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Preparation of Specimens for Macro and Microscopic Examinations of Dissimilar Friction Welded Steel Joints and Their Evaluation According to Applicable Standards

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Summary
The article describes the issues related to the preparation for microscopic and macroscopic observation of dissimilar joints obtained by friction welding of 2H13 martensitic steel with B500B reinforcing steel. The results of etching and the main difficulties connected with the preparation of this type of samples for research were presented and discussed.

Keywords: specimen preparation, macroscopic examinations, microscopic examinations, dissimilar joint, friction welded joints

1. Introduction

The success of microscopic and macroscopic examination mainly depends on the quality of metallographic specimen preparation. This process consists of several steps and in some cases requires great care and attention. The specimen preparation procedure begins with sampling, followed by rough and fine grinding, and then polishing and etching. In the process of sample preparation, at each stage of processing the surface of the sample, two principles are most important:
1) avoiding excessive pressure on the sample surfaces,
2) avoiding overheating of the sample material.

Excessive pressure on the sample can cause surface plastic deformation, which leads to falsification of the true image of the microstructure of the examined material, while overheating the material can cause the formation of a new structure under the influence of heat, which does not reflect the real structure of the material [1]. Samples from welded joints are cut through the cross-section of the joint to the longitudinal axis of the weld so that the surface of the specimen includes the weld, the heat affected zone and part of the parent material. After grinding the sample, internal defects and inconsistencies can already be observed on the surface of the specimen, such as: cracks, lack of penetration, pores and cavities, non-metallic inclusions and some physical inhomogeneities, such as hardening of the heat affected zone. However, this information can be considered cursory and indicative, as it does not contain most of the data relevant to a proper evaluation of the joint's macrostructure.

Polishing can be carried out mechanically, electrolytically or chemically. Mechanical polishing is most often carried out at the Laboratory of the Railway Research Institute, as it is a universal method of processing materials across the entire hardness spectrum.

Full information about the macrostructure is obtained only after it is etched in a properly selected metallographic reagent. The type of reagent depends on the type of sample material and the purpose of the examination. The most commonly used macroscopic reagent used for steel, particularly in the evaluation of welds, is Adler's reagent, which was developed in 1934 by Otto Adler, an employee of the German Reich Railway Research Center in Wittenberg.

When revealing the microstructure, the aim is to contrastively differentiate the various components of the microstructure by selectively dissolving the polished surface of the specimen. Depending on the type of etching reagent used, different microscopic images can be obtained and different information about the examined material can be obtained. Due to the predominance of research conducted on iron-carbon alloys, Nital is used most often in the Laboratory of the Railway Research Institute. This reagent was developed by a Pole, Eng. Alfons Rzeszotarski, in the 1880s [2].

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The following part of the paper presents the methodology of sample preparation and the results of etching the specimens from dissimilar joints produced by friction welding of B500B reinforcing steel with martensitic alloy steel 1.4021 (2H13). The possibilities of qualitative evaluation of the joints and identification of welding imperfections based on macroscopic and microscopic examinations are also described.

2. Preparation of specimens

The examined specimen (Figure 1) was a friction welded joint, according to the developed welding technology, made between a ribbed bar of B500B reinforcing steel and martensitic alloy steel 1.4021 (2H13). The chemical compositions of the native materials are given in Tables 1 and 2.

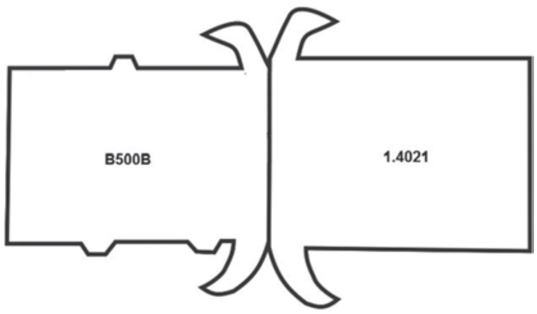

Fig. 1. Cross-section of examined joint with symbols of joined materials [own study]

Struers Rotopol-22 grinding/polishing machine was used for the preparation of the specimen (Fig. 2). The samples were grinded with diamond grinding discs of gradations: 80, 120, 600, 1200, and then polished with a 1 μm polycrystalline diamond suspension from Buehler.

