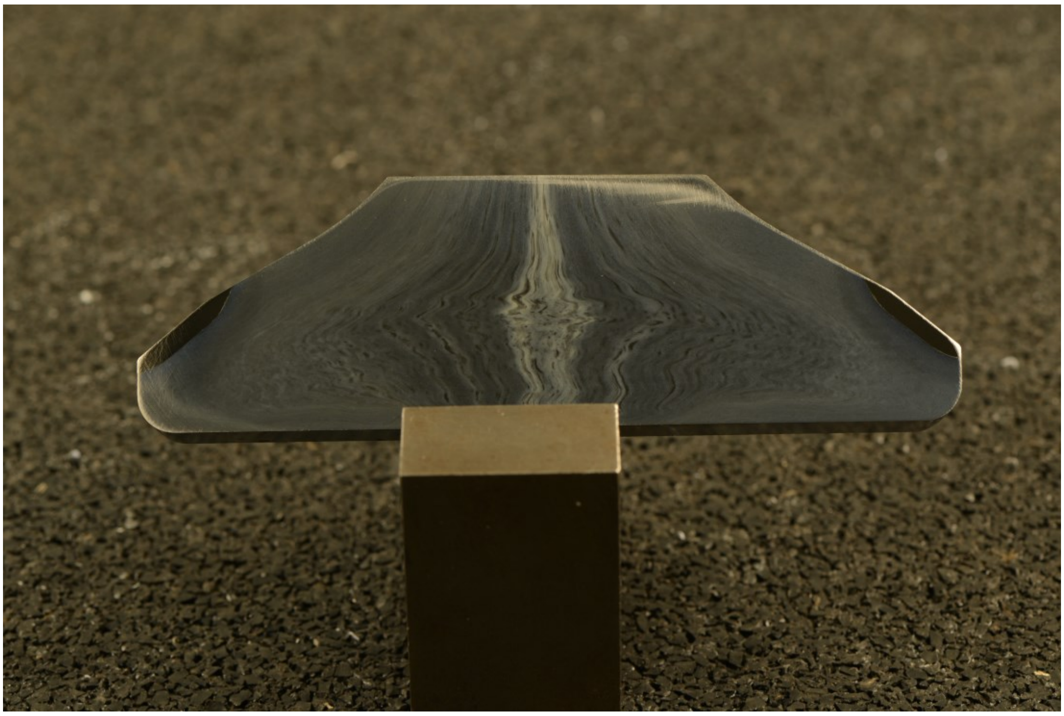

Figure 15 Polished grain flow sample etched with nital (2).

6.3 Cold mounting with vacuum

Sometimes I have experienced issues with hot-mounted samples during polishing. Achieving a scratch-free surface can be impossible if there are cracks, porosity, or if the sample has a complex shape with many holes. One possible solution to this problem could be switching from hot mounting to cold mounting with vacuum impregnation. Because the surface quality would be improved, etching would become easier, which in turn would reduce chemical usage.

When cold mounting, the sample is prepared in the same way as for hot mounting: it is cut to a suitable size, cleaned (for example with ethanol), and dried. The specimen is then placed in a cup of the correct size, and resin is poured over it. The resin is either acrylic or epoxy. Epoxy systems consist of two components: resin and hardener. Acrylic resin also has two components: powder and liquid. Before pouring the resin into the cup, these components must be mixed. Curing time varies from 20 minutes to 20 hours (Struers, 2025e).

Vacuum impregnation can only be implemented with epoxy resins because of their low viscosity and low vapor pressure. One machine that can perform this process is the Struers Citovac. It also makes filling the cups with resin easier and safer, reducing the risk of spills. The sample is placed in the Citovac vacuum chamber, and the lid is closed. Evacuation takes a few minutes to ensure that air is removed from cracks and pores. Then the epoxy is allowed to fill the cups. Once the specimens are completely covered in epoxy, the epoxy valve and vacuum pump are closed (Struers, 2025d). Atmospheric pressure then forces the epoxy into the pores and cracks. Figure 16 shows the filling process on the Struers Citovac, and Figure 17 shows a polished sample that contained significant porosity but no scratches. This sample was mounted to epoxy by using the Struers Citovac and then polished.

Figure 16 Strues Citovac.

