AUTOMATION OF METALLOGRAPHIC SAMPLE CLEANING PROCESS

[1]Jan ČERMÁK, [1]Ondřej AMBROŽ, [1]Patrik JOZEFOVIČ, [1]Šárka MIKMEKOVÁ

[1]Institute of Scientific Instruments of the CAS, Brno, Czech Republic, EU, cermak@isibrno.cz

https://doi.org/10.37904/metal.2022.4478

Abstract

Specimen cleaning and drying are critical processes following any metallographic preparation steps. The paper focuses on automation by reason of absence of the process repeatability during manual sample handling. An etchant or electrolyte results in inhomogeneous surface quality because the solution runs off the specimen surface during its removal from the beaker. High-quality specimen cleaning is absolutely crucial for the acquisition of the specimen suitable for characterization by a scanning electron microscope operated at very low landing energies of the primary electrons (SLEEM). The SLEEM technique is a powerful tool for the characterization of advanced steels, as described by many scientific papers. The SLEEM requires the specimen absolutely free of water and any organic residues on the surface. This work presents a novel unique apparatus enabling automatic specimen cleaning and drying after the etching or electropolishing processes. Automation reduces the influence of dependent variables that would be introduced into the process by the metallographer. These variables include cleaning time, kinematics, and motion dynamics, but the process can also be affected by variables that are not obvious. Performed experiments clearly demonstrate our in-house designed apparatus as a useful tool improving efficiency and consistency of the sample cleaning process. The high quality of the specimen surface is verified using a light optical microscope, an electron scanning microscope, and above mentioned SLEEM technique.

Keywords: Metallography, sample cleaning, process automation, repeatability

1. INTRODUCTION

Process of cleaning and drying is probably the most underrated part of specimen preparation. Cleaning process ensures removing of residues originated from prior preparation out of surface. Cleaning can be divided into two categories - 1. intermediate cleaning (cleaning between mechanical grinding and polishing) and 2. final cleaning and drying. Final cleaning is crucial step prior to specimen examination [1]. One of the most common requirements of materials analysis is to reveal the microstructure. The suitable method for microstructure revealing is chemical etching. But the etchant easily adheres or dries on the sample surface. The presence of these artefacts makes further analysis of the samples using a microscope difficult. Especially in the case of advanced electron microscopy, imaging methods using low energy. The SLEEM method is characterized by a small interaction volume due to the low impact energy of the electron. For this reason, SLEEM is very sensitive to the cleanliness of the resulting surface. It is true that prior to the actual observation with the UHV-SLEEM method, ion cleaning is used to remove the oxide layer from the sample surface [2]. But ion cleaning is not able to remove large artefacts therefore the surface must be free of residues after etching or electropolishing itself. As a standard, samples are etched either manually in a beaker with an etchant or using commercial electrolytic etching equipment. However, these devices do not address cleaning of the sample after etching. Thus, in either case, the metallographer must clean and dry the samples by hand. Etchants (usually alcohol-based) dry very quickly at room temperature and eliminate contaminants from the surface. For this reason, it is necessary that the surface removed from the etchant be immersed very quickly in the cleaning liquid. During the removal of the sample from the etchant, some of the reagent is also transferred to the cleaning liquid, which is contaminated with this reagent. Therefore, it is necessary that the cleaning process is multi-stage.

Most cleaning liquids are also alcohol-based, so they dry very quickly and can create inhomogeneous hydrocarbon contamination on the sample surface depending on the direction of flow from the surface. To minimize this form of contamination, a stream of hot air is usually used to blow some of the liquid off the sample surface and dry the rest very quickly [3].

This work focuses on the use of automation tools for the final cleaning and drying of metallographic steel samples for observation in the electron microscope. Manufacturers of metallographic sample preparation equipment offer models with so-called automation, which, however, automates only one sub-process of the preparation. For example, Struers' Lavamin automates sample cleaning but does not address inter-process automation. For example, cleaning with the Lavamin after preparation in LectroPol Struers for "automated" electropolishing and etching is realized [4, 5]. Currently, there are not many truly automated sample preparation stations on the market. Perhaps the only company that offers comprehensive automation of metallographic sample preparation is UES with its Robo-met station [6]. For our needs, to eliminate as many stochastic variables entering the process as possible, we developed a device using a programmable five-axis robotic arm. In this case, we use a robotic arm with tweezers to hold the sample for etching and subsequent cleaning and drying. The sample was etched first because etching highlights the surface topography and therefore the reagent sticks to it more. For a better cleaning effect, the beakers with cleaning fluids were placed in an ultrasonic bath.

