THESIS – MASTER'S DEGREE PROGRAMME

TECHNOLOGY, COMMUNICATION AND TRANSPORT

MATERIALS RESEARCH LABORATORY

Implementation for educational use

Savonia UAS, Tekniikka Varkaus

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Abstract

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Abstract

Savonia University of Applied Sciences' Varkaus campus also teaches Materials Engineering, as part of the Mechanical Engineering subjects included in the Degree in Energy Engineering. Materials engineering has been a very theoretical subject, which needed to be supported by laboratory work, which can be used to combine theory with practice and to enhance and enliven the learning experience through it.

The basic knowledge of the theory already existed in the field of teaching, but more had to be done about the behavior of the materials and the crystal structures. Appropriate content for teaching with cycles was designed, which at the same time provided information on the required hardware with its features.

The preconditions for the materials research laboratory were already in place, including a suitable operating environment and previously acquired equipment. The layout of the laboratory had to be designed and implemented to be functional and at the same time safe. The equipment had to be installed and tested, which also saw the need to purchase new equipment. An alternative solution for studying and imaging the microstructure of materials was also investigated.

A practical learning environment supporting the teaching of materials technology was achieved, which supports the teaching of the author of the thesis and diversifies the content of the course. This is also a direct benefit for the research subscriber. During the work process, a contact surface was also obtained for the corporate sector for research, development and innovation (RDI) operations. It could therefore be concluded that the objectives of the work had been achieved.

Keywords

Crystal structure, Grain boundary, Grinding, Polishing, Etching, Hardness test, Impact resistance

PREFACE

This thesis research has been done to support the teaching of Materials Engineering on the Varkaus campus of Savononia University of Applied Sciences. The research is based on materials science and the interpretation of the crystal structures of materials. The research environment on the Varkaus campus has been very supportive and the cooperation with the staff and students has been pleasant. The research itself has been developing my own work and teaching methods. Therefore, I would like to express my humble thanks to the Savonia UAS Varkaus campus and especially to Mr. Olli-Pekka Kähkönen, PhD, experimental material physics and to Mr. Ari Mikkonen, an experienced lecturer in mathematics and chemistry.

Varkaus 29.3.2022
Arto Brask

CONTENTS

LIST OF FIGURES

1 INTRODUCTION

Based on the information researched in this thesis, the Varkaus campus of Savonia University of Applied Sciences has a functioning materials research laboratory, which can also be applied to RDI activities.

This thesis research report also serves as a guide for the Materials Research Laboratory of Savonia University of Applied Sciences' Varkaus campus.

At the beginning of the thesis report, material science is introduced as a theory, from which we move on to practical implementations and the user-oriented part with regard to different devices. At the same time, it is explored what the different methods are based on and what they effect for in the research of materials. Equipment purchases and quotations are also observed, although most of the basic equipment already existed on campus. The summary discusses the usability of the end result and whether the goals have been achieved.

2 EDUCATIONAL USE FOR MATERIALS RESEARCH LABORATORY

Savonia University of Applied Sciences also teaches mechanical engineering subjects in the Department of Energy Technology, of which materials technology and manufacturing and welding technology are related to the topic of the thesis. Of the other professional subjects mentioned above, mention should be made of technical mechanics and strength theory, as well as various courses related to design and design software.

In particular, the teaching of materials technology as a theory is not nearly as illustrative as if, in addition to the theory, practical experiments could be performed in laboratory conditions. In addition, there is an opportunity to increase study activity and interactivity with interesting laboratory exercises.

Recording the results of laboratory exercises also adds value to other areas of learning. In addition to the management of reporting methods, the preparation of laboratory reports also develops the processing of results and the illustrative presentation of results. The above things support the learning of communication technology. Skills in using different software are also evolving. Experimental methods as well as the observation of phenomena are also important elements in reporting the results of laboratory exercises. It is noted, therefore, that laboratory exercises are not limited to the properties of materials and the development of related knowledge.

2.1 Materials Engineering

Materials technology is based on different materials and their properties, which will be discussed in the next chapter. These properties and their changes can be studied, for example, at different temperatures and illustrated in teaching through laboratory experiments and exercises based on them. For example, a simple experiment in which the hardness of a particular material is measured with a Rockwell tester. It gives the hardness of the material in the current crystal structure. What if the part is hardened and the hardness is measured again? How much did the hardness increase? In addition, the sample can be modified and ground from a piece that can be etched if necessary, making the grain boundaries more visible.

2.2 Manufacturing and Welding Engineering

The basics of Manufacturing and Welding Technology are also taught at the Varkaus Campus of Savonia University of Applied Sciences. The teaching of welding technology laboratories requires appropriate fire work facilities. This possibility is also being explored. The welding facility also supports the research and development of damage analyzes in the materials laboratory.

3 MATERIAL SCIENCE

Materials technology and related knowledge of materials is a key area of expertise in product design and manufacturing. The structure and properties of the material determine the material's suitability for the researched application, also influencing the choice of manufacturing technique.

The equipment of the materials laboratory of Savonia University of Applied Sciences' Varkaus Campus is suitable for metal analysis. The main materials in the energy industry, as in many other industries, are various ferrous metals and their alloys.

3.1 Properties of materials

The main factors for the behavior and properties of metals (Sorsa 2015):

- Type of atom bonding (to metal structure)
- Natural internal energy minimization
- Crystal structure and deformation mechanisms (dislocation)
- Thermal vibrational motion of atoms in a structure
- Allotropy
- Attitude of metal structure to foreign atoms
- Behavior of foreign atoms in a metal structure

In the next subsection, more on crystal structure and allotropy.

3.2 Microstructure and phases of materials

Common metal crystal structures are space-centric cubic, surface-centric cubic, and hexagonal (Figure 1).

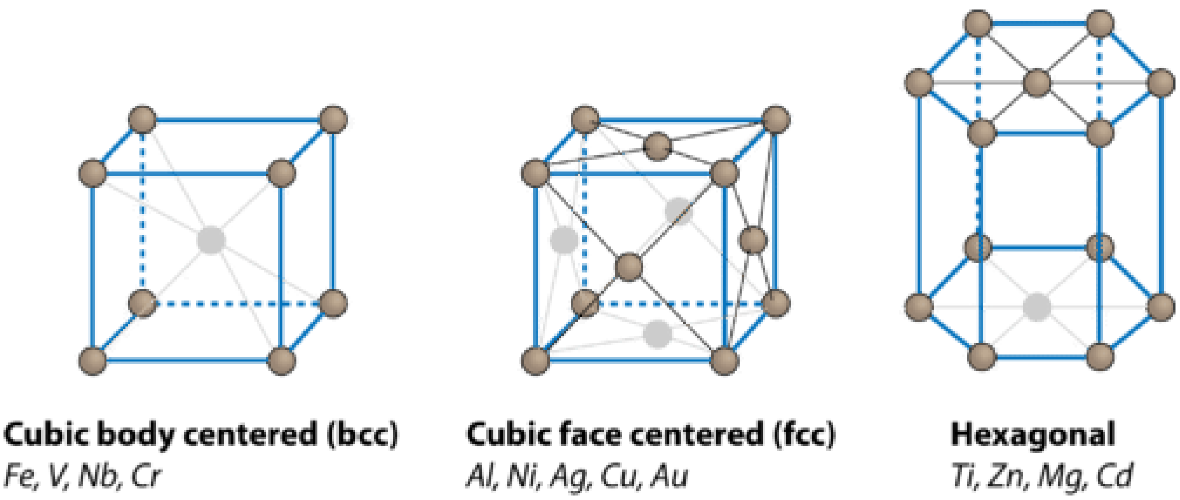

FIGURE 1. Crystal Structures of Metals (CK-12)

Focusing on the microstructure of steels and cast irons, which are the main materials in industrial applications.

In steels, the phases are degrees and types of organization of the crystal structure that are independent of the state. The phases of steel are Ferrite, Austenite, Cementite, Perlite, Bainite and Martensite.

The Iron-Carbon Equilibrium Diagram shows the temperatures at which the different phases are formed with respect to the carbon content (Figure 2).

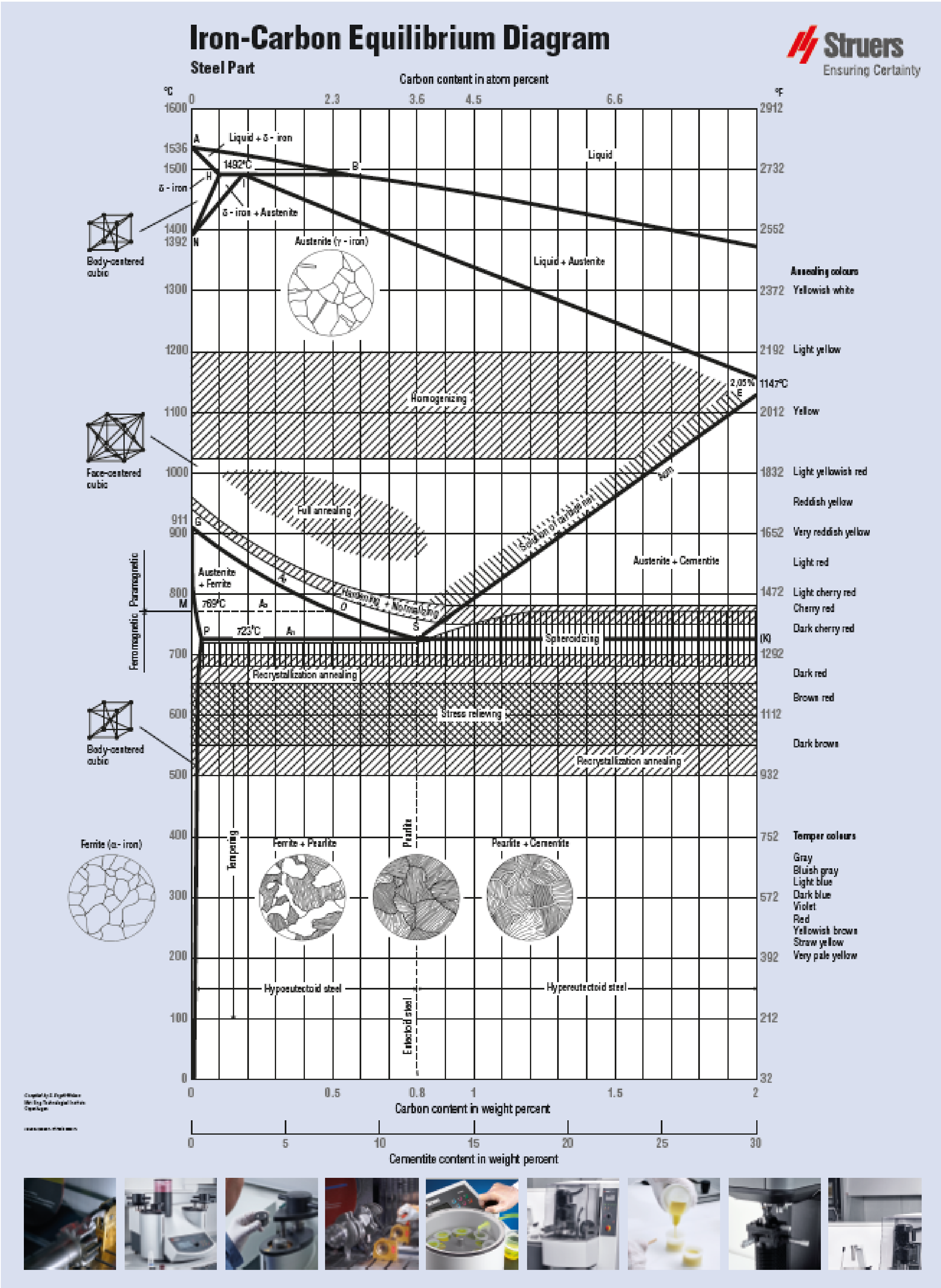

FIGURE 2. Iron-Carbon Equilibrium Diagram. Copyright Struers, not allowed to publish without Struers approval (Struers 2022)

An example is a micrograph of the microstructure of hypoeutectoid steel (Figure 3). Perlite (dark) on ferrite base (light). Presented as hot-rolled steel, resulting in a banded structure. (Struers 2022.)

