METALOGRAPHY IN BOSNIA AND HERZEGOVINA BEFORE 30 YEARS AND TODAY

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ABSTRACT
In modern times, the science of materials cannot be imagined without metallography. The history of metallographic testing in Bosnia and Herzegovina is primarily related to Zenica. Today, most of the research related to the metal industry in BiH is conducted in laboratories at the Kemal Kapetanović Institute in Zenica. The Institute's Metallographic Laboratory has a history to be proud of and a future to nurture. This paper will review the capabilities of the Institute's Metallographic Laboratory 30 years ago and now.

1. INTRODUCTION
Metallography, as part of material science, deals with the examination, description and evaluation of microstructures of metals and alloys. The development of metallography in the world begins more than 150 years ago [1]. The beginnings of applying metallography in Bosnia and Herzegovina can be found at the Institute in Zenica, back in 1961.

2. METALLOGRAPHY
The beginning of studying the microstructure of metals, according to literature, goes back to the second half of the nineteenth century. Namely, in England H.C. In 1864, Sorby made the first macrograph of the microstructure of steel. The copy of the macrograph given in Figure 1, taken at magnification 9 times, clearly shows the grain boundaries [2].

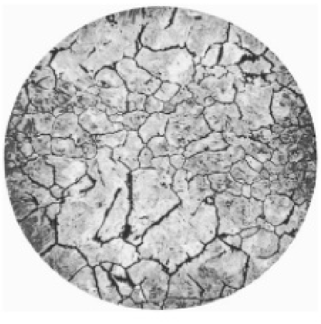


Figure 1. The first macrograph of a microstructure of steel, from 1864, etched in a very dilute solution of nitric acid, x9 [1,2,3]

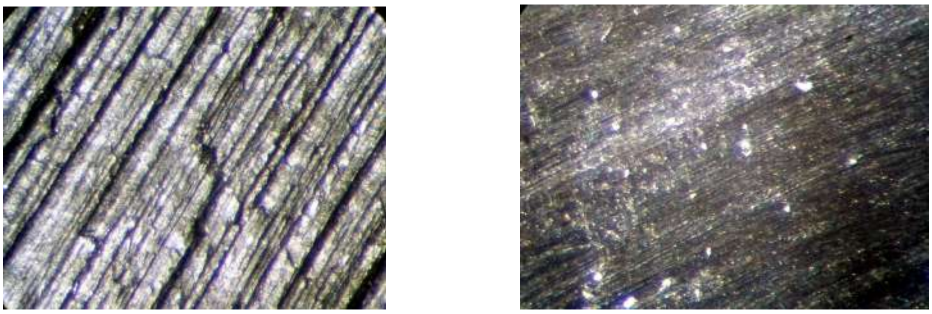
Contributions to the beginnings of metallography development were: A. Martens in Germany, F. Osmond in France, W.C.Roberts-Austin in England, H.N. Howe in America.

3. PREPARATION OF METALLOGRAPHIC SAMPLES

The metallographic laboratory basically consists of sample preparation room, etching room and test room. Due to possible contamination of the dust during cutting, the sample preparation room was separated by a wall from the test part of the laboratory.
The preparation of metallographic specimens consists of:

- cutting,
- pressing in,
- grinding,
- polishing and
- etching.

The basic differences in the steps of preparing metallographic samples 30 years ago and today are given in Table 1.
The etching of the prepared test surface is still done 30 years ago in a digester, a ventilated area using reagents, which are selected depending on the type of material being tested. The appearance of the cutting surface taken with a digital camera after the coarse cutting and modern cutt of wheels is shown in Figure 2.



a) Cut with a rough cutting blade *b) Cut with a fine cutting blade*
Figure 2. Layout of the cutting surface, magnification 50x

Table 1. Differences in sample preparation

Steps of sample preparation	30 years ago	Today	Differences and advantages today compared to 30 years ago
Cutting	Cutting with cutt of wheels that gave a rough surface to the sample.	Cutting of the specimens is performed on a specimen cutting machine using cutt of wheels that produce fine smooth surfaces. In this way, the preparation time of the sample is shortened by one to two steps of grinding the samples.	Fine cutting surface Applying an appropriate lubricant to cool the cutting surface does not cause the sample to heat up and have unintended consequences on the original microstructure.