Since this is the setup, we commonly use for sample preparation, the use of etchant as a contrast agent is most telling. The objective of this experiment was to determine the smallest number of beakers of cleaning fluids that must be used to keep the sample free of etchant contamination.

This experiment is part of our team's larger work on automating some metallographic processes for steel sample preparation. The motivation for this approach is to increase the repeatability of sample surface preparation in order to obtain a large set of consistent microstructure data for use with deep neural networks.

2. METHODS

2.1. Specimen fabrication methodology

The specimens were obtained from hot rolled steel plate Usibor 1500-AS (22MnB5) with a thickness of 1.5 mm. The sheet is treated on both sides with an aluminium-silicone coating called "Alusi" (Al 90%, Si 10%), which was removed by grinding in further preparation [7, 8]. Usibor 1500 was used because of its good reactivity with the etchant and because it fits into our area of interest of AHSS steels. The samples were cut to a size of 7 × 5 mm. The specimens were ground using 220-2000 grit diamond disk, then with 4000 grit SiC foil. After that, specimens were polished with diamond paste with 3, 1, 0.25 μm grain sizes.

Table 1 Cleaning times in each beaker

Number of beakers	Name of sample	Time in beaker 1 (s)	Time in beaker 2 (s)	Time in beaker 3 (s)	Time in beaker 4 (s)	Time in beaker 5 (s)	Time in beaker 6 (s)	Time in beaker 7 (s)
1	U09	28	-	-	-	-	-	-
2	U10	1	27	-	-	-	-	-
3	U16	1	2	25	-	-	-	-
4	U18	1	2	3	22	-	-	-
5	U24	1	2	3	4	18	-	-
6	U21	1	2	3	4	5	13	-
7	U22	1	2	3	4	5	6	7

Samples were etched for 2.75 s in Kourbatoff No. 4 etchant, which is a combination of Nital (4 vol. % HNO_3) and Picral (4 vol. % HNO_3) being used to differentiate between austenite, martensite and tempered martensite [9]. The beaker with the etchant was placed in an ultrasonic bath (Elmasonic P 30 H) at a frequency of 80 kHz. Because during the preliminary experiments it was shown that etching with ultrasound has a better repeatability. The same ultrasonic bath was used to place beakers of cleaning fluid (Isopropyl alcohol). Samples were etched in the first beaker and then cleaned sequentially in one to seven beakers (**Table 1**). After each sample was prepared, the cleaning fluids were changed. Subsequently, after cleaning in the last beaker, the sample was dried with a stream of hot air from a hot air gun. A programmable five-axis robotic arm (Hiwonder xArm) was used for sample handling. A schematic of the apparatus is shown in **Figure 1**.

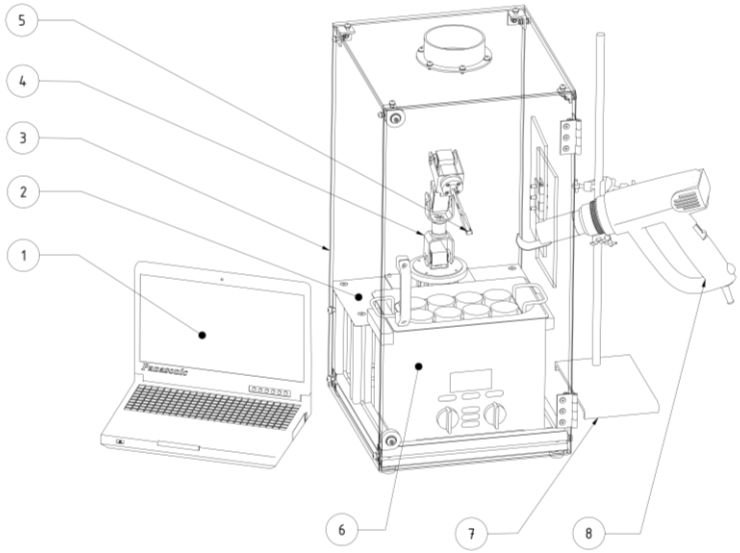

Figure 1 Schematic of the apparatus: 1) Laptop for robot control, 2) robot stand, 3) protective box, 4) 5-axis robotic arm, 5) metallographic specimen, 6) ultrasonic bath with beakers, 7) laboratory stand 8) hot air gun

