



# A MACRO- AND MICROSCOPIC INSPECTION OF THE SAFETY CRITICAL COMPONENTS

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## Abstract

In many cases related to critical infrastructures, we find equipment that is extremely important from a safety point of view and key safety-critical components in them. Such parts are turbine blades, engine elements of jet aircraft, as well as high-pressure steam lines of power plants and pressure equipment for liquid gases. In the case of safety critical components, non-destructive and the destructive tests are indispensable. The damage to these components can cause catastrophic failures that need to be avoided. During the inspection of the components, microscopy tests give information about macrostructure and microstructure. Carrying out the tests consists of several steps, during which it is necessary to follow the relevant standards. During the examination of macrostructure, conditions that may lead to premature failure and breakage of the component can be detected. Getting to know the microstructure can provide information about manufacturing, welding, or microstructural defects, which can be a key issue from the point of view of safe operation.

**Keywords:** *microscope, destructive testing, welding, microstructure.*

## 1. Introduction

Numerous safety-critical components are known, and their failure can lead to disasters. Currently, there are no specific regulations for the inspection of these components. In many cases, components containing welded joints, pipelines, or turbine blades are considered safety critical. There are regulations for inspecting welded joints, such as examinations targeting macrostructure and microstructure analysis. In this paper, we aim to analyse the sample preparation for inspections used in welded structures and emphasize the importance of training the personnel responsible for performing this task.

## 2. Sample Preparation

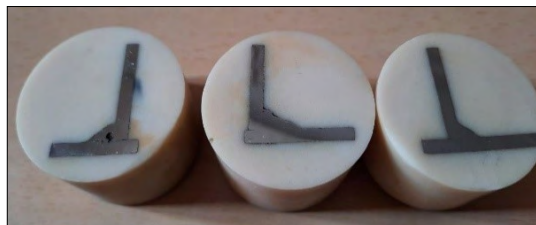
During the preparation the size and shape of the sample are crucial. The object to be examined may be too small to grind the surface (e.g., the

cross-section of a needle or a wire) or too large (e.g., a welded piece). If the object to be examined is small in size, an embedded sample of the appropriate size can be prepared, allowing us to further prepare the surface. However, if it is too large, it is necessary to create a representative sample specific to the material under investigation [1, 2, 3]. During the cutting process, it is important to ensure that the material structure of the sample is not altered. Therefore, cutting should not cause the specimen to heat up or undergo plastic deformation, as both can lead to changes in the material structure and yield false examination results. When examining welded joints, it is advisable to follow the recommendations of the standard regarding the location of sample extraction [4, 5]. Cutting can be performed using water jet cutting or machining. In practice, specialized laboratory cutting equipment can also be used, typically employing a diamond abrasive cutoff wheel, adjust-

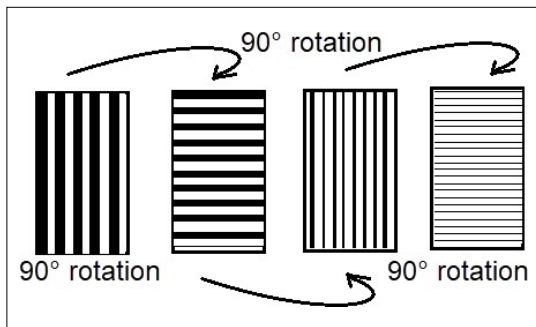
able speed, and water cooling to perform the sample cutting. For thin sheets and foils, cutting with scissors is also an option. The cut surface will inevitably undergo deformation, so it is necessary to further work on the cut surface by grinding whenever possible [1, 2, 3].

### 3. Specimen mounting

Mounting refers to encapsulating the machined sample or small-sized specimen in a polymer mounting material. Depending on the type of resin used and the nature of the sample, cold or hot mounting methods can be applied. The primary objective here is to ensure that neither the structure nor the chemical composition of the sample is altered during the mounting process [1, 2, 3]. In cold mounting, a two-component resin is typically used. During the curing of the resin, minimal reaction heat is generated, but this does not cause structural changes in the case of metals. To prevent any accidents, it is necessary to follow the recommended proportions and mixing sequence provided by the manufacturer of the mounting material in all cases. **Figure 1** shows the samples prepared using the cold mounting method.



**Figure 1.** Samples using cold mounting method.



**Figure 2.** Steps of grindings.

Hot mounting is performed by heating and polymerizing the powder mounting material (such as Bakelite, epoxy, acrylic, etc.). The temperature for hot mounting can reach up to 200°C, depending on the specific mounting material. However, it is crucial to consider that for certain metals with low melting temperatures, this temperature range can cause changes in the microstructure, such as recrystallization.

### 4. Grinding

The surface of the sample is ground using progressively finer abrasive wheels. The abrasive material used can be silicon carbide-based or diamond grinding discs. Abrasive materials are commercially available in various grit sizes ranging from P60 to P4000. Abrasive materials with grit sizes of P2000 to P4000 are considered polishing agents as well. Whether manual or automated grinding is employed, during each grinding step, the sample is rotated 90 degrees to remove the previous grinding scratches. Thus, grinding eliminates the previous grinding scratches with perpendicular and finer scratches (see **Figure 2**). Grinding is performed with a continuous water supply, where the water helps remove loose particles, debris, and heat generated by friction.