Steps of sample preparation	30 years ago	Today	Differences and advantages today compared to 30 years ago
Pressing in	Pressing into warm plastic mass	Pressing into cold or warm plastic mass	Nowadays can be press larger samples than the size of the mold for warm press
Grinding	Grinding test surface on a wet grinder was started from 120SiC and 180 SiC paper to eliminate the cutting marks created by using a rough cutting board. Further grinding steps of test surface were either manual or semi-automatic on SiC grinding paper gradation: 240, 400, 600, 1000.	Grinding of test surface is done automatically on self-adhesive grinding paper SiC gradations: 240, 400, 600 and 1000.	Due to the finer cutting surface, the number of grinding steps is reduced from 6 to 4. Multiple specimens can be automatically prepare at the same time, thus reducing the preparation time and thus the consumption of electricity required for preparation.
Polishing[1)]	Diamond paste 9μ, DP lubricant applied on felt polishing cloth Diamond paste 3μ, DP lubricant applied on soft polishing cloth Alumina 0,05μ applied soft polishing cloth	Diamond suspension 9μ, DP lubricant applied on felt polishing cloth Diamond suspension 3μ, DP lubricant applied on soft polishing cloth	The use of diamond suspensions reduced one preparation step. The use of lubricant saves on diamond suspension significantly. Multiple samples can be polished automatically at the same time. Preparation time is shortened and thus the electricity required for preparation is consumed.

1) This step for sample preparation applies to steel

4. MICROSCOPE

The basic tool without which one can imagine a metallographic laboratory is a light optical microscope. The prepared metallographic specimen is impermeable to light and therefore the basic part of the metallurgical microscope is a source of light which is reflected from the surface of the metal specimen.

4.1. Briefly about the microscope

The word microscope comes from the Greek words "micros" meaning small and "scopin" meaning to see. The name of the microscope was introduced by the Roman scientist Giovani Faber back in 1625. In 1665, English microscopist Robert Hook used the microscope given in Figure 3 [2].

Figure 3. Sketch of the microscope used by Robert Hook, in 1665. [2]

4.2. Metallurgical microscope

There are two types of metallurgical microscopes: vertical and inverted. The difference between the two types lies in the location of the sample placement. For the vertical type of microscope, the sample is placed below the lens, while for the inverted, the sample is placed above the lens. The light source of metallurgical microscopes should be strong, stable and controlled as an intense single beam of light [2].

The appearance of the light optical microscope used 30 years ago is given in Figure 4. The appearance of the modern light optical microscope used today in the Metallographic Laboratory is given in Figure 5.

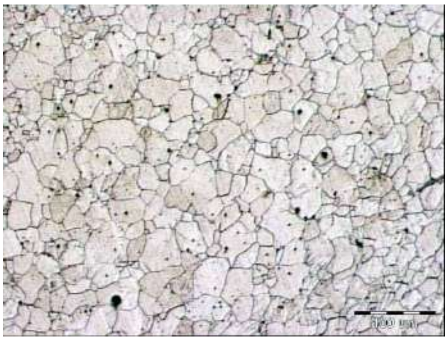
Figure 4. Layout of a light optical microscope used 30 years ago