2.2. Image acquisition and evaluation methodology

Microstructure images were obtained using three devices:

- Zeiss Axio - Light Optical Microscope (LOM)
- FEI Magellan 400L - Scanning Electron Microscope (SEM)
- Thermofisher Scientific Helios 4G - Dualbeam FIB/SEM with SLEEM

First, the samples were observed in a light microscope immediately after preparation at 500× magnification and resolutions of 2464 × 2056 px (representing 328.5 × 274.1 µm; 7.5 pixels/µm), then at 1,000× magnification and the same resolution (representing 164.3 × 137 µm; 15 pixels/µm). Images were taken at both the outer edge and the center of the sample at both magnifications. Namely, we know from experience that the perimeter of the sample is harder to clean than the center of the sample.

The samples were then observed in an FEI Magellan 400L scanning electron microscope at 1500× magnification and a resolution of 1536 × 1024 px (representing 99.1 × 66 µm; 15.5 pixels/µm), and 5000× with the same resolution (representing 29.5 × 19.7 µm; 52 pixels/µm). An ETD detector in secondary electron (SE) mode with an accelerating voltage of 5 kV, a bias of 0 V and a working distance of 3 mm was used for imaging. As with the light microscope, images were taken at the outer edge and in the center of the sample at both magnifications.

Finally, the suitability of the preparation in the FIB/SEM electron microscope Helios 4G from ThermoFisher Scientific in the mode with very low landing energy of primary electrons (SLEEM) was verified. The surface

of sample U16 (three beaker cleaning) was observed at accelerating voltages of 100 V to 25 kV using CBS detector at magnifications of 10,000× and a resolution of 1536 × 1023 (representing 41.3 × 27.5 µm; 37.2 pixels/µm). Due to the time-consuming nature of the observations, images were taken only in the center of one sample.

3. RESULTS AND DISCUSSION

3.1. Results

Although an etchant with good reactivity and a non-corrosive sample material was chosen, only seven out of thirteen samples were etched on the first attempt. We cannot explain this phenomenon, but it will be investigated further.