FIGURE 3. Hypoeutectoid steel, Hot rolled (Struers 2022)

The allotropic properties of iron are detectable at different temperatures. When the temperature exceeds 910 °C, the crystal structure changes from Cubic body centered to Cubic face centered. (Hannula, Haimi & Lindroos 2020). When iron is alloyed with carbon, at said temperature the phase of the steel obtained by alloying is austenitic with a carbon content of 0.01 to 1.5 %. Steel microstructure with a carbon content of 1.5 %, Austenitized at 930 °C, quenched in oil and normalized at 350 °C (Figure 4).

Alloying steels greatly affects the allotropic properties of the material. There are a really large number of different steel alloys with standardized alloying and different national standards are comparable. (Verlag Stahlschlüssel Wegst GMBH 2019.)

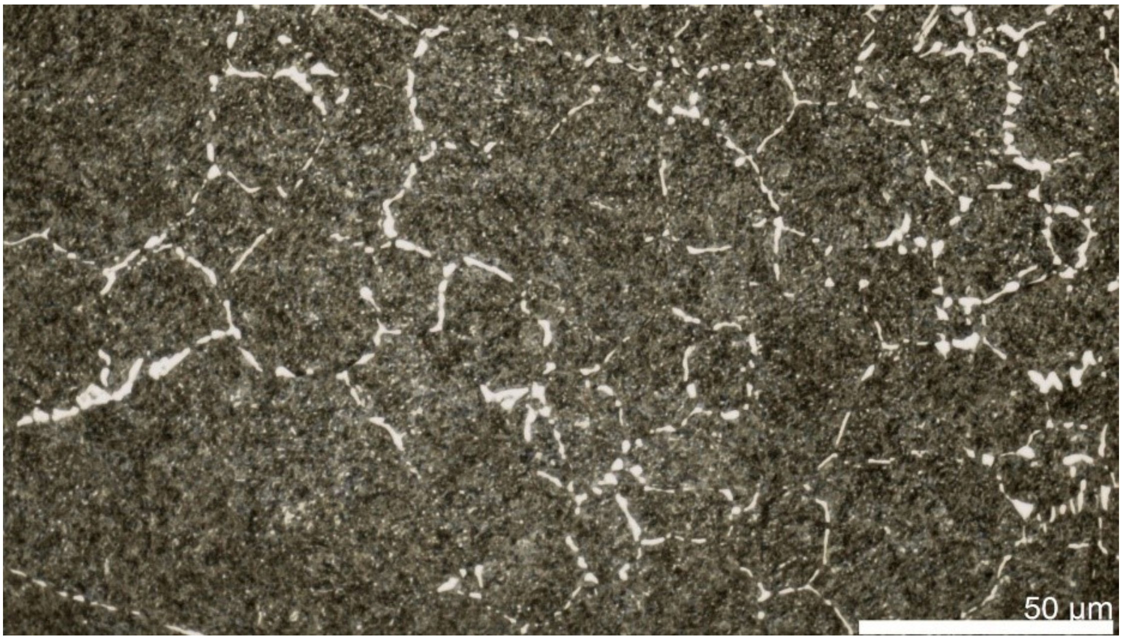

FIGURE 4. Grain boundary cementite (light) and martensite (dark) (Struers 2022)

The research in the materials laboratory is based on the above theory. The microstructure of a material determines the properties of the material and the results obtained by studying this indicate the behavior of the material in the problem that led to the study.

4 STARTING POINT

In the starting point, the laboratory facilities already exist. The equipment of the old materials research laboratory is stored in these rooms. Desks have been built for the equipment, as well as the necessary drainage for a grinding machine, for example. This is because in the past, it has been an idea over time to explore the uses of equipments in RDI activities.

As shown in Figure 5, some of the equipment, such as the impact test hammer, was stored on forklift pallets and storage boxes.

FIGURE 5. Impact test hammer in storage (Brask 2021)

In the initial phase, the progress of the thesis project would continue as follows:

The thesis project begins with an inspection of existing equipment and at the same time a preliminary assessment of its suitability for teaching use. The basic assumption is that suitability for educational use also indicates suitability for RDI activities if the activities performed with the device are included in commercial research at all. The project will be continued by creating a functional floor plan for the laboratory, after which the equipment can be installed. This is followed by a trial run of the equipment, which explains its use and corrects any faults and deficiencies. This provides information on the need for potential investment.

After the introduction of the equipment, the desired methods for teaching use are selected and cycles suitable for teaching are planned for the research. Studies are tested by completing them and assessing their suitability for teaching and the time spent completing them. As a result of the test runs, new procurement needs may arise.

Information on various testing methods, materials and laboratory practices is sought in the literature as well as in the local partners' own materials research laboratories.

5 LABORATORY EQUIPMENT AND ASSEMBLY

The following describes the equipment for laboratory research in materials research and their installation. Most devices were installed on the desktop, so they do not require larger mechanical installations. Behind the table was an electrical console ready, where the sockets can be placed in the necessary places. Inlet water and drain connections were required for the grinder and hot capsule casting machine.

Niskala (2021) said that Struers' equipment is of a high quality and Savonia's Varkaus campus equipment is modern and technically suitable for professional use. He checked the condition of our equipment and instructed us to use it. In addition, we received the latest information on related product packages and their features.

5.1 Impact test Hammer (pendulum impact)

The impact test hammer is a test device in accordance with standard SFS-EN 10045-2 with a pendulum of type C (Figure 6). The device performs impact tests on Charpy-C and Charpy-U test pieces. The actual impact test accordance with standard SFS-EN 10045-1. The hammer was installed in the space reserved for it, taking into account the safety of use with installing a safety cage around it.

FIGURE 6. Impact test hammer, Frank (Brask 2022)

5.2 Sample Cutter

From the material to be analyzed, the specimen is removed from the relevant site with a sample cutter. This type of cutter uses a cutting blade, the composition of which is selected according to the hardness of the material to be machined. The blade and the sample to be cut are cooled by the cutting fluid, which is pumped in the internal circulation of the device. The mower was installed on a solid level, taking into account the weight and height position of the machine based on usability (Figure 7). This unit uses 3-phase electrical power. The sample is attached to a screw clamp and after closing the protective cap, the device can be started, at which point the cutting fluid begins to flow and the blade is transported through the sample by turning the lever.

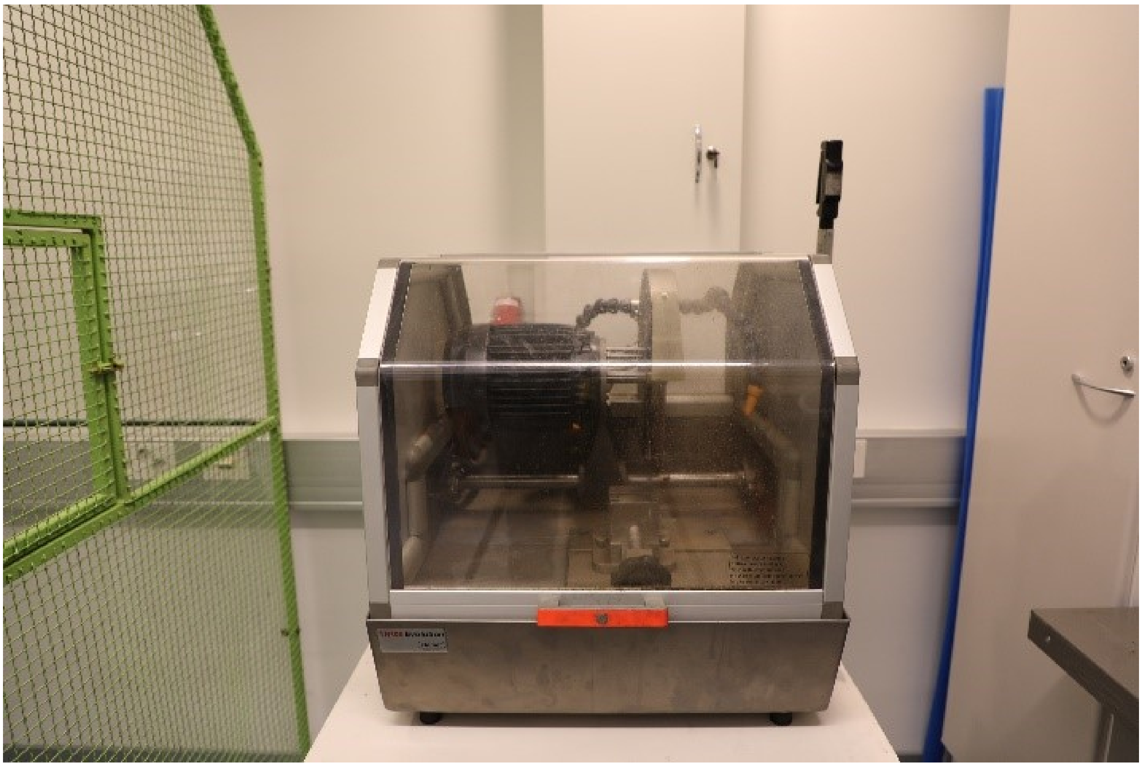

FIGURE 7. Sample cutter, Remet (Brask 2021)

5.3 Hardness tester (Rockwell)

Figure 8 shows a hardness tester that measures the hardness of a material on a Rockwell scale. Rockwell C hardness is obtained with a diamond cone test tip and Rockwell B hardness is obtained with a steel ball test tip. The hardness tester also includes different models of anvil for samples of different shapes. The Rockwell hardness scale is comparable to Brinell and Vickers hardnesses. The sample is placed on the anvil and the pre-force is adjusted by tightening the sample against the test tip of the anvil transmission, after which the device produces the required force for the test. The result can be read on the device screen and printed with a ribbon printer integrated into the device or saved directly to a computer for storage and reporting.