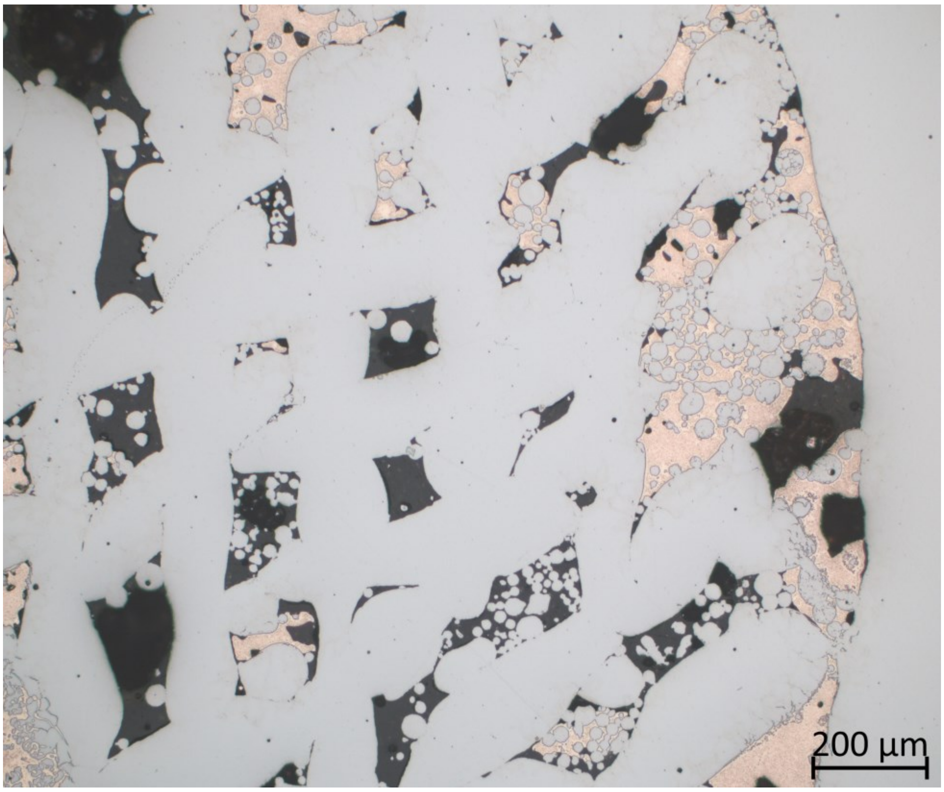

Figure 17 Sample mounted with Struers Citovac (Struers, 2025d).

I understand that implementing cold mounting with vacuum in our laboratory may not currently be ideal. This is mainly because we have relatively low sample volumes. Cold mounting is most efficient when producing large batches of samples at once. Additionally, it is rare that we cannot achieve a good enough surface with hot-mounted samples. Another consideration is that for scanning electron microscopy (SEM), which many samples require, the specimen must be conductive, epoxy is not. However, in the future, if our department grows and we need to process a large number of samples or lot of samples with significant porosity, this method could become a valuable option.

7 DISCUSSION AND CONCLUSIONS

This thesis is not intended as a complete guide to metallography or electrolytic etching. However, it provides a solid starting point for implementing electrolytic etching in the daily operations of our laboratory and demonstrates what can be achieved with oxalic acid. In addition, the work introduced one improvement in the pre-etching stages that saves time and proposed other enhancements related to chemical handling and labeling.

The main research question was whether electrolytic etching can replace chemical etching. Based on the scope of this thesis, a definitive answer cannot be given. However, according to the research and experiments conducted, the answer is both yes and no. Electrolytic etching cannot fully replace chemical etching, but both methods should be considered to achieve the best possible results for microstructure and grain boundary visibility. Whenever possible, the safer method with less toxic chemicals should be chosen. I am confident that we will continue experimenting with electrolytic etching now that the process has been introduced. For future work, I propose studying the long-term performance and reliability of electrolytic etching in routine laboratory use.

Regarding improvements in chemical handling and disposal processes, the current practices were already in good condition, so no major changes were proposed. However, for used chemicals, I suggest implementing a neutralization protocol. Additionally, although slightly outside the main topic, I proposed improvements to the etching diary, which is closely related to chemical handling.

As for optimizing earlier steps in the sample preparation chain to support safer and more consistent results, together with a colleague, we successfully implemented one improvement in the grinding and polishing stage. I also made one proposal for the future, which would become relevant if certain conditions arise.

Recommendations for Implementation

To ensure practical benefits from this work, the following steps are recommended for the laboratory:

- Adopt electrolytic etching for 316L stainless steel, as it proved effective and forgiving compared to chemical etching.
- Update the etching diary by adding electrolytic etching parameters and observations for different materials. Include ASTM E407 standard recipes where applicable, along with easily accessible reference images for each etchant.
- Introduce a neutralization protocol for used chemicals to improve safety and environmental compliance.
- Continue monitoring and documenting electrolytic etching performance in routine operations to build long-term reliability data.
- Consider implementing cold mounting with vacuum for samples with cracks, porosity, or complex geometries.

Comparison of etching methods by material

From a safety perspective, electrolytic etching I used only oxalic acid, which is significantly less hazardous than the hydrochloric and nitric acids that were used in chemical etching. In terms of time, both electrolytic and chemical methods typically complete etching in under one minute, so the difference is negligible. Regarding waste, the electrolytic method generates less because the solution can be reused. In my trials, I used the same solution more than 80 times without noticing any deterioration in its effectiveness. Table 3 summarizes the etching outcomes for four materials examined with both electrolytic and chemical etching techniques

Table 3 Comparison of etchings methods by material.