Figure 5. Layout of a modern light optical microscope

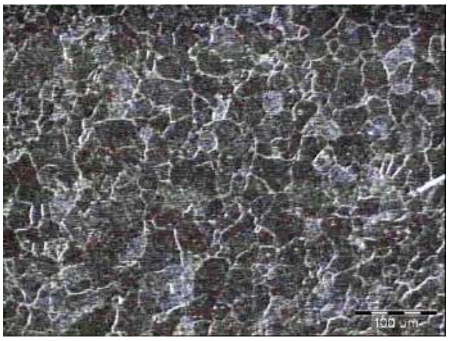
The basic differences in metallurgical microscopes 30 years ago and today in the Metallographic laboratory at the Institute are:

- Light source: 30 years ago used tungsten filament lamp, and in nowadays tungsten halogen bulb, with advantages - the lamp life is longer and the light quality is better, without tungsten deposits on the lamp wall
- Ilumination – nowadays light microscopes have possibility for examination in:
    - bright field
    - dark field
    - polarized light
    - nomarski prism

Illumination of the light and dark fields of the steel pattern is given on Figure 6.



low carbon steel grain size (NITAL)

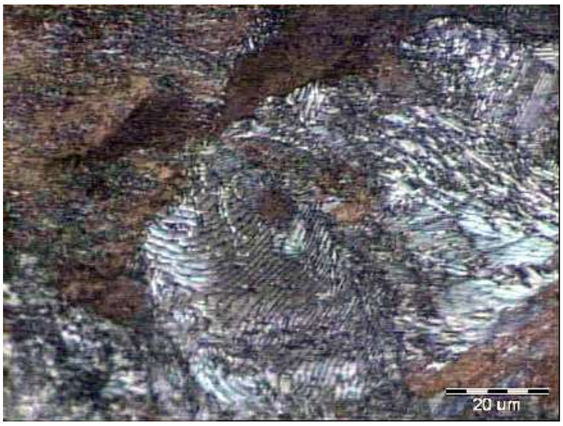


low carbon steel grain size (NITAL)

Figure 6. Micrograph (a) was taken in bright-field illumination, and micrograph (b) was taken with dark-field illumination.

Figure 7. shows flat etched surface of steel sample under normal bright field a) and under differential interference contrast b) with crossed polarizers.

a) without DIC



b) with DIC

Figure 7. Microstructure of steel using differential interference contrast, Picral, magnification 500x

5. CALIBRATE AN EYEPIECE RETICLE SCALE

Metallographic laboratory at Institute "Kemal Kapetanović" in Zenica has accreditation from National accreditation body BATA almost 22 years. In order to prove the competence of the staff employed in the laboratory for the application of modern testing methods, regular supervision checks are carried out by expert teams. To ensure accuracy, the stage micrometer must be traceable to the International System of Units through calibration provided by a competent accredited laboratory [4].

Measurement of a microstructural feature is done using an eyepiece reticle with a graduated linear scale and using calibrated input in software.. Before making such a measurement, the scale must be accurately calibrated. This is done by using a calibrated stage micrometer in combination with the reticle eyepiece.

Line up the two scales until they are parallel, as shown in Fig. 8, and align the "0" of the two micrometer scales, as shown. In this stage micrometer, one small division equals 0.01 mm (100 divisions 1 mm).

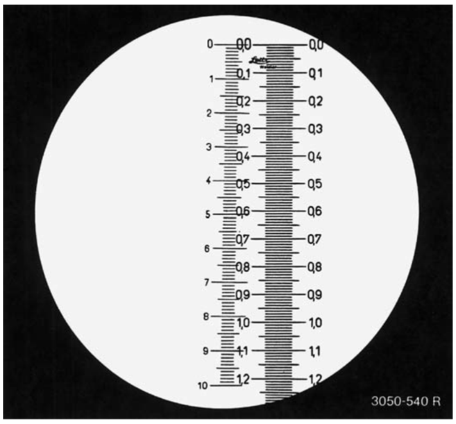


Figure 8. Micrograph of stage micrometer and eyepiece micrometer

As shown in Fig.8, count the number of divisions on the stage micrometer scale that match the full 100 divisions on the eyepiece reticle scale. In this example, 100 divisions on the eyepiece scale equal 122 divisions on the stage micrometer.