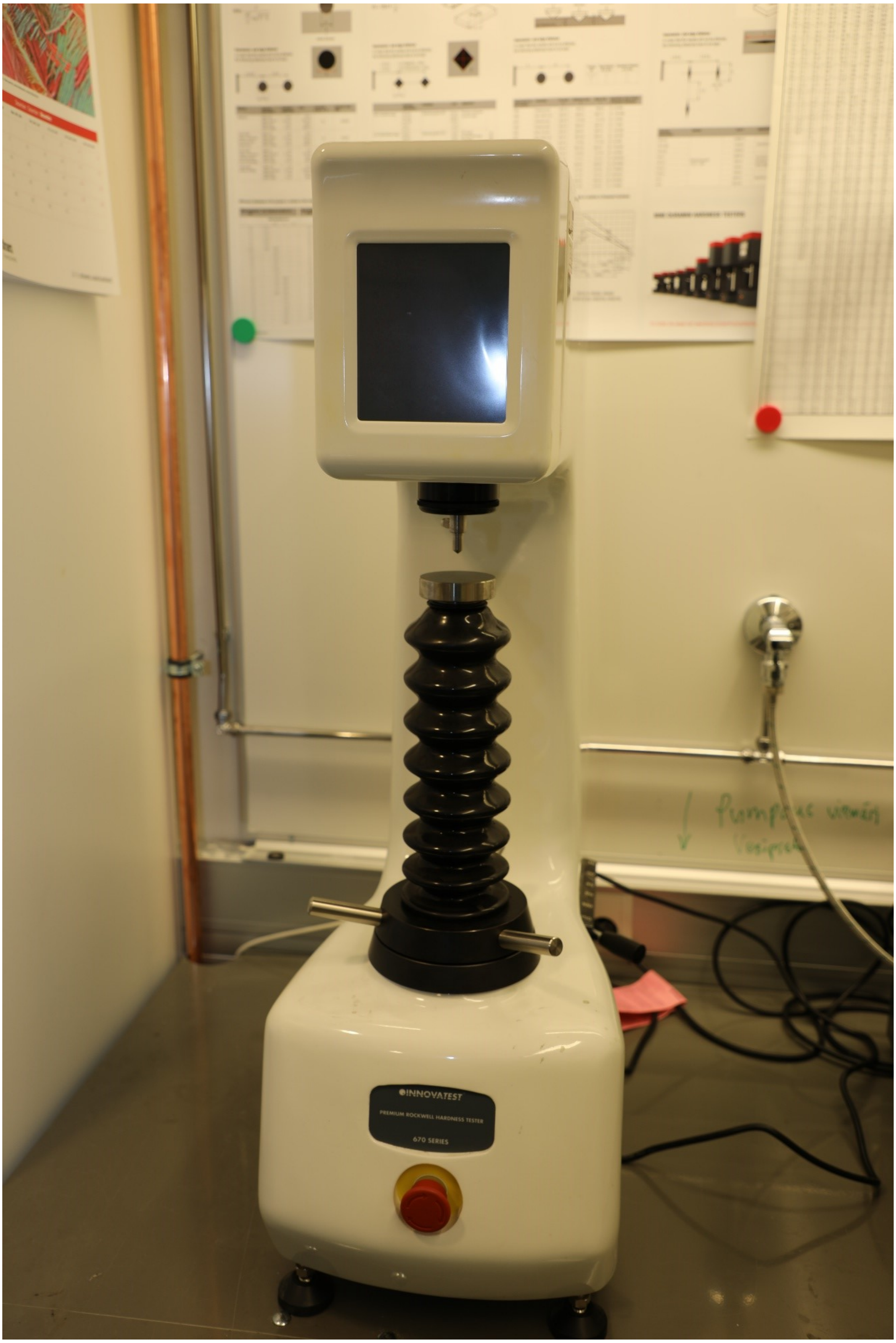

FIGURE 8. Rockwell hardness tester (Brask 2021)

5.4 Sample Grinder

The sample grinder (Struers TegraPol-21) was installed on a sturdy work surface with water connections and drainage. The lifting cylinder of the grinder's sample holder arm and the fingers of the sample holder need clean compressed air (this part of the device is TegraForce-5), for this purpose filters and an oil-free compressor with a sound-absorbing housing were installed. The grinder can be pre-programmed with cycles for the grinding methods required by different materials, as well as with the emulsion dosing pump (TegraDoser-5) connected to the grinder. Grinding of conventional steel samples requires four different methods, which are run automatically according to pre-programming. The grinding wheels adhere to the magnetic base. Diamond blades were chosen as the blades for the coarser grinding methods because of their durability and good efficiency. Finer grinding methods are performed with fabric discs. As an abrasive emulsion in the coarse process water and in the finer various diamond suspensions and aluminum oxide suspensions. The dosing pump was also programmed with a washing program guided by Juha Niskala from Struers (Figure 9).

It was found that in the studies included in the students' course, the pre-programming of the parameters of complex methods is especially important in order to maintain attention in the study of the sample itself.

FIGURE 9. Juha Niskala/Struers with Degra Doser (Brask 2021)

5.5 Sample encapsulation machine

Figure 10 shows the hot encapsulation molding machine installed. A sample and a capsule-forming granulate are placed in the encapsulation cylinder. Heating is provided by resistors and cooling by a water circuit, for which water and drain connections were installed. The general temperature of the encapsulation process is 250 °C. During the heating and cooling cycle, the encapsulated sample is compressed by a hydraulic cylinder at a maximum process pressure of 300 bar. Immediately after the cooling cycle, the sample can be removed from the device and ready for grinding.

FIGURE 10. Encapsulation machine, Struers (Brask 2022)

5.6 Heat treatment oven

The hardening oven was also installed on a solid and heat-resistant base (Figure 11). The maximum oven temperature is 1200 °C. The oven can be pre-programmed with heating, holding and cooling cycles as a function of temperature and time.

FIGURE 11. Heat treatment oven, Bartlett (Brask 2022)

5.7 Dynamic stress tester

In Figure 12 is the dynamic stress tester. This old device was introduced as the only laboratory device to study the test piece by exposing it to a constantly changing load. So this is a fatigue test. It rotates a dimensionally accurate test rod with an electric motor equipped with a tachometer and loads radially by pressing through a roller.

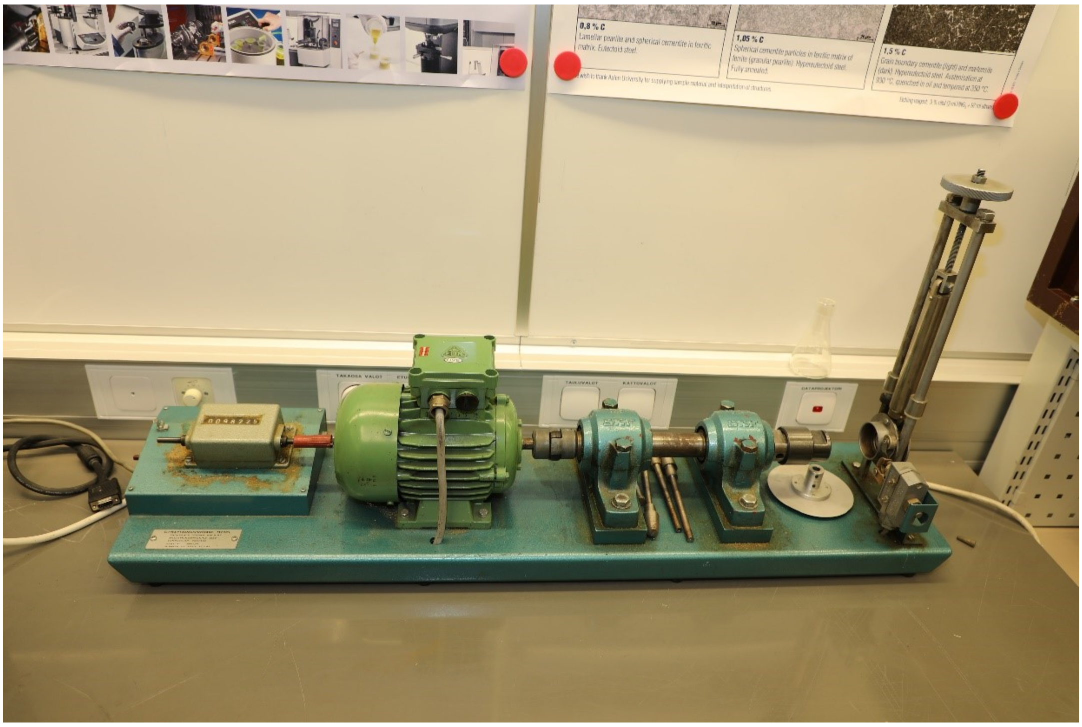

FIGURE 12. Dynamic stress tester (Brask 2021)

5.8 Etching station and ventilated work cabinet

The etching of the ground sample is performed in an air-conditioned cabinet as shown in Figure 13. The cabinet was preferably obtained with very little use, mainly as a sample. The installation of air conditioning and drainage was commissioned by Savo vocational college students. Etching chemicals are stored in an air-conditioned acid cabinet (Figure 14).

FIGURE 13. Installation of air-conditioned cabinet (Brask 2021)

FIGURE 14. Storage cabinet for etching chemicals (Brask 2021)

5.9 Microscopes

An optical microscope with a camera with a maximum magnification of 50 times was chosen as the main microscope (Figure 15). Through the camera, the image is viewed on a computer screen and the image can be enhanced and saved. It is also possible to save a series of images.