Material	Electrolytic Etching Outcome	Chemical Etching Outcome
316L	Good visibility at 6 V / 60 s; easier and more forgiving process	Difficult to optimize; rapid under- or over-etching
X45CrSi9-3	Grain boundaries somewhat visible; less detailed than chemical	Clear martensitic structure visible
Inconel 718	Strong coloration; risk of over-etching at high voltage	Clear grain boundaries with Marble's etchant
Nimonic 80a	Effective grain boundary visibility; coloration removable by soap	Easy grain boundary etching with Kalling's etchant

Lastly, I would like to thank my Vamk supervisor, Joel Songok, my supervisor at Wärtsilä, Harri Koivisto, and my manager, Jyrki Suutala, for making this thesis possible. I also want to express my gratitude to my team Materials and Tribology for answering my questions and providing materials. Also, thanks to the Service team (VIL) which shares the lab space with us and to the Additive Manufacturing team for supplying prints and materials.

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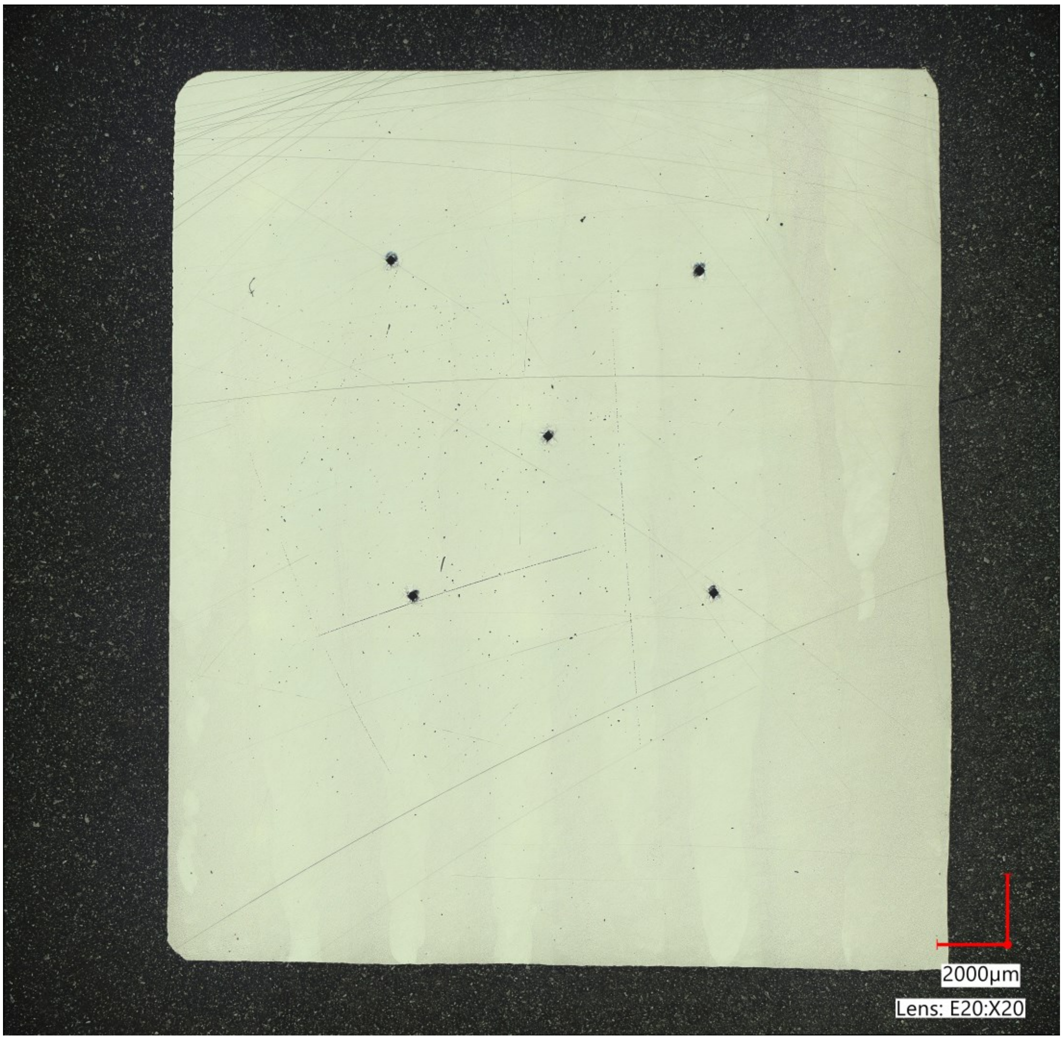
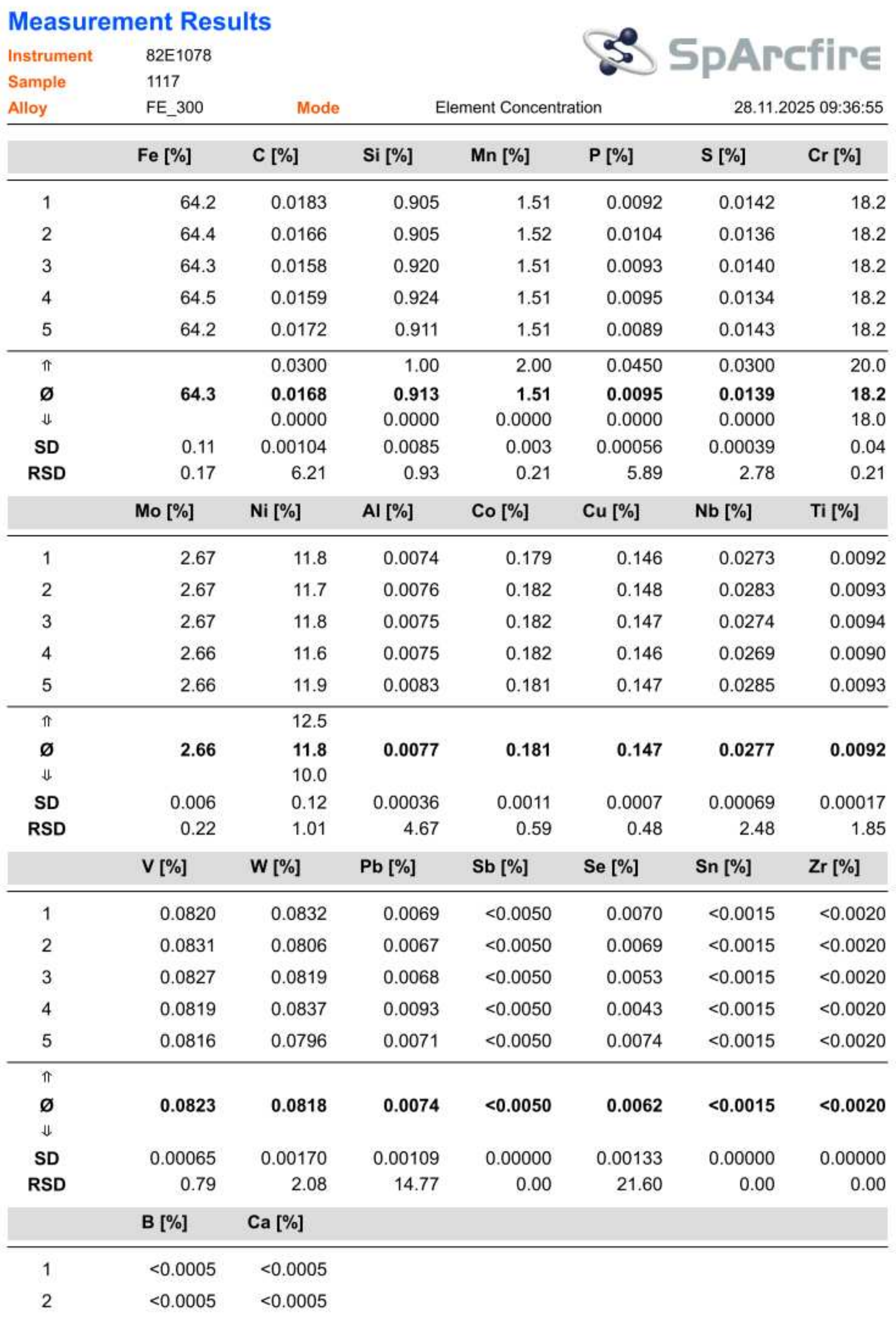
9 APPENDICES