Therefore, 122 divisions equal a length of 1.22 mm. From the following equation, calculate the length of each division on the eyepiece scale:

$$Eyepiece\ scale\ division = \frac{Length\ of\ stage\ micrometer\ division}{Number\ of\ divisions\ on\ eyepiece\ reticle\ scale}$$

Thus, each eyepiece scale division 1.22 mm/100 divisions = 0.0122 mm per division, or 12.2 µm per division (1 mm= 1000 µm). This means that everything that is magnified by this objective-eyepiece combination on this microscope is 1.22 greater than actual.

Thus, a 5x objective with a 10 x eyepiece would have a magnification of 5x10x1.22= 61x.

This step should be repeat for each objective at microscope.

Because of the it is need to prepare a table of eyepiece scale (or eyepiece micrometer) calibrations and magnification corrections for each objective-eyepiece combination for each microscope in the metallographic laboratory [2].

6. SCANNING ELECTRON MICROSCOPE

Few years ago, Metallographic laboratory start using scanning electron microscope PHILIPS XL30, figure 9. Possibilities of this device are wide.

Figure 9. Scanning electron microscope in Metallographic laboratory

Getting micrographs of interested point at very high magnification (more than 10 000 x) and its EDX analysis is very important for researchers work. Figure 10 shows one of test results of precipitation hardened stainless steel 17-7PH getting at this device.

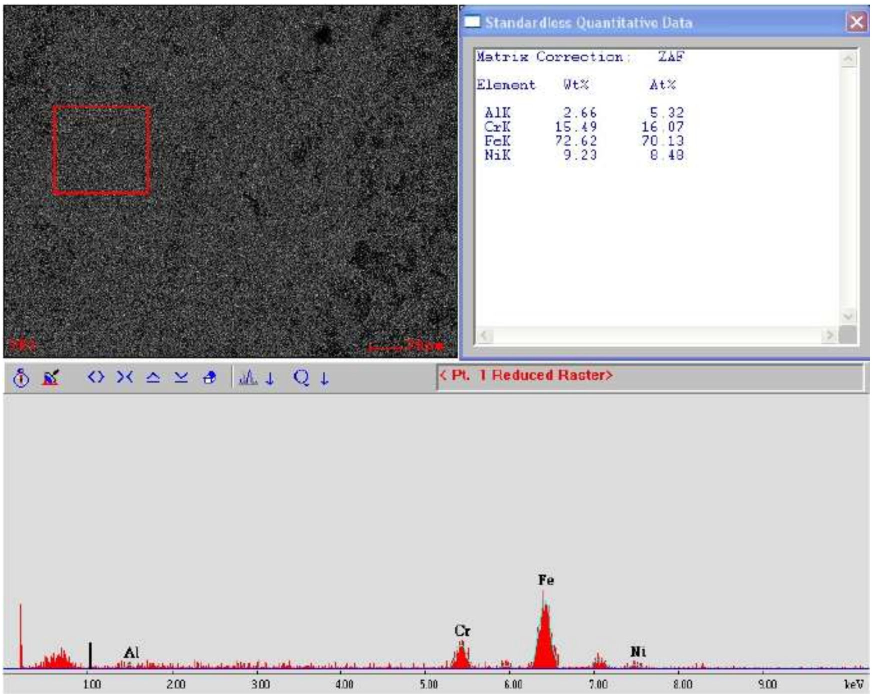


Figure 10. Microstructure and EDX analysis [5]

7. CONCLUSION

Metallography is part of Materials science and it is irreplaceable instrument for research of metal and their alloys. Metallography plays very important role in metallic material research. Examination of metals and alloys purity, grain size, microstructure after different heat treatment and their influence on mechanical properties are very important for everyone who works with this material. Metallography are eyes of science of Materials science.

8. REFERENCE

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