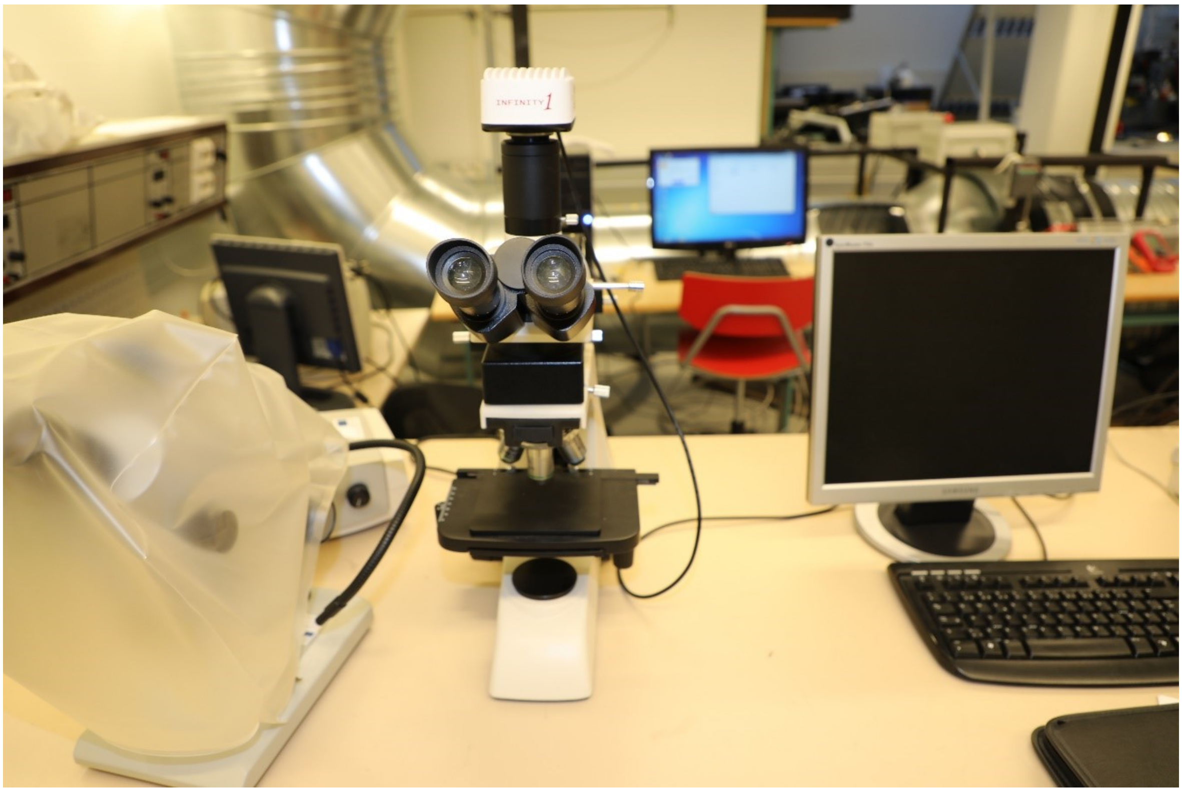

FIGURE 15. Microscope with a camera (Brask 2021)

6 LABORATORY LAYOUT

It follows how the laboratory layout was formed.

6.1 Layout functionality

Functionality was sought in the layout of the laboratory by placing the equipment centrally, however considering sufficient functional distances. Safety was taken into account so that those working with different devices do not endanger each other. In addition, the laboratories are performed in small groups, which increases not only the quality of teaching but also safety. Movement in the vicinity of the equipment must be unobstructed because the transitions between the phases must be able to be handled smoothly.

6.2 The master layout of the laboratory

The placement of the equipment was a horseshoe-like environment to ensure the best transportability. At the same time, the equipment is well accessible and observable, taking into account the ongoing process. For example, the next encapsulation can be made to process at the same time as the previous one is in grinding (Figure 16). The etching is performed in the Energy Technology Laboratory. This avoids excessive movement in the vicinity of the process of handling highly corrosive chemicals.

FIGURE 16. Overview of the laboratory (Brask 2022)

7 PREPARATION OF THE SAMPLE

Sample preparation is the roughest step in sample handling.

7.1 The shape of the test piece

The sample is cut with a sample cutter from a larger sample (Figure 17). The size is chosen so that the sample can be encapsulated without getting too close to the walls of the encapsulation cylinder. The minimum clearance is 3 mm. An area is selected for the sample where the test factors are detectable, for example, the weld cross-section / damage area.

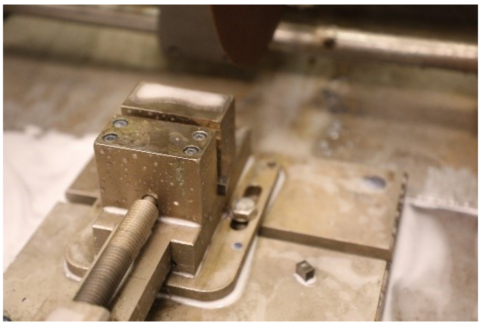

FIGURE 17. Sample cutting (Brask 2021)

7.2 Encapsulation of the test piece

Particularly good purity must be observed at the various stages of sample handling. It is important that the attachment of the encapsulation cylinder to the casting cylinder is complete and that the indicator arrows indicating it are aligned as shown in Figure 18.

Anti-stick powder is applied to the walls of the encapsulation cylinder to prevent adhesion of the encapsulant (Figure 19). The sample prepared in Figure 20 is well cleaned and placed in the raised piston of the casting cylinder. The piston is lowered and the hot mounting resin granules into a cylinder on top of the sample (Figure 21). The cylinder cap is placed on its support arm at the top of the cylinder and lightly tightened into its threads (Figure 22). For encapsulation, e.g. Multi-Fast encapsulant consisting of bake-lite and wood chips. For this method, the process parameters are set to a heating temperature of 180 °C, a heating time of 3.0 minutes, a compression pressure of 300 bar and a high cooling time of 2.0 minutes (Figure 23). After the cooling period, the cylinder cap is removed from its threads and lifted via the encapsulated sample by the piston and turned to the side by its support arm, whereby the encapsulated sample can be removed from the top of the piston. Figure 24 shows an encapsulated sample for grinding.

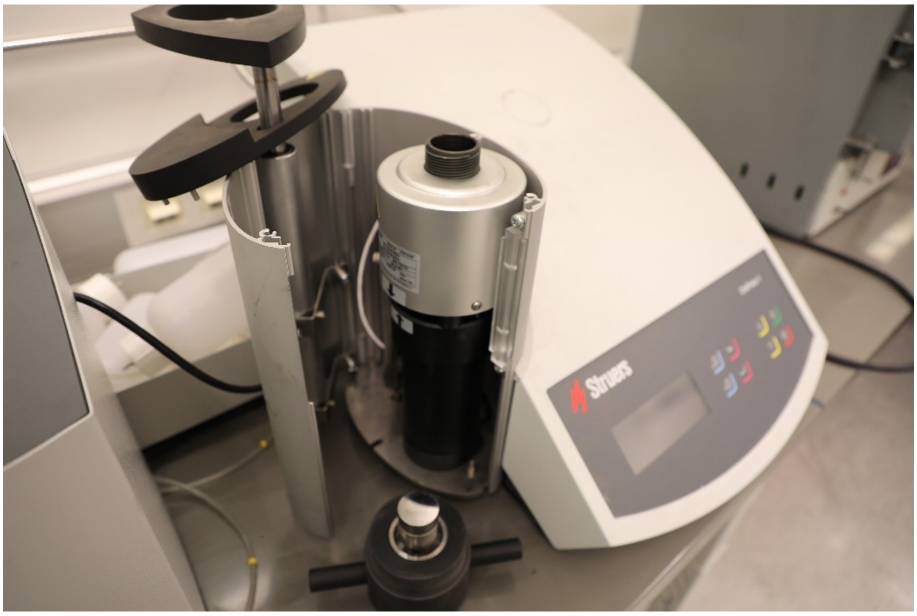

FIGURE 18. The indicator arrows are aligned (Brask 2021)

FIGURE 19. Cylider cap with anti-stick powder and scraper (Brask 2021)

FIGURE 20. Sample on the raised piston (Brask 2021)

FIGURE 21. Hot mounting resin cranules, Stuers MultiFast (Brask 2021)

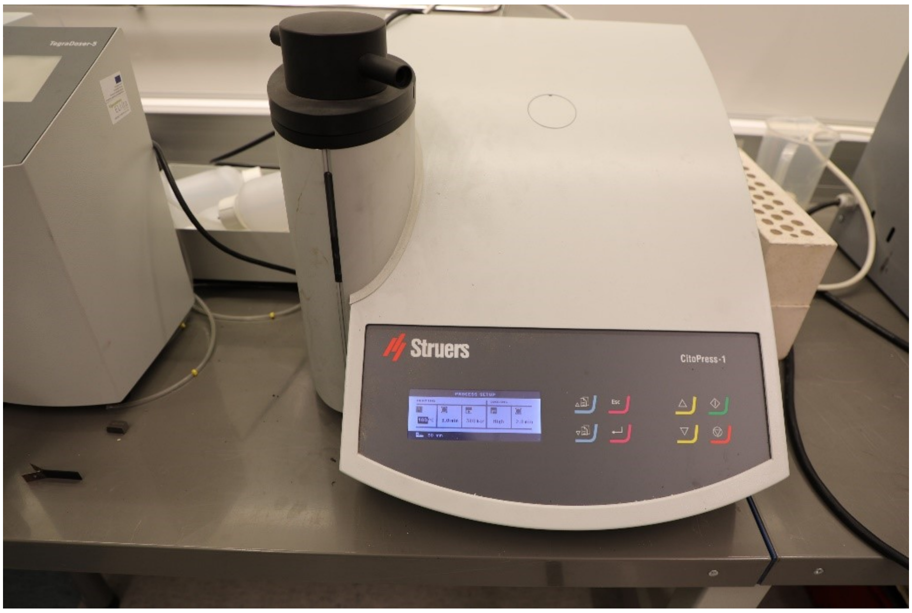

FIGURE 22. Ready for the encapsulant process (Brask 2021)

FIGURE 23. Process parameters (Brask 2021)

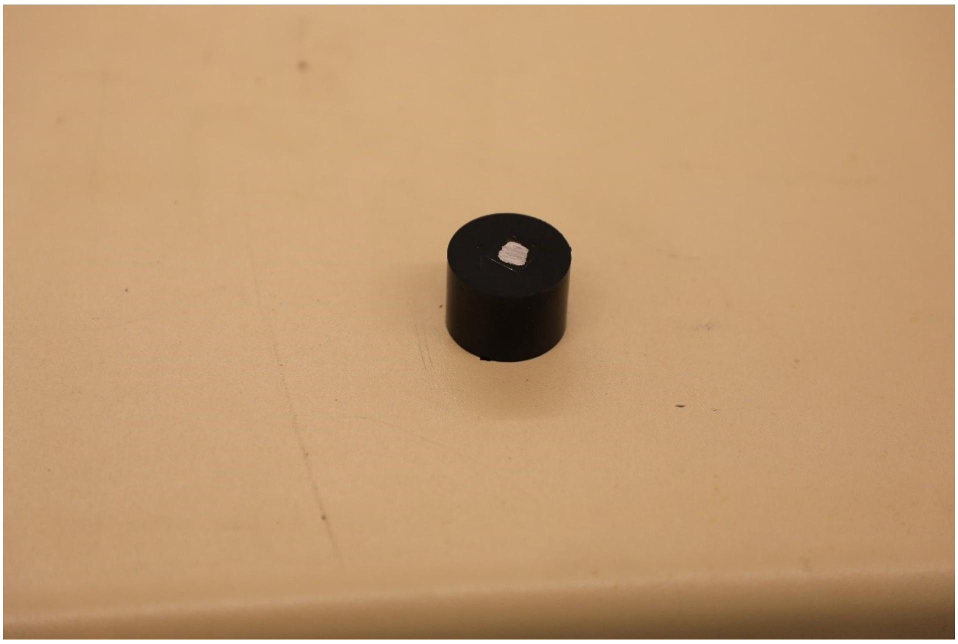

FIGURE 24. Encapsulated sample for grinding (Brask 2021)

8 GRINDING AND ETCHING THE TEST PIECE

Prior to microscopic examination, the sample must be grinded and etched to give a clear view of the grain boundaries.