Marble's	10g $CuSO_4$ 50 mL HCl 50 mL water
Kalling's 2	2g $CuCl_2$ 40 mL HCl 40-80 mL ethanol (95%) or methanol (95%)
Kalling's	2g $CuCl_2$ 40 mL HCl 40-80 mL ethanol (95%) or methanol (95%) 40 mL water
Aqua regia	20 mL HNO_3 60 mL HCl

Appendix 1 Recipes of mentioned chemical etchants (ASTM international, 2023).



Appendix 2 316L full sample.



Measurement Results

Instrument 82E1078
Sample 1117
Alloy FE_300 **Mode** Element Concentration 28.11.2025 09:36:55

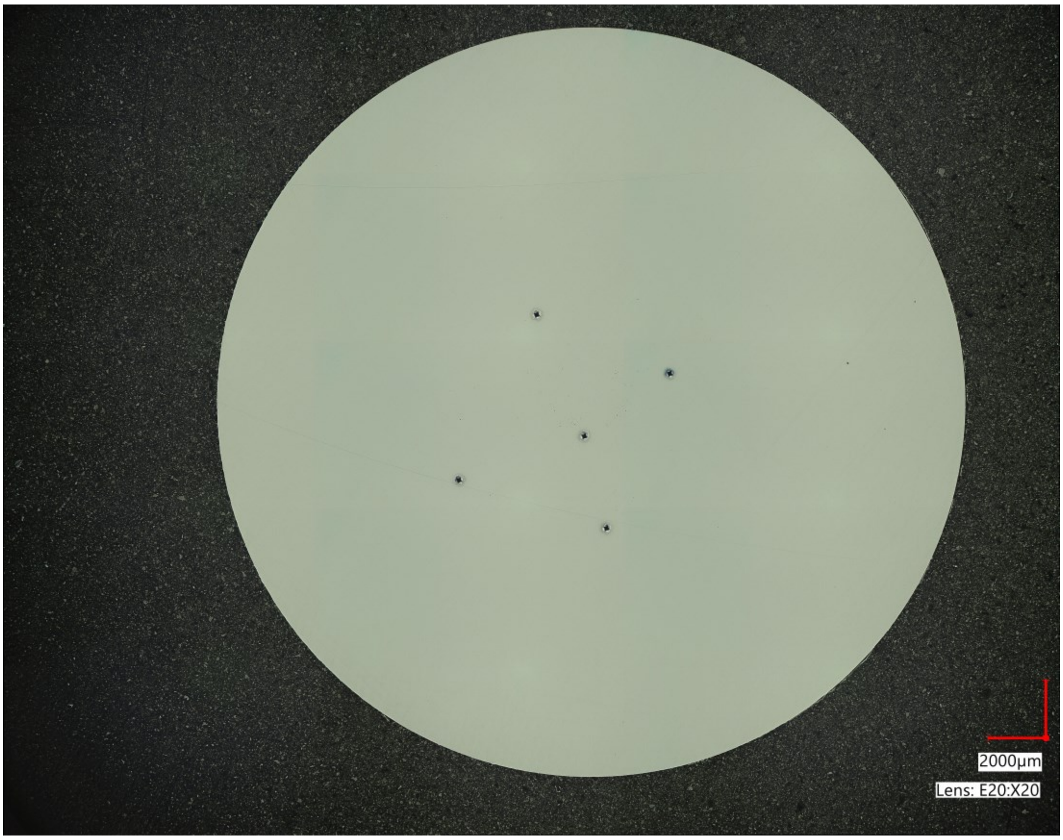
	Fe [%]	C [%]	Si [%]	Mn [%]	P [%]	S [%]	Cr [%]
1	64.2	0.0183	0.905	1.51	0.0092	0.0142	18.2
2	64.4	0.0166	0.905	1.52	0.0104	0.0136	18.2
3	64.3	0.0158	0.920	1.51	0.0093	0.0140	18.2
4	64.5	0.0159	0.924	1.51	0.0095	0.0134	18.2
5	64.2	0.0172	0.911	1.51	0.0089	0.0143	18.2
⇑		0.0300	1.00	2.00	0.0450	0.0300	20.0
Ø	**64.3**	**0.0168**	**0.913**	**1.51**	**0.0095**	**0.0139**	**18.2**
⇓		0.0000	0.0000	0.0000	0.0000	0.0000	18.0
SD	0.11	0.00104	0.0085	0.003	0.00056	0.00039	0.04
RSD	0.17	6.21	0.93	0.21	5.89	2.78	0.21

	Mo [%]	Ni [%]	Al [%]	Co [%]	Cu [%]	Nb [%]	Ti [%]
1	2.67	11.8	0.0074	0.179	0.146	0.0273	0.0092
2	2.67	11.7	0.0076	0.182	0.148	0.0283	0.0093
3	2.67	11.8	0.0075	0.182	0.147	0.0274	0.0094
4	2.66	11.6	0.0075	0.182	0.146	0.0269	0.0090
5	2.66	11.9	0.0083	0.181	0.147	0.0285	0.0093
⇑		12.5					
Ø	**2.66**	**11.8**	**0.0077**	**0.181**	**0.147**	**0.0277**	**0.0092**
⇓		10.0					
SD	0.006	0.12	0.00036	0.0011	0.0007	0.00069	0.00017
RSD	0.22	1.01	4.67	0.59	0.48	2.48	1.85