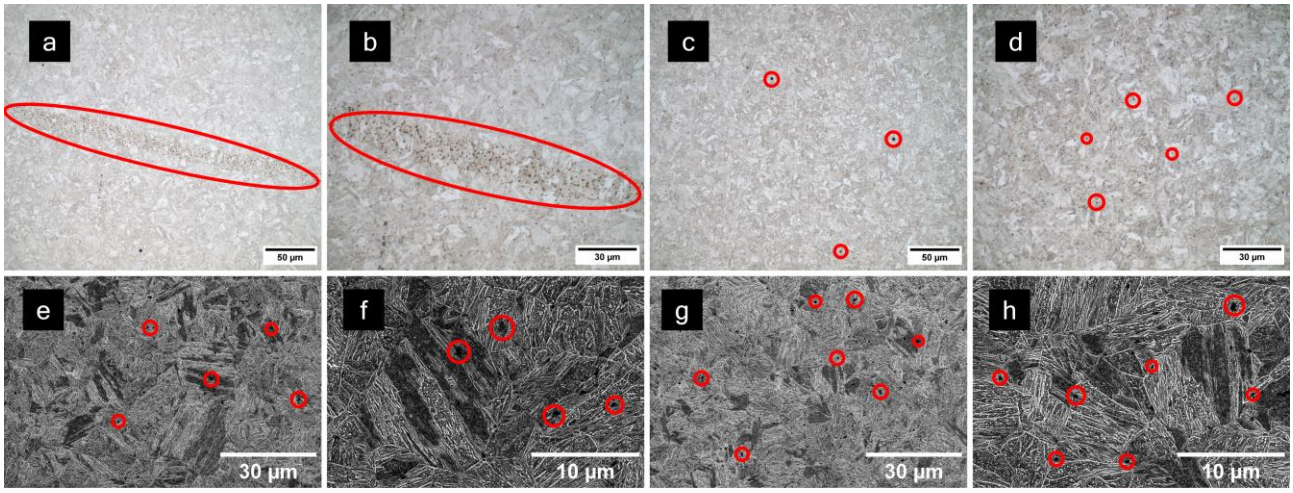

Figure 2 Micrographs of sample U09 with examples of contamination: a) LOM outer perimeter, b) LOM outer perimeter, c) LOM center, d) LOM center, e) SEM outer perimeter, f) SEM outer perimeter, g) SEM center, h) SEM center

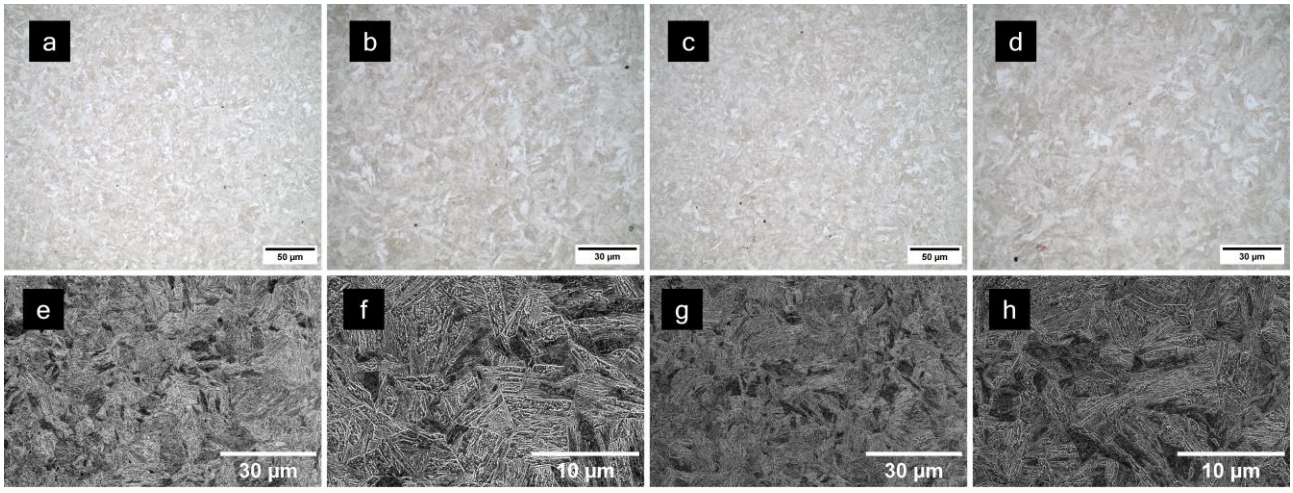

Figure 3 Micrographs of sample U12 (a-h same as **Figure 2**)

On sample U09, which was cleaned in only one beaker, contamination is already visible at the lowest magnification. In **Figure 2**, contamination can be observed at all magnifications in both LOM and SEM. Random contaminations are marked in red circles for illustration. After cleaning in two beakers, a significant reduction of contaminations on the sample surface was observed, see **Figure 3**. The surface of the sample

cleaned in three beakers was already of sufficient quality for observation by SLEEM. **Figure 4** shows a series of the SLEEM micrographs of the same point of view obtained at various landing energies of the primary beam. As visible, the micrographs acquired at lower landing energies (1 keV and down) contain more information about the surface state. Inelastic mean free path diminishes with decreasing landing energy of the primary electrons and the signal electrons originate from shallow depth [10]. Contamination seen in the middle of the images is probably abrasive particles from mechanical preparation. This particle was chosen for ease of navigation on the sample and shows the differences between the different landing energies.