8.1 Grinding

The methods of grinding are determined by the hardness of the sample. The methods have been tabulated by Struers and the correct method is found by measuring the hardness of the sample and comparing it with the above table. In our example case presented below, the hardness of the sample is measured with the hardness of our hardness tester on a Rockwell scale of 35 HRC.

In Figure 25, we see how the sample is ground with a diamond blade with cooling water with a blade roughness of 220 µm. This method begins the grinding of a sample in our example. The grinding wheels are magnetically attached to their base (Figure 26).

FIGURE 25. Rough grinding with a diamond blade (Brask 2021)

FIGURE 26. Placing the grinding wheel on the magnetic base (Brask 2021)

After the diamond blade shown in Figure 27, a change to a finer diamond blade is made using the suspension during the grinding process. Thus, the next cycle is performed using the MD Allegro method, in which a diamond suspension with a grain density of 9 µm is dispensed onto a fine diamond blade (Figure 28). The dosing of the suspension is handled programmatically by DegraDoser-5. At the end of the grinding cycle, the Allegro blade is not rinsed, as the grinding power of the blade remains constant when the suspension is allowed to soak into the surface of the blade.

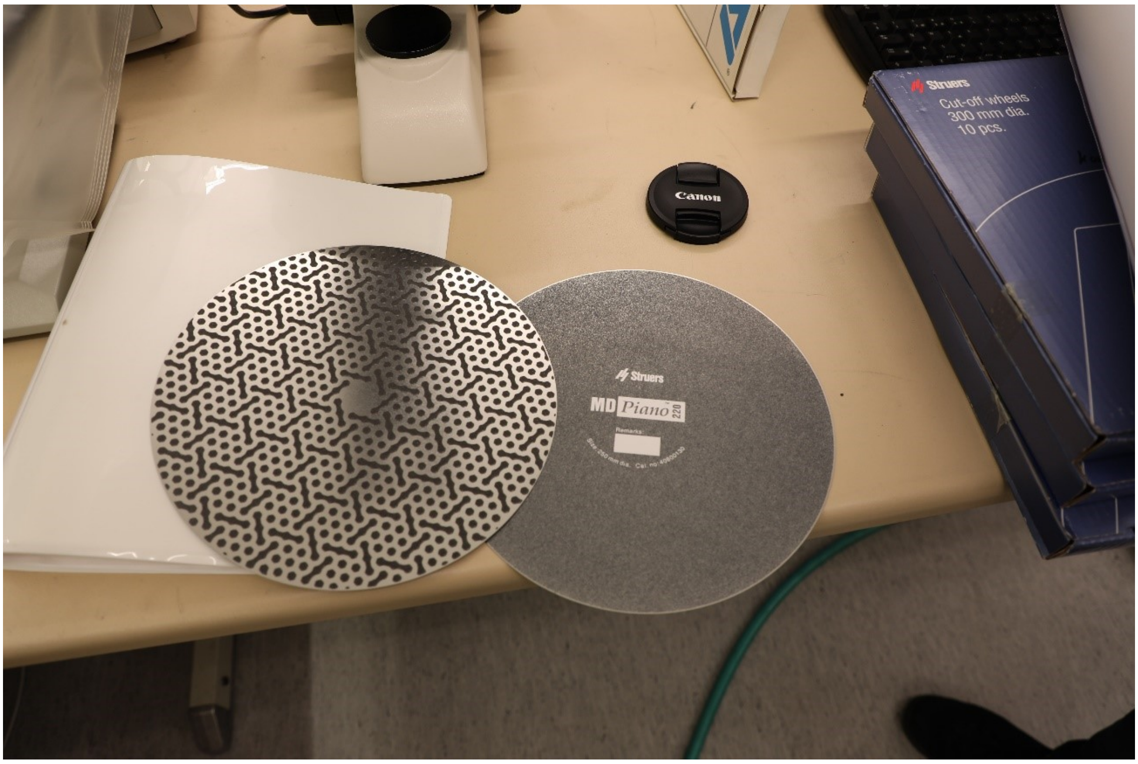

FIGURE 27. MD Piano -method diamond blade 220 µm, Struers (Brask 2021)

It is important that after each grinding cycle, the plate of the sample holder and the sample are rinsed under running water. This prevents the coarser material from getting scratched in the next, always finer grinding cycle.

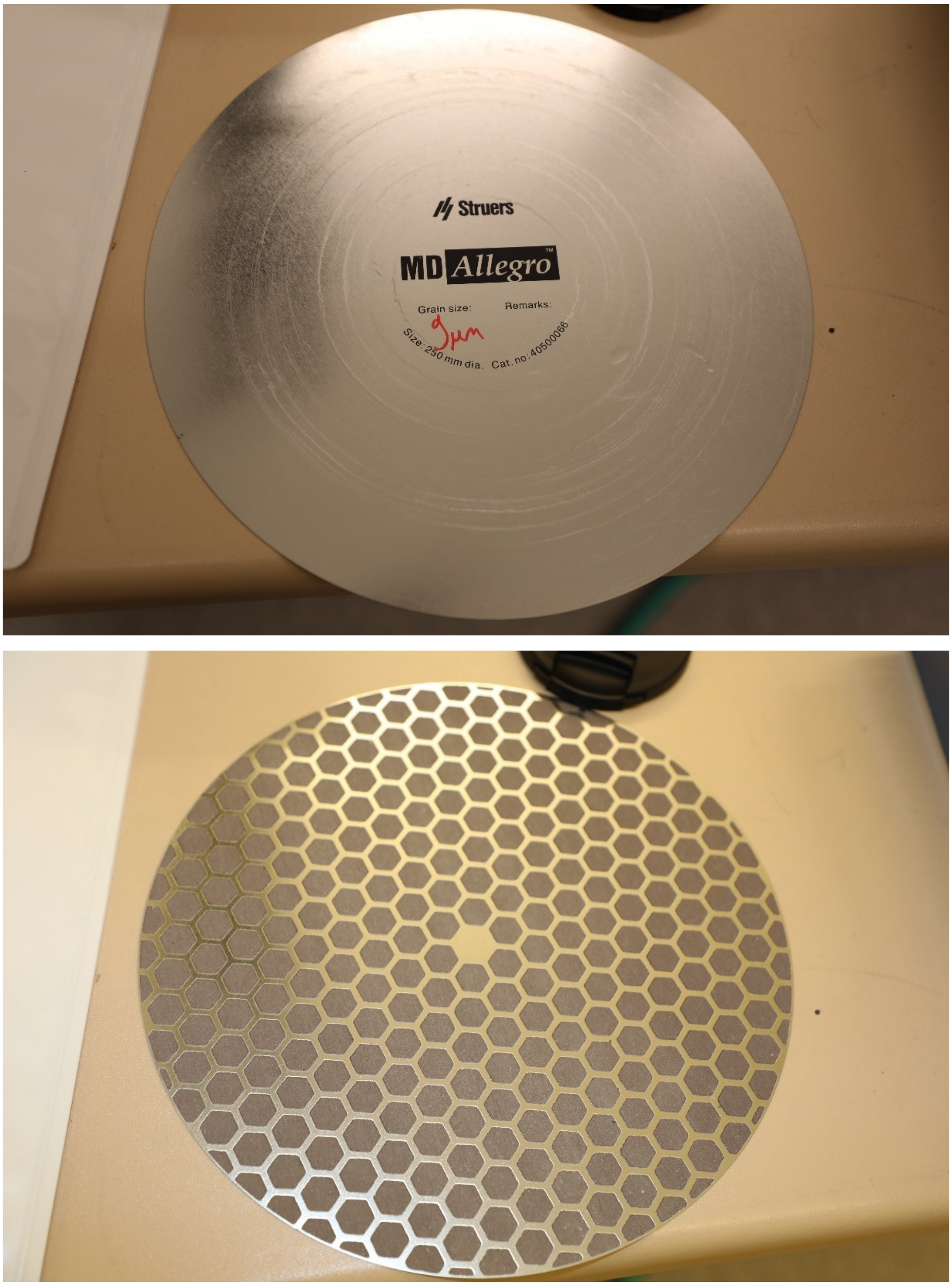

FIGURE 28. MD Allegro -method diamond blade, Struers (Brask 2021)

The next two grinding cycles are mainly polishing because the grinding discs have a fabric surface. Figure 29 shows the MD Dac method disc. In this method, a 3 μm grain size diamond suspension is dosed. The blade is not rinsed after performing the phase but the sample holding plate and sample are rinsed again. The grinding discs and the suspensions used in the different methods are identical in their names, so that the placement of the correct chemical in the dosing pump is clear. The doser has several pumps and each has its own suction hose, which is screwed onto the suspension canister and that pump is programmed to use the method cycle.