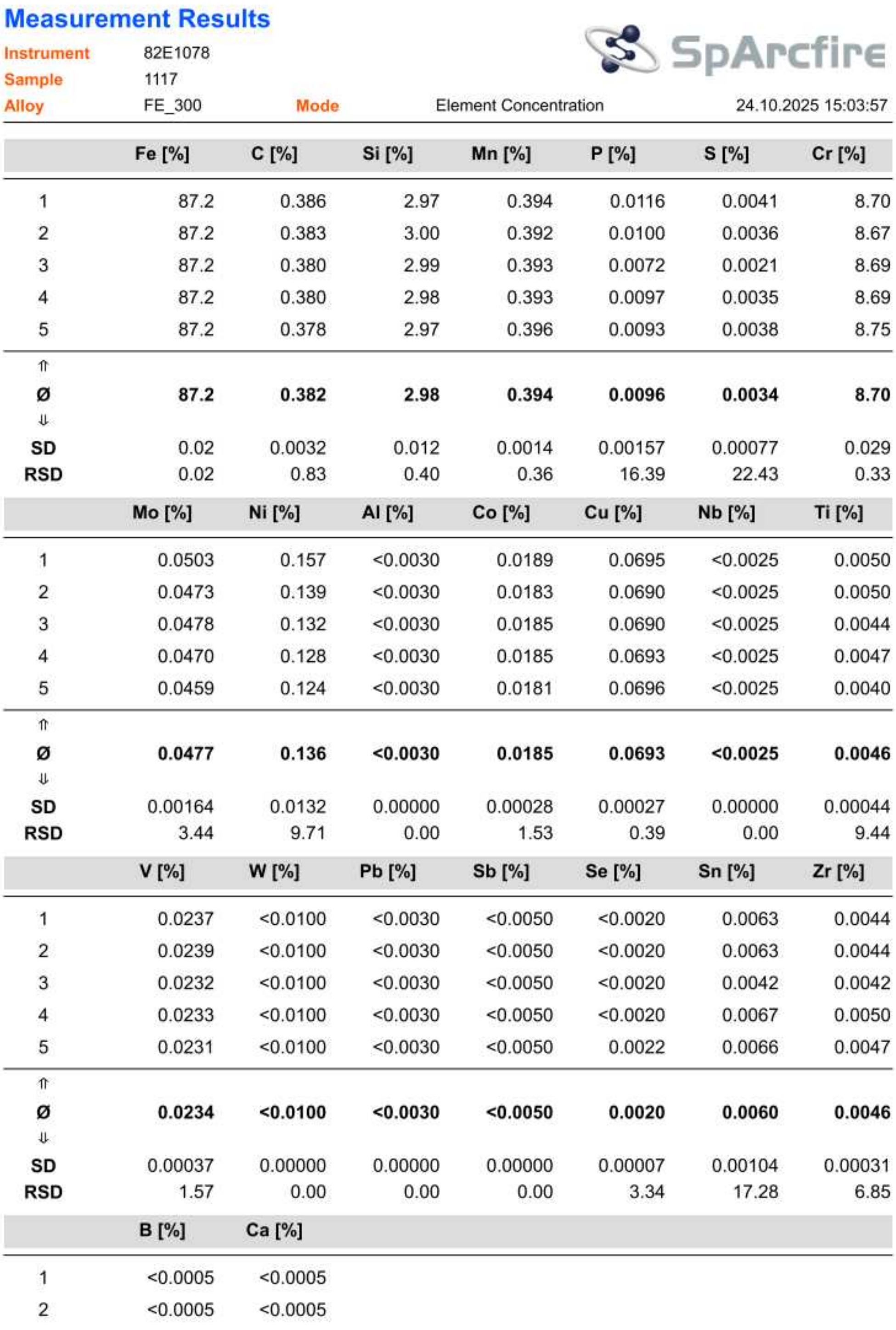
	V [%]	W [%]	Pb [%]	Sb [%]	Se [%]	Sn [%]	Zr [%]
1	0.0820	0.0832	0.0069	<0.0050	0.0070	<0.0015	<0.0020
2	0.0831	0.0806	0.0067	<0.0050	0.0069	<0.0015	<0.0020
3	0.0827	0.0819	0.0068	<0.0050	0.0053	<0.0015	<0.0020
4	0.0819	0.0837	0.0093	<0.0050	0.0043	<0.0015	<0.0020
5	0.0816	0.0796	0.0071	<0.0050	0.0074	<0.0015	<0.0020
⇑							
Ø	**0.0823**	**0.0818**	**0.0074**	**<0.0050**	**0.0062**	**<0.0015**	**<0.0020**
⇓							
SD	0.00065	0.00170	0.00109	0.00000	0.00133	0.00000	0.00000
RSD	0.79	2.08	14.77	0.00	21.60	0.00	0.00

	B [%]	Ca [%]
1	<0.0005	<0.0005
2	<0.0005	<0.0005

Appendix 3 316L OES analysis.



Appendix 4 X45CrSi9-3 full sample.