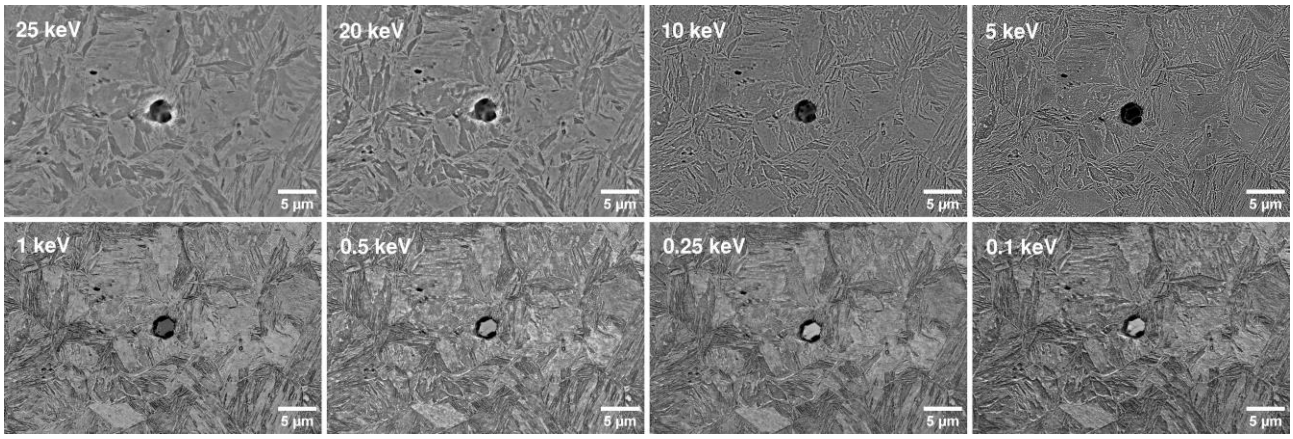

Figure 4 Same point of view on the USIBOR steel surface visualized at various landing energies of the primary beam.

Not surprisingly, the sample cleaned in the maximum number of beakers (seven) is cleaned very satisfactorily, the micrographs of sample U22 can be seen in **Figure 5**.

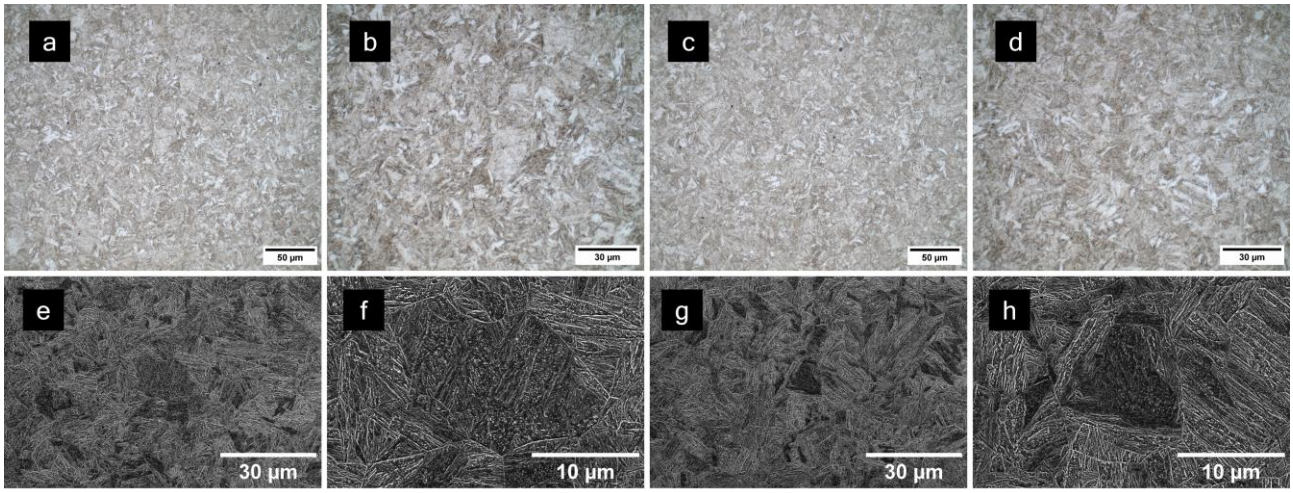

Figure 5 Micrographs of sample U22 (a-h same as **Figure 2**)