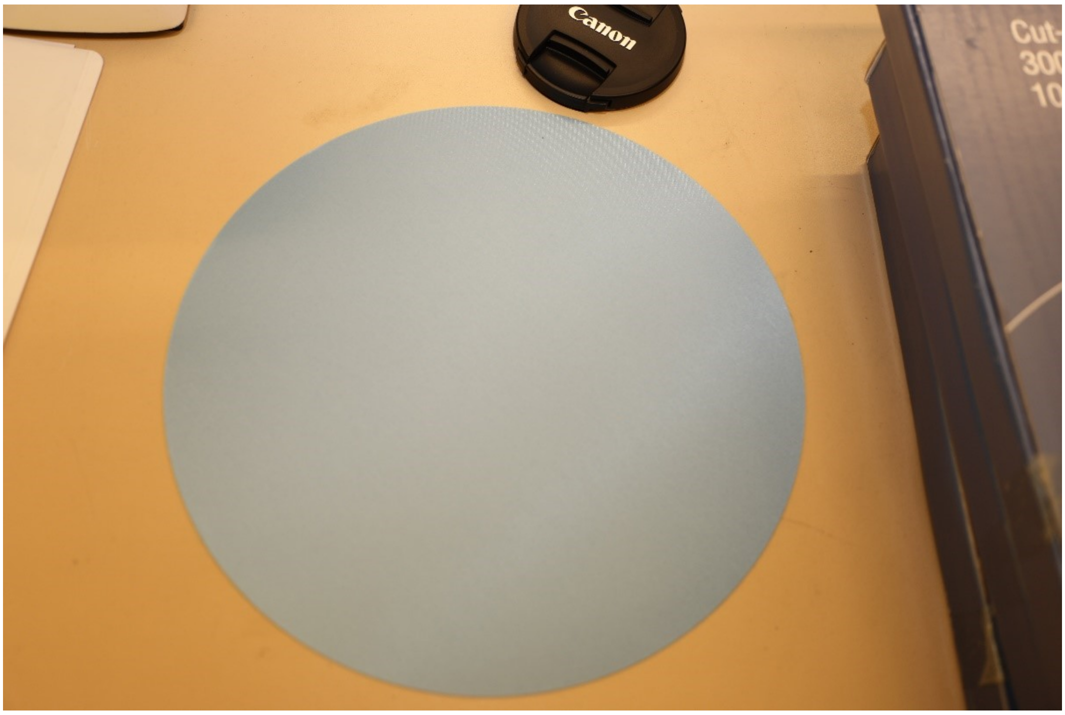

FIGURE 29. MD Dac -method fabric blade, Struers (Brask 2021)

In the last cycle, the MD Chem method is used (Figure 30). Aluminum silicate and a 1 µm rough diamond suspension are added to the disc. The cycle of this stage includes an automatic flush to flush the dosing pump and its hoses. The aluminum sulphate hardens and thus clogs the dosing channels and hoses, so that the dosing suction hose is placed in clean water during the rinsing cycle. In addition, the doser takes clean water from the water mains to flush the pressure side hoses.

FIGURE 30. MD Chem -method fabric blade, Struers (Brask 2021)

The polished sample is washed with water and dried immediately to prevent corrosion. As shown in Figure 31, the result is scratch-resistant and shiny.

FIGURE 31. Polished sample (Brask 2021)

8.2 Etching

The polished sample is etched according to standard CEN ISO / TR 16060. The etching method causes the grain boundaries visible under microscopic examination. This is based on the more sensitive corrosion of ferritic grain boundaries in carbon steels compared to, for example, martensitic grains. Even at mild acid concentrations of about 2 % in the alcohol base, etching is obtained in a few seconds enough for the microstructure to be observed under microscopic examination (ISO/TR 16060:2003). Different alloys need a different etching, which will be returned to in more detail in the subsection. The etching of the sample is monitored visually and, when the surface begins to darken, it is rinsed with purified water and then with ethanol.

Etchings are highly corrosive in all cases and special safety precautions must be taken when handling them. Vapors must also be safely removed from the work area, making a ventilated cabinet essential for etching (Figure 32).

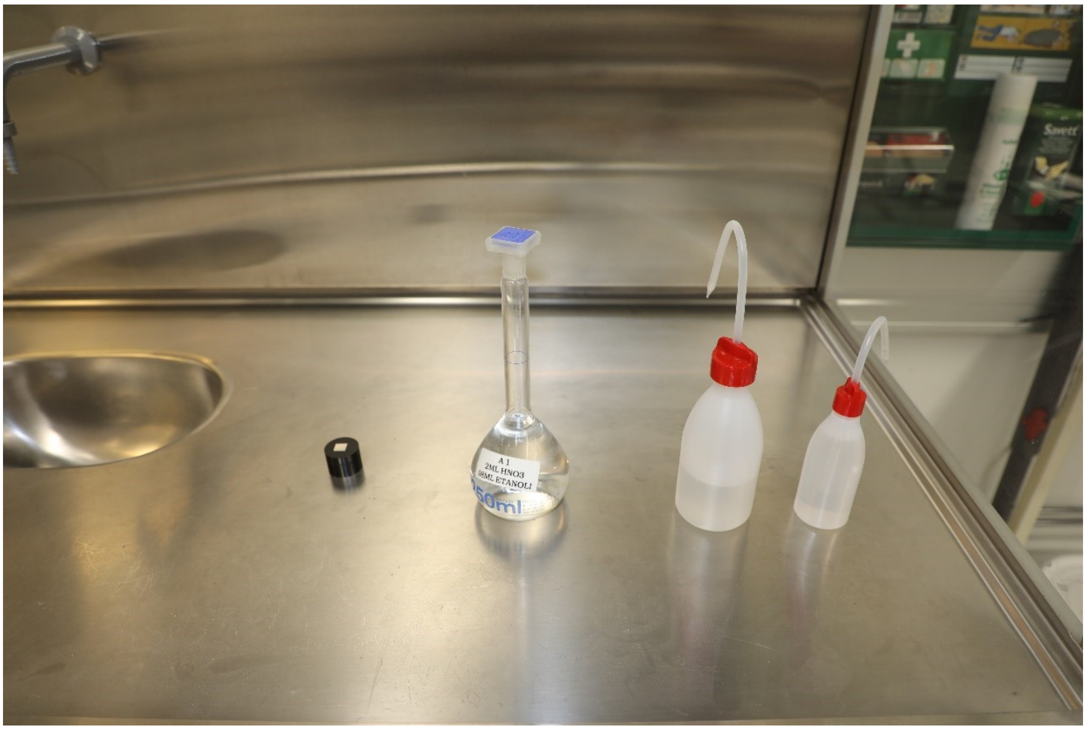

FIGURE 32. Etching operation in a ventilated cabinet (Brask 2022)

8.2.1 Etchings

Different alloys require different etchings. Figure 33 shows the most common etchings used laboratory. A1 is called Nital acid with 2 % nitric acid in ethanol. This etch works well for "black" carbon steels, like for structural steels. The B8 type etch is for stainless steels and the Marbles reagant is for nickel alloy stainless steels.

The content of nitric acid obtained was 65 % and that of hydrochloric acid 37 %, respectively. Ethanol alcohol content 95 %. The volume percentage of copper sulfate in the Marbles reagant recipe was determined based on aqueous copper sulfate. The copper sulphate obtained in the laboratory was anhydrous, so the correct amount had to be calculated on the basis of molecular weights.

The liquid substances to be mixed are dispensed into the solution bottle using precision pipettes and powdered substances such as copper sulphate using a balance and a laboratory spoon. The pipettes used for mixing and other equipment in contact with the substances to be mixed are washed with a neutralizing detergent and rinsed and dried thoroughly.

The mixing of etchings must be done with precision and safety must be taken into account. When mixing acids and ethanol, the behavior and warming of the solution should be monitored. Mixing is safest in a ventilated cabinet, taking into account the same safety concerns as when handling corrosive substances in general. Acid-resistant rubber gloves, protective clothing and eye / face protection must be worn. The vapors emanating from the acid bottles are so intense that they must not be smelled; this can result in painful pain in the respiratory system which, as a result of the incoming reaction, can cause the acid bottle to come loose and further damage to occur.

FIGURE 33. Etchings (Brask 2021)

9 MICROSCOPIC EXAMINATION

Microscopic examination reveals the grain structure at the phase level. In the same context, the mechanism of damage can be studied, for example. In connection with welding, various errors can occur, which can be e.g. serum from the incorporation of impurities into histometallic metal or changes in the weld metal during solidification. An example is the solidification crack, which can also be examined microscopically from a sample. (Lippold 2009.)

9.1 Preparation for microscopic examination

The dust cover on the microscope is removed and the computer running the camera software is turned on. After the microscope light source and program start, place the etched sample on the microscope sample table and refine the image by adjusting the focal length on the coarse / fine adjustment handwheels.

9.2 Interpretation of microscopic examination

Etching reveals the grain boundaries and grain structure as previously mentioned. Figure 34 shows the grain structure of a sample taken from carbon steel, martensite on a cementite base. Cementitic grain boundaries appear light and martensitic grains appear dark. The view is focused at 50x magnification under the microscope. The view of that image shows the full screen with the software interface, but screenshots can be taken individually or in timed series. Color / brightness balance can also be adjusted programmatically. The microscope itself also has light-modifying filters for the light source.

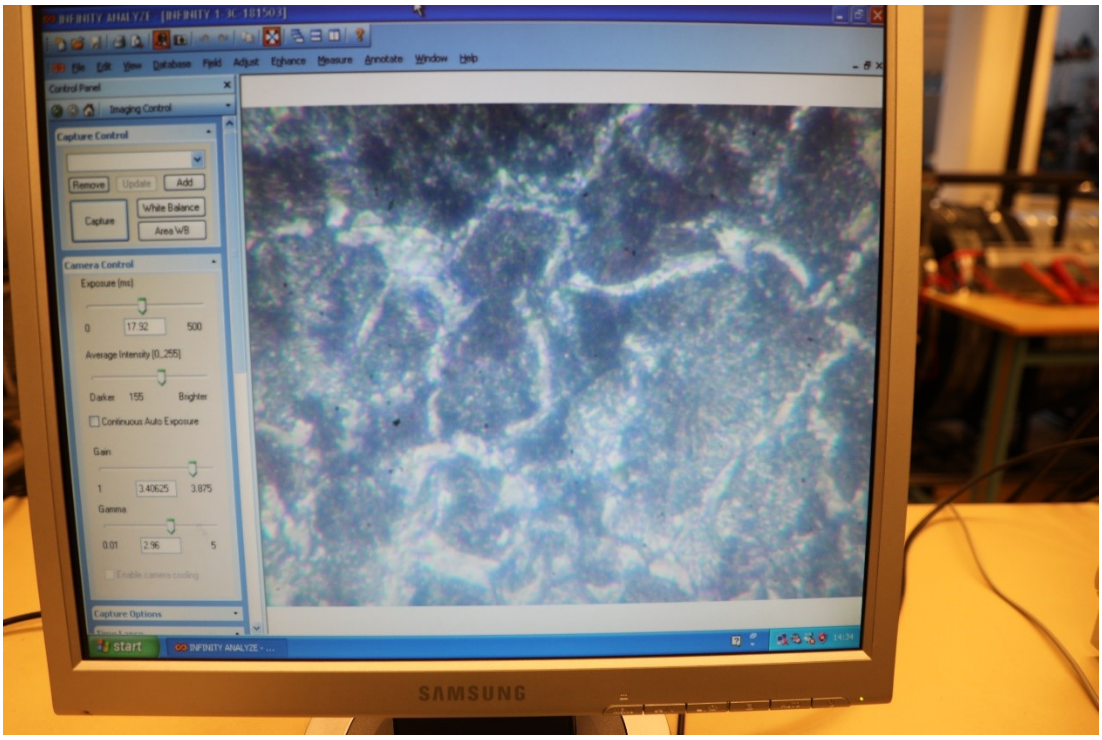

FIGURE 34. Microscopic view (Brask 2022)

10 HARDNESS TEST

The hardness of a material is its ability to resist an external object penetrating it, which also includes cutting, wear, and scratching. However, hardness is not a property of the material, but depends on several other properties of the material. The above-mentioned properties include e.g. tensile strength, toughness, modulus of elasticity. Impact resistance is also affected, although neither is in itself a property of the material. (Sorsa 2015.)