Measurement Results

Instrument 82E1078
Sample 1117
Alloy FE_300 **Mode** Element Concentration 24.10.2025 15:03:57

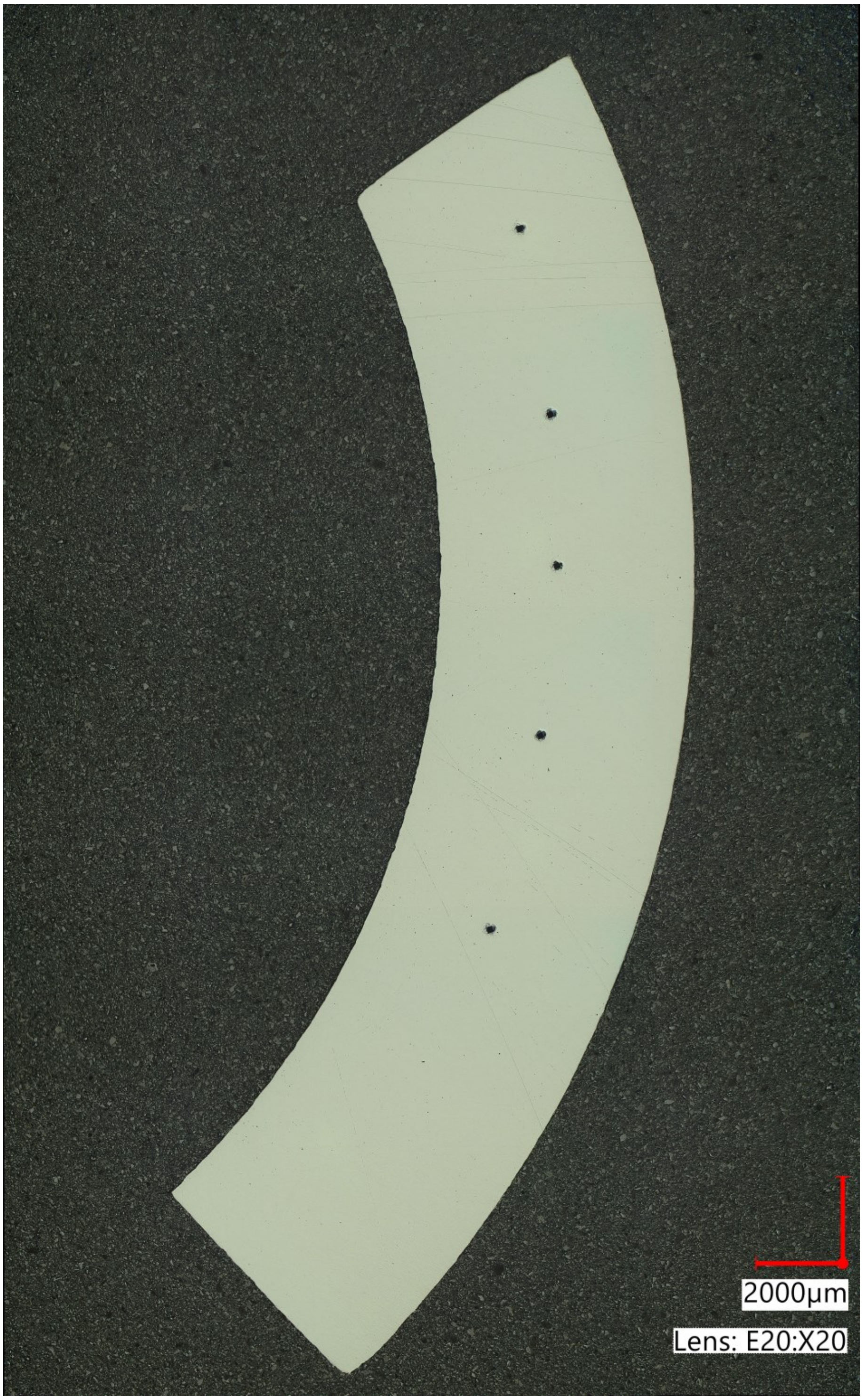
	Fe [%]	C [%]	Si [%]	Mn [%]	P [%]	S [%]	Cr [%]
1	87.2	0.386	2.97	0.394	0.0116	0.0041	8.70
2	87.2	0.383	3.00	0.392	0.0100	0.0036	8.67
3	87.2	0.380	2.99	0.393	0.0072	0.0021	8.69
4	87.2	0.380	2.98	0.393	0.0097	0.0035	8.69
5	87.2	0.378	2.97	0.396	0.0093	0.0038	8.75
⇑							
Ø	**87.2**	**0.382**	**2.98**	**0.394**	**0.0096**	**0.0034**	**8.70**
⇓							
SD	0.02	0.0032	0.012	0.0014	0.00157	0.00077	0.029
RSD	0.02	0.83	0.40	0.36	16.39	22.43	0.33

	Mo [%]	Ni [%]	Al [%]	Co [%]	Cu [%]	Nb [%]	Ti [%]
1	0.0503	0.157	<0.0030	0.0189	0.0695	<0.0025	0.0050
2	0.0473	0.139	<0.0030	0.0183	0.0690	<0.0025	0.0050
3	0.0478	0.132	<0.0030	0.0185	0.0690	<0.0025	0.0044
4	0.0470	0.128	<0.0030	0.0185	0.0693	<0.0025	0.0047
5	0.0459	0.124	<0.0030	0.0181	0.0696	<0.0025	0.0040
⇑							
Ø	**0.0477**	**0.136**	**<0.0030**	**0.0185**	**0.0693**	**<0.0025**	**0.0046**
⇓							
SD	0.00164	0.0132	0.00000	0.00028	0.00027	0.00000	0.00044
RSD	3.44	9.71	0.00	1.53	0.39	0.00	9.44