### 5. Surface polishing

The next step is to polish the surface prepared to the desired level of smoothness. Polishing can be performed through mechanical, chemical, or electrolytic methods. Mechanical polishing is carried out using a polishing disc or a polishing cloth. The material of the polishing cloth can be natural or synthetic. For polishing, a polishing paste or a distilled water suspension containing aluminium oxide, magnesium oxide, or diamond particles ranging from 0.05 to 15 µm in size, can be used, which should be compatible with the material and the hardness of the polishing cloth. During manual polishing, the sample should be moved in a circular motion opposite to the direction of rotation on the polishing disc. After polishing, the surface is washed with distilled water followed by alcohol. The applicability of alcohol should be checked for non-metallic samples. The surface is then dried with warm, blown air. Once the surface is polished, macroscopic examinations can be conducted. At 50 times magnification, certain cracks, welding defects, and inadequate fusion become visible. **Figure 3** shows a polished and etched sample obtained from a welded joint. In the case of cast iron, the graphite or certain non-metallic inclusions can be recognized, and their shape, size, and distribution can be examined.



**Figure 3.** Welded joint, polished and etched.

## 6. Surface Etching

More detailed examinations are conducted by etching the surface following the recommendations of ISO 16060 [7]. The most used etchants are summarized in [Table 1](#).

The etchant should be selected according to the quality of the material being examined. The etchant selectively attacks the sample surface to varying degrees at grain boundaries and grain surfaces, making them visible under a microscope. Altered microstructures resulting from heat treatment can also be effectively examined, especially in the case of tool steels [7]. Colour etching techniques provide more information and enhance recognition. The etchant forms a stable-coloured film on the sample surface, which can be an oxide, sulfide, chromate, or complex compound. Strict adherence to the mixing order and ratios is essential during etchant preparation.

**Table 1.** Etchants. [1, 2, 3, 6]

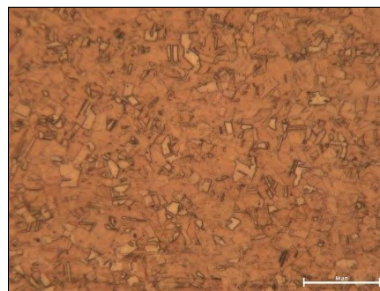
| Name                            | Etchant                                                                                                 | Application area                                  |
|---------------------------------|---------------------------------------------------------------------------------------------------------|---------------------------------------------------|
| Nital                           | 97 ml ethyl alcohol;<br>3 ml 69% nitric acid                                                            | Unalloyed and low-alloy steel                     |
| Pikral                          | 100 ml ethyl alcohol;<br>4 g picric acid                                                                | Unalloyed and low-alloy steel                     |
| Hydrochloric acid-iron chloride | 960 ml ethyl alcohol; 20 ml 35% hydrochloric acid; 50 g iron(III) chloride                              | Copper and its alloys                             |
| Keller                          | 950 ml distilled water; 25 ml 69% nitric acid, 15 ml 35% hydrochloric acid; 10 ml 40% hydrofluoric acid | Titanium and its alloys, aluminium and its alloys |
| Kroll                           | 10 ml 40% hydrofluoric acid; 30 ml 69% nitric acid; 960 ml distilled water                              | Aluminum and its alloys                           |

For chemically resistant materials, electrolytic etching can be used. In conventional etching, the sample surface is immersed in the etchant and gently moved. The etching time also depends on the microscope resolution, with longer durations required for lower-resolution microscopes and shorter durations for higher-resolution microscopes. After etching, the sample is rinsed thoroughly with water, followed by rinsing with alcohol, and then dried with warm blown air. The microscopic image of an etched surface of a copper sample is shown in [Figure 4](#).

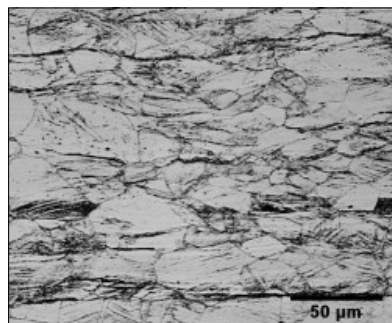
During the welding process in the case of the austenitic microstructure steels (ex. X5CrNi18-10, 1.4301) some precipitation in the heat-affected zone can be established, which decreases the corrosion resistance [8, 9]. Precipitations formed at the grain boundaries can be identified by metallographic examination ([Figure 5](#)) [10].

## 7. Conclusions

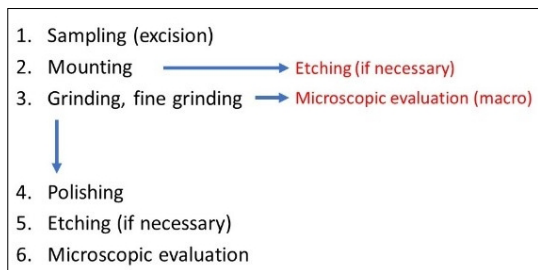
The macroscopic and microscopic examinations presented in this study are widely used in materials testing. [Figure 6](#) summarizes the steps involved in the examination of macro- and microstructures.



**Figure 4.** Etched copper sample (etched with hydrochloric acid-iron chloride).



**Figure 5.** Ausztenites rozsdamentes acél szemcséhatár-kiválások [10]



**Figure 6.** Steps in macroscopic and microscopic examination

These examinations must be conducted by properly trained personnel in materials testing who possess a high level of knowledge in both sample preparation and evaluation of the tests. The preparation of samples is typically a task performed at the laboratory technician level while conducting macroscopic examinations requires more knowledge and practice. Determining the microstructure of materials assumes a high level of expertise in materials science and practical experience. Naturally, traditional testing techniques can be complemented with modern methods offered by advanced equipment, such as scanning electron microscopy (SEM) or X-ray diffraction (XRD) for phase identification. For the examination of welded structures, the European Welding Federation (EWF) has established a specialized training system (EWF-627-07) for personnel responsible for the metallographic examination of structural materials and their joints produced by welding and related processes [11].

### Acknowledgment

The following work was carried out in the framework of the project registered under Erasmus+ 2021-1-RO01-KA220-VET-000025835, the authors want to declare acknowledgement for the support.

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