10.1 Rockwell hardness test

There are various methods for measuring hardness, the main ones being the Rockwell, Brinell and Vickers hardness test. These methods are comparable using a conversion table. The hardness tester at the Savonia University of Applied Sciences' Varkaus campus material research laboratory is based on the Rockwell hardness test. The equipment of the hardness tester includes various tips with which the sample is tested and anvil on which the sample is placed. Using a steel ball test tip gives hardness on the HRB scale and diamond cone using the HRC scale.

The hardness tester method selector is set to R and the pre-force to 150 when the HRC hardness is tested. The sample is placed on the anvil and the hand lever is used to tighten the pre-force to the position indicated by the sliding bar on the display. When the correct pre-force is reached, the machine locks the hand levers and the actual testing starts automatically after 4 seconds. The result is visible on the screen and can be printed with a ribbon printer integrated in the device (Figure 35). Previously mentioned printout shall include the results of all measurements taken after the list has been reset. The results can also be saved directly to a computer if the device is connected to the computer with a USB cable and the necessary software is installed.

Before starting the actual test series, it is important to test the hardness tester on a similar material a few times to determine the repeatability of the measurement. Especially if the measuring tip and / or anvil have been removed or replaced, settling in may cause the measurement result to deviate at the beginning. The calibration pieces supplied with the device may be used from time to time to check the accuracy of the measurement, and official calibration must be carried out by an official testing institute every year.

FIGURE 35. Rockwell hardness test ongoing (Brask 2022)

11 IMPACT TEST

The toughness of a material is determined by an impact test. The toughness of metal materials varies at different temperatures. The amount of impact energy from the kinetic energy of the pendulum test hammer to break the test piece is read from the scale. Based on this, the breaking energy of the test material is known, which is reported in Joules.

11.1 Preparation of the test rod

The test rod is manufactured in accordance with standard (SFS-EN 10045-1: 1990). It measures 10x10x55 mm and has a V-shaped or U-shaped center, the dimensions of which are also specified in that standard. The V-model notch is most often used. The notch shapes are named either Charpy V or Charpy U. However, the most commonly used Charpy V test is used.

11.2 Testing Impact resistance

When a standard test rod has been made from the material to be tested, the actual test is performed. The test bar is placed against the supports of the impact test hammer, the notch being exactly in the center of the dimension between the supports, with a tolerance of ± 0.5 mm (Figure 36). The direction of the notch is away from the pendulum, i.e. the pendulum strikes the opposite side of the notch. A normal impact test without a specified temperature is performed at normal room temperature ± 23 °C. (SFS-EN 10045-1: 1990).

The pendulum of the impact test hammer is raised from the handwheel to the position where the pendulum pointer shows the marking on the instrument gauge at about 11 o'clock. and the indicator of elapsed impact energy is turned from the knob in the center of the meter to indicate the zero point of the degree scale at six o’clock. The pendulum is released by pulling the trigger lock knob and turning the trigger, before doing so, however, care must be taken to ensure that the range of the pendulum is unobstructed. The test rod is intended to break due to the impact force of the pendulum, after which the pendulum is stopped by depressing the brake lever. The energy required to break the test rod can be read from the inside scale of the meter when no additional pendulum weight is installed (Figure 37). When reporting the result in Kiloponds, the result is converted to Joules by multiplying the fall acceleration by 9.81 m/s².

The impact testers have their own hatches in the protective cage through which they can be accessed. The same applies to the setting of the test rod and the collection of its broken halves, which may be assisted by a telescopic magnet if necessary.

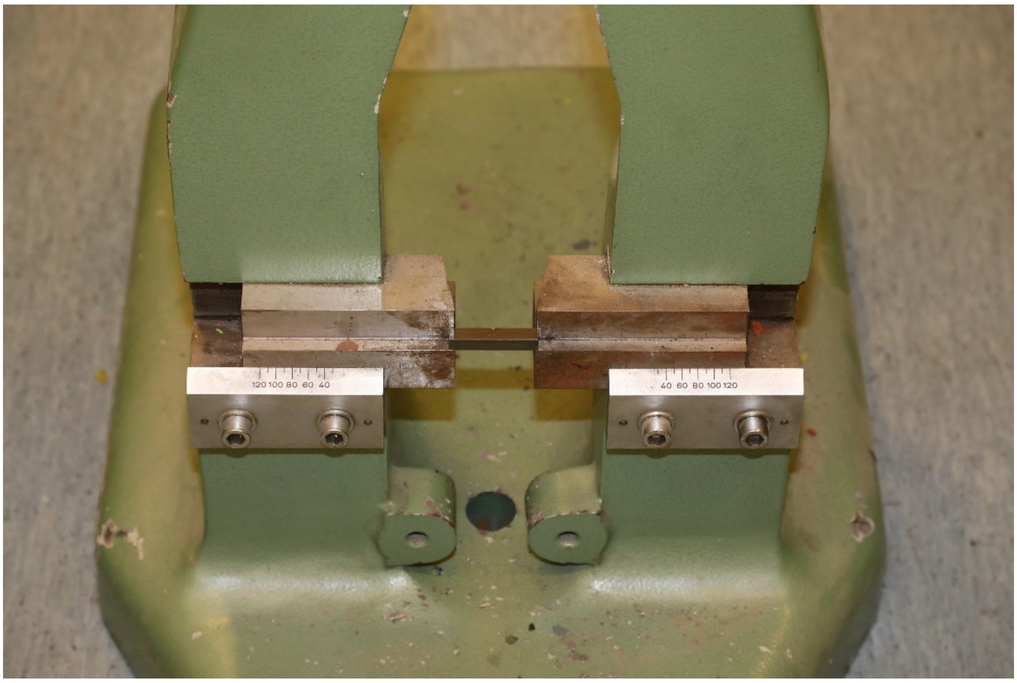

FIGURE 36. Test rod set (Brask 2022)

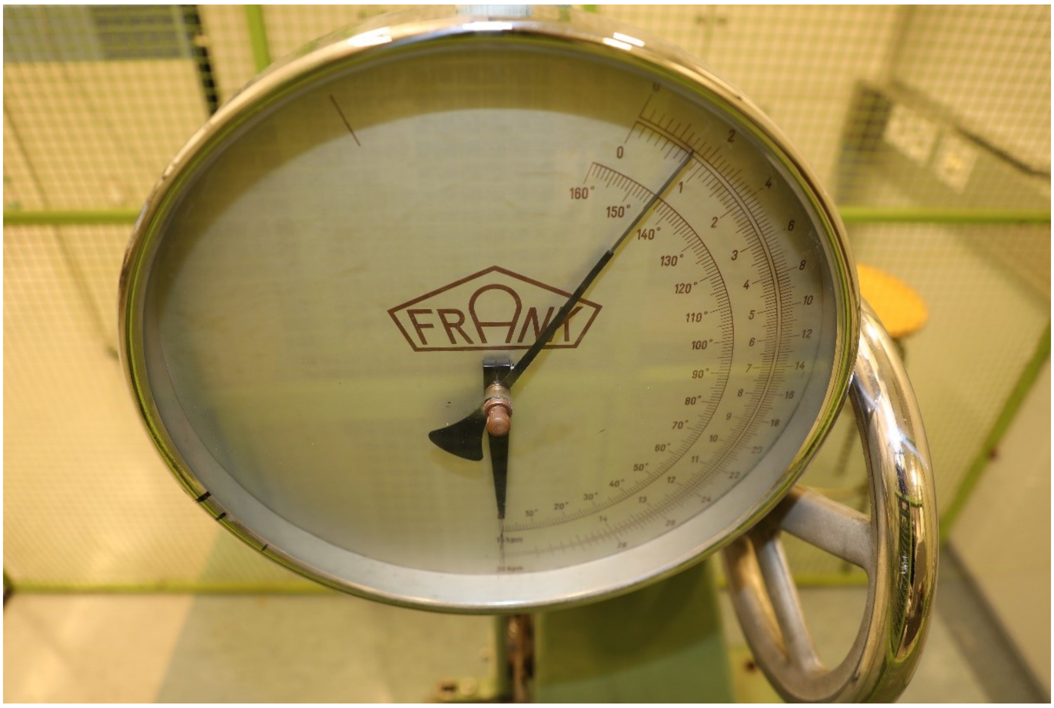

FIGURE 37. Fractional energy is read from a scale (Brask 2022)

11.3 Impact test, hardened test piece

The test set can be diversified in educational use by hardening a test rod of the same material as that which was first tested under the original material properties. This allows a comparison of how the phase change of the material affects the toughness.

In hardening, a change in the crystal structure of the metal is caused by influencing the temperatures and the quenching rate. During the hardening of carbon steels, the temperature is raised to the austenitic range and quenched rapidly as the crystal structure changes to martensite. Quenching prevents the austenite from decomposing into ferrite and perlite / bainite. Martensite is very hard but brittle, which is why the steel is released after hardening to achieve a hard but tougher structure with martensite on a ferrite & cementite base. (Hosford 2010.)

From this, an option at educational use was observed in which the hardness measurement and the impact toughness test can be performed separately for the tempered and the released sample.

11.4 Impact test, material in a cold environment

There is also an excellent opportunity to study the cold properties of materials in an impact test environment. This is done very simply by cooling the test rod in a freezer set to -22 °C. The impact test result is compared with the value measured at normal room temperature.

The test rod should remain in the freezer for at least 30 minutes and the impact test should be performed within 5 seconds of removal from the freezer (SFS-EN 10045-1: 1990). This is possible by cooling the test rod together with an open tyrox box, the lid of which is closed before being removed from the freezer. The box with the test rods is moved as quickly as possible to the impact test hammer and opened, placing the test rod as quickly but carefully as possible against the supports. The sample setting door must be closed before the instrument operator door is opened and the pendulum is triggered. This is very important for safety.