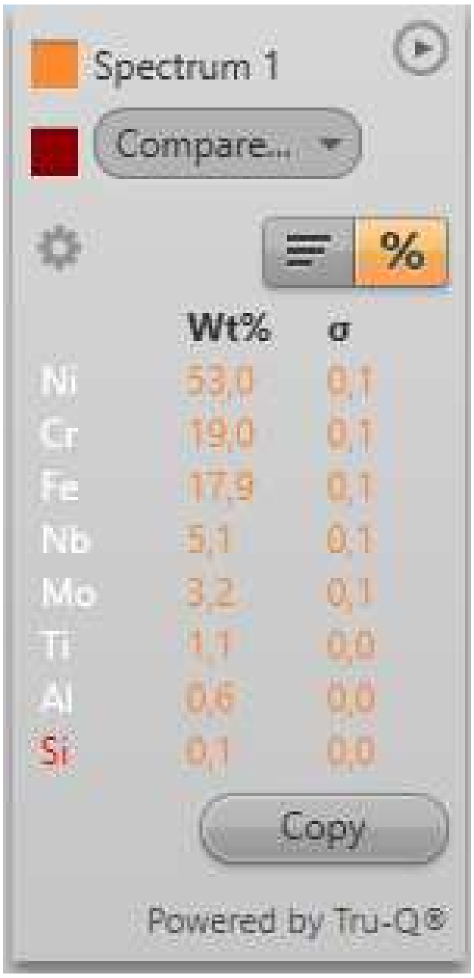
	V [%]	W [%]	Pb [%]	Sb [%]	Se [%]	Sn [%]	Zr [%]
1	0.0237	<0.0100	<0.0030	<0.0050	<0.0020	0.0063	0.0044
2	0.0239	<0.0100	<0.0030	<0.0050	<0.0020	0.0063	0.0044
3	0.0232	<0.0100	<0.0030	<0.0050	<0.0020	0.0042	0.0042
4	0.0233	<0.0100	<0.0030	<0.0050	<0.0020	0.0067	0.0050
5	0.0231	<0.0100	<0.0030	<0.0050	0.0022	0.0066	0.0047
⇑							
Ø	**0.0234**	**<0.0100**	**<0.0030**	**<0.0050**	**0.0020**	**0.0060**	**0.0046**
⇓							
SD	0.00037	0.00000	0.00000	0.00000	0.00007	0.00104	0.00031
RSD	1.57	0.00	0.00	0.00	3.34	17.28	6.85

	B [%]	Ca [%]
1	<0.0005	<0.0005
2	<0.0005	<0.0005

Appendix 5 X45CrSi9-3 OES analysis.



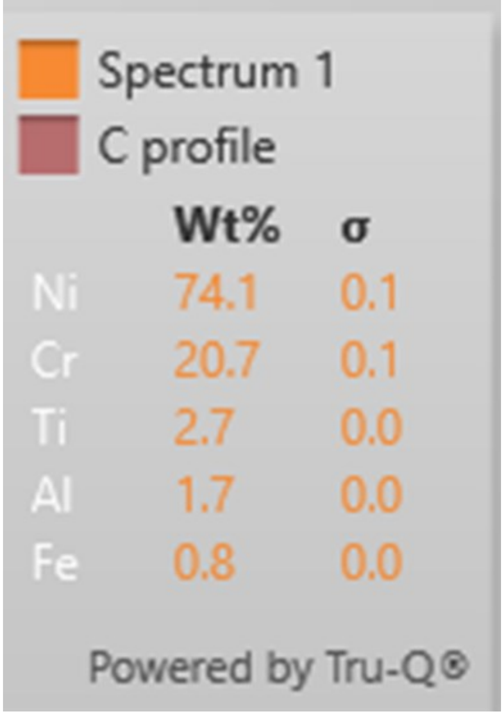
Appendix 6 Inconel 718 full sample.



	Wt%	σ
Ni	53,0	0,1
Cr	19,0	0,1
Fe	17,9	0,1
Nb	5,1	0,1
Mo	3,2	0,1
Ti	1,1	0,0
Al	0,6	0,0
Si	0,1	0,0

Appendix 7 Inconel 718 EDS analysis.

Appendix 8 Nimonic 80a full sample.



🟧 Spectrum 1
🟥 C profile

	Wt%	σ
Ni	74.1	0.1
Cr	20.7	0.1
Ti	2.7	0.0
Al	1.7	0.0
Fe	0.8	0.0

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Appendix 9 Appendix 6 Nimonic 80a EDS analysis.