3.2. Discussion

Unfortunately, outside of any trend, the surface of sample U24 cleaned in five beakers shows a high degree of contamination (similar to sample U09). This pollution is not due to the number of cleaning beakers, but to a variable entering the process that we have not been able to eliminate. In future work, we will deal with the detection and elimination of these hidden variables. For an exact investigation of these processes, it would be advisable to work in clean rooms without the risk of contamination from the air. Furthermore, it would be appropriate to monitor hydrocarbon contamination using EDS maps on a carbon-free standard. In the future,

the low-cost robotic arm will be replaced with a more accurate robot. Furthermore, the development of advanced sample drying methods that do not risk further contamination of samples is planned.

4. CONCLUSION

We designed our own apparatus for automatic etching, cleaning and drying of metallographic samples. Using LOM and SEM imaging methods, we verified the suitability of this approach. The dependence of surface quality on the number of beakers of cleaning liquid was studied. Although no obvious trend emerged from the results, the microstructure of the steel was observable even with the SLEEM method, which is super-sensitive to surface quality. And this is an important milestone for our future work.

ACKNOWLEDGEMENTS

The research was financially supported by the Lumina Quaeruntur fellowship established by the Czech Academy of Sciences as an instrument to support scientific excellence in the CAS, and by Technology Agency of the Czech Republic - National Center of Competence No. TN01000008.

REFERENCES

[1] Metallographic Cleaning Information. 2022. *Metallographic.com* [online]. USA: PACE TECHNOLOGIES. Available from: https://www.metallographic.com/Metallographic-Technical/Metallography-Technical-Cleaning.htm.

[2] MIKMEKOVA, S., HOVORKA, M., MULLEROVA, I., MAN, O., PANTELEJEV, L., FRANK, L. Grain Contrast Imaging in UHV SLEEM. *Materials Transactions.* 2010, vol. 51, no. 2, p. 292-296. Available from: https://doi.org/10.2320/matertrans.MC200919.

[3] ECHLIN, Patrick. *Handbook of Sample Preparation for Scanning Electron Microscopy and X-Ray Microanalysis.* Boston: Springer, 2009, s. 135.

[4] Lavamin. 2022. *Struers* [online]. United States: Struers. Available from: https://www.struers.com/en/Products/Automatic-cleaning/Automatic-Cleaning-equipment/Lavamin.

[5] LectroPol. 2022. *Struers* [online]. United States: Struers. Available from: https://www.struers.com/en/Products/Electrolytic-Preparation/Electrolytic-equipment/LectroPol.

[6] Robo-Met Materials Characterization. 2021. *UES: Excellence in Science & Technology* [online]. United States: UES. Available from: https://www.ues.com/robomet.

[7] ArcelorMittal Usibor® 1500 Ultra high strength steel, Hot Rolled. c1996-2022. *MatWeb* [online]. MatWeb. Available from: https://www.matweb.com/search/datasheettext.aspx?matguid=407ac4f81e734649843a8012927bcc09.

[8] ÇAVUŞOĞLU, Onur, ÇAVUŞOĞLU, Oktay, YILMAZOĞLU, Ahmet Gürkan, ÜZEL, Uğur, AYDIN, Hakan, GÜRAL, Ahmet. Microstructural features and mechanical properties of 22MnB5 hot stamping steel in different heat treatment conditions. *Elsevier Journal of Materials Research and Technology* [online]. 2020, vol. 9, no. 5. Available from: https://www.sciencedirect.com/science/article/pii/S2238785420315416.

[9] VOORT, George F Vander. *Metallography: principles and practice*. 4. Materials Park, Ohio: ASM International, 2007.

[10] MIKMEKOVÁ, Šárka, JÁNSKÝ, Pavel, KOLAŘÍK, Vladimír and MÜLLEROVÁ, Ilona. Surface imaging with UHV SLEEM and SEM LEEM. *Microscopy and Microanalysis* [online]. 2019, vol. **25,** (S2), pp. 444-445. Available from: https://www.cambridge.org/core/journals/microscopy-and-microanalysis/article/surface-imaging-with-uhv-sleem-and-sem-leem/261F5B8B49B98F7B3F8AA0163DE72BA1.