11.5 Impact test, material in a hot environment

The impact toughness of the material can also be studied at raised temperatures. This is accomplished by heating the test rod in the oven for at least 30 minutes. The test piece can also be immersed in the medium in an oven, in which case the transfer for testing can be performed while the medium keeps the temperature at the desired value. The medium can be, for example, sand.

12 CYCLES IN TEACHING USE

After the installation and trial operation of the laboratory equipment, a stage was reached where it was possible to start designing suitable tests for teaching use in the Laboratory tasks of Materials Engineering. The most consistent test cycle for teaching was thought to be a series of different tests on the same sample material. The order of the test methods was designed to support the following method. As the shape and dimensions of the impact test rod shall be exactly as specified in the standard, the impact test shall be performed first. After the impact test, the hardness of the test pieces made from the sample material and broken in that test may be measured and a sample prepared for microscopic examination. It was noted that it is advisable to perform a hardness measurement before reducing the sample with a sample cutter for hot encapsulation, as this allows the measurement traces to be left on the surface of the sample inside the capsule. The result of the hardness measurement is also needed when selecting suitable grinding and polishing methods for the sample. After hot encapsulation and grinding / polishing, the sample is etched and finally examined microscopically. The results are reported in a complementary laboratory report template.

Prior to research in the laboratory, the matter is studied in theory and the necessary standards are introduced.

12.1 Impact test

Information useful to students on the impact strength of the material can be concretized by performing a series of tests on test rods made of the same material at room temperature and at reduced temperature. Testing the material at elevated temperatures can be challenging in educational use, as providing adequate safety has its own limitations. However, the teacher can perform the safety-intensive steps on behalf of the students.

For safety reasons, it was decided that of the tests to be included in the test series, testing at room temperature and -22 °C under reduced temperature would be performed by the students and the tempering step of the hardened sample would be performed with the assistance of a teacher.

The result of the study can be deduced from the change in the toughness of the material in question after the change in temperature and after the change in hardness due to the change in phase.

Impact toughnesses of test pieces are reported. The test pieces are marked using different colored paints.

12.2 Hardness test

Hardness is measured by the Rockwell method for each of the three types of samples (see previous subheading). Changes in the hardness of the material in differently treated samples can be elucidated. In addition, the methods suitable for grinding the sample are determined e.g. according to the hardness of the sample.

Hardnesses of test pieces are reported.

12.3 Investigation of microstructure

12.3.1 Preparation

From each of the three sample types, a piece about 15 mm long is shaped with a sample cutter. The samples are thoroughly cleaned by rinsing with water and finally with alcohol.

12.3.2 Hot encapsulation

Hot encapsulation is performed using a suitable hot mounting resin for each sample separately. The capsules are marked using different colored paints (the marks on the samples are no longer visible).

The hot mounting resin used is reported.

12.3.3 Grinding and polishing

Select appropriate methods for grinding and polishing these samples. Unhardened samples may be run simultaneously using the same methods, but the hardened test piece shall be ground and polished separately.

The methods used are reported.

12.3.4 Etching

The encapsulated and polished samples are etched with a etching suitable for the material in question. This step is performed by the teacher for the safety of the students.

The etching used are reported.

12.3.5 Microscopic examination

Finally, the samples are examined under a microscope. The grain structure is refined, a screenshot is taken, and the composition of the structure and other possible observations are interpreted.

The magnification of the microscope used, the observed grain structure and other possible observations are reported.

13 EQUIPMENT PURCHASES

The need for equipment was identified with the test methods desired for teaching use. Most of the equipment was already ready on campus. Initially, it was decided to procure only the equipment that will be used to start the operation. Additional purchases can be made according to future needs. An alternative approach to microscopic examination was also investigated, more on this under the subheading AFM.

13.1 Oil-free air compressor

The manufacturer of the sample grinder requires the use of clean, compressed air as defined in the standard. Compressed air is required for sample holder functions. The air volume requirement of the device is small, only 3 liters/minute.

It was decided to purchase an oil-free compressor whose compressed air meets the requirements and falls below the permissible noise level without hearing protection. The device was put out to tender among local industrial equipment dealers. Surprisingly, the best option in terms of operation was the cheapest this time.

In addition, a water separator and an activated carbon filter were installed in the compressed air line just before the device.

13.2 Freezer

A small freezer with a capacity of 33 liters was purchased to reduce the temperature of the samples before the impact test. With a low price, the purchase could be made through a pre-competitive listed supplier.

13.3 Ventilated cabinet

The ventilated cabinet required for acid mixing and etching caused some background work prior to its acquisition. Inquiries were made which either failed to supply the device due to the strength of the acids or were unreasonably high in relation to the use. In the end, the cabinet was purchased second-hand, which had only been a model in the product development of the corresponding device. The cabinet was found to be suitable for our use and in good condition.

13.4 Atomic force microscope (AFM)

The AFM principle is based on the tip used to scan the surface of the sample. The tip controls the lever, the movements of which reflect the laser beam, allowing it to image the structure even at the atomic level (Figure 38). There are different methods for oscillating the tip, shaker piezo and photothermal actuation. (Nanosurf, 2022.)

This technique would also be the most interesting solution for studying the microstructure of materials, but too expensive due to experimentation and does not directly add value in teaching compared to an optical microscope. It was decided not to make the acquisition, at least for the time being. In the future, the next microscopic research will probably be a new, more powerful optical microscope and a related camera and software.

Correctly accurate structures could be interpreted with a Scanning electron microscope (SEM) but they are far too expensive, at least in the early stages of use. They would image the material at the nanostructure level. (Rusk 2016.)

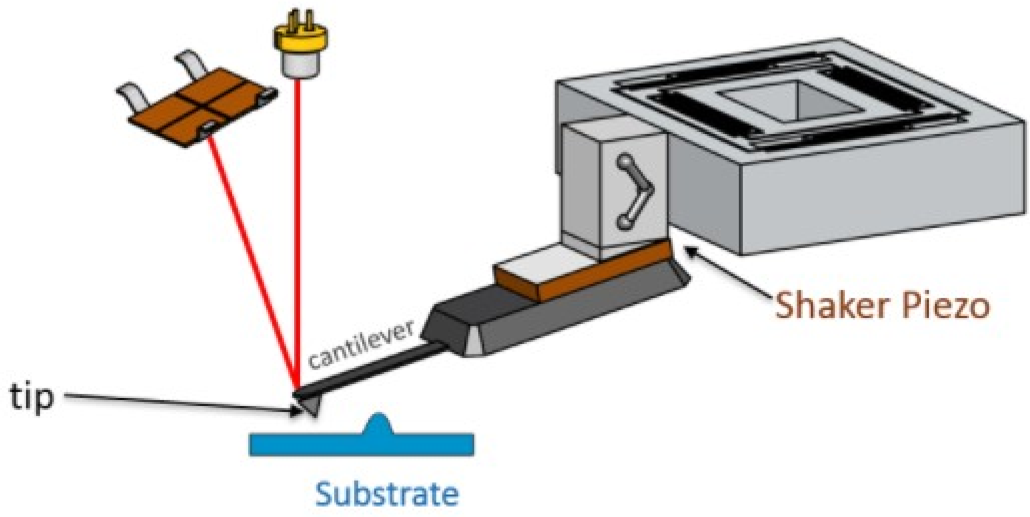

FIGURE 38. The principle of AFM (Nanosurf 2022)

14 SAFETY

Taking care of safety is important in both the work and study environment. Safety thinking starts with small enough groups of students, leaving a safe space around the devices to move and work. The small group size also helps to create a peaceful and effective learning event.

14.1 Equipment

It is very important that the protective equipment of the equipment is maintained. As shown in Figure 39, the devices have emergency stop switches. Students must be familiar with the devices before using them. In this context, the handling of potential hazards must be taken into account. The locations of first aid supplies must also be made clear.

FIGURE 39. Emergency stop switches (Brask 2022)

14.2 Chemicals

All chemicals used on campus are listed and can be found in the Safety Data Sheets. This must also be brought to the attention of students. Mr. Ari Mikkonen, responsible for chemicals at Savonia University of Applied Sciences, is responsible for matters related to chemical safety. Chemicals must be handled in the manner specified for their properties. One of the most dangerous chemicals in the materials laboratory is etching acids. These substances must not be handled by students on their own.

15 RESEARCH, DEVELOPMENT AND INNOVATIONS (RDI)

RDI activities were thought to be an option to use the material research laboratory in addition to the actual educational use. The challenge is to manage the workforce of students with a sufficiently short response time.

15.1 Opportunities for commercial research

In addition to teaching, the use of the materials laboratory in RDI activities was enquired. In the long run, it could be possible, for example, to carry out damage analyzes commercially. Such activities are entirely possible over time, but require more experience in managing laboratory methods and, above all, analyzing the results before they take place. It is also important to be sure that the laboratory work is successful with enough frequency from the students as the students constantly change. This is facilitated by the clarity of the instructions, and once again the experience that is best gained through educational use.

However, there has been preliminary discussion with a major player in the energy industry about impact resistance tests for cold-resistant steel grades at reduced temperatures. This test suite is a good example of how laboratory-related RDI activities could be taken up.

16 CONCLUDING REMARKS

There were very clear starting points in this thesis: In addition to theory, the teaching of materials technology needs laboratory work to concretize the learned information in practice and to enliven the course. On the other hand, the practical precondition for the organization of the laboratory was in the form of a suitable environment ready and equipment previously acquired. The simultaneous execution of several things brought an extra kick to the work; the construction and commissioning of a laboratory supporting the teaching of the author of the thesis, as well as the commissioning of stored equipment should be mentioned as the most significant of these.

The teaching of materials engineering was also supported in terms of the study of the theory on which materials research is based. The construction of the laboratory also strengthened our own practical knowledge of the equipment and their use on behalf of the crystal structures of the materials. Piloting for educational use is under way and experience to date has shown that the achievement of the goals has been expected, or even exceeded expectations. Students understand how laboratory work can be used to test different properties of different materials under different conditions and then, by comparing the results, conclusions can be drawn about the behavior of the materials according to, for example, the purpose and environment of use.

The Materials Research Laboratory is a welcome addition to teaching and is constantly evolving as experience grows. The use of RDI is also constantly mirrored in relation to the needs of energy companies. Students have the opportunity to gain contact with companies by working as part of a laboratory user group in connection with various studies.

The implementation of the thesis with its laboratory plans and its introduction, also with the creation of teaching-related deliverables, was an extensive experience for the author and brought more opportunities to support the diversity of learning.

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