Fig. 2. Grinding on a RotoPol-22 grinding/polishing machine of Struers mould in the Materials & Structure Laboratory of the Railway Research Institute [photo: M. Ostromęcka]

During grinding, particular attention was paid to how the sample was pressed against the disc. The dissimilar joint was composed of materials of similar hardness, but in the weld area the hardness was three times higher compared to the parent materials. This was the reason for the difficulty in grinding the sample, so it was crucial to the successful preparation of the sample that the pressure was uniformly distributed over the surface of the entire specimen. Prior to polishing, the sample was thoroughly rinsed under running water to ensure that no filings that could scratch the specimen were transferred to the cloth. Polishing was continued until the grinding scratches disappeared. After polishing, the specimen was rinsed again under running water, wiped with spirit and dried using hot air.

The macrostructure of the joint was revealed using the Adler reagent. The results of the etching with

Table 1

Chemical composition of reinforcing steel (parent material 1)

Reinforcing steel B500B	C [%]	N [%]	S [%]	P [%]	Cu [%]	Carbon equivalent C_{eq}
	≤ 0.24	≤ 0.013	≤ 0.055	≤ 0.055	≤ 0.85	≤ 0.52

[Own study based on approvals].

Table 2

Chemical composition of martensitic steel (parent material 2)

Steel 1.4021 (2H13)	C [%]	Mn [%]	Si [%]	P [%]	S [%]	Cr [%]	Ni [%]	Cu [%]
	0.16 – 0.25	< 1.5	< 1.0	< 0.04	< 0.03	12.0 – 14.0	–	–

[Own study based on approvals].

this reagent are shown in Figure 3. The chemical composition of the etching reagents, Adler and Nital, is defined by the American ASTM E407 [3] and Polish (withdrawn) standards: PN-61/H-04502 [4] and PN-61/H-04503 [5]. The current Polish standard presenting the chemical compositions of reagents for microscopic and macroscopic examination is a fusion welding standard and is designated PN-CR 12361: 2002 [6]. The chemical compositions of the reagents used in the preparation are given in Table 3.

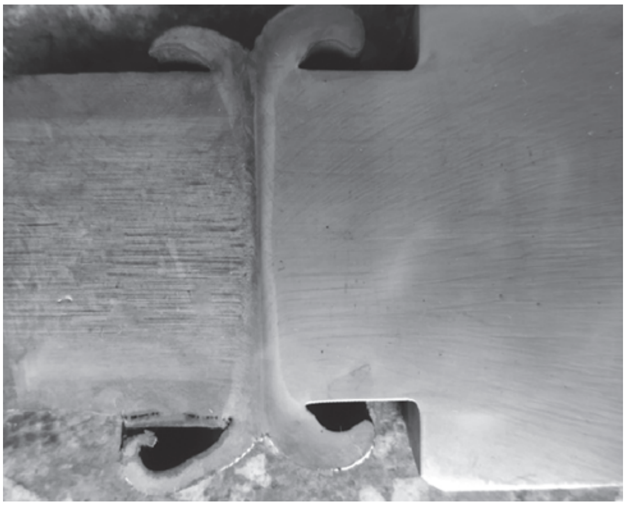

Fig. 3. Macrograph of a friction weld. Etching with Adler's reagent according to the withdrawn standard PN-61/H-04502 [4] [photo. M. Ostromęcka]

The surface should be observed during the etching process, as the process should be stopped when the revealed image of the macrostructure becomes sufficiently clear. According to the definition given in PN-EN ISO 17639:2013-12 [7], the term macroscopic examination refers to examination performed with naked eye or low magnification of the etched or un-etched surface**.** The purpose of the examination of fusion welded joints is to observe the weld zone, the heat affected zone, the number of seams and layers, and essentially to evaluate the quality of the joint, i.e. to identify possible imperfections.

The evaluation of joint quality is carried out on the basis of PN-EN ISO 6520-1:2007 [8] for fusion welding processes and PN-EN ISO 6520-2:2005 [9] for welding processes. These standards introduce the concepts of welding imperfections and, taking into account their size, they are divided into:

- macroscopic imperfections (e.g. excessive dents, overlaps, lack of fusion, etc.),
- microscopic imperfections (e.g. microcracks, non-metallic inclusions, brittle metallographic structures, etc.).

Sometimes sub-microscopic imperfections are also distinguished, which are detected by special methods of both destructive and non-destructive examination. Microscopic examinations are related to the observation of metallographic specimens in the etched or un-etched state using a microscope in the magnification range from 50 to 500 times. The aim of microscopic examination of joints is to determine the components of the microstructure in individual areas of the joints, to detect and identify defects as well as welding imperfections on a micro scale.

Identification of the components of a structure is made by observing their morphological characteristics, i.e. appearance, shape, colour and distribution. In some cases, it proves expedient to use quantitative metallography methods, where specialised image analysers based on specialised computer software are now usually used. When evaluating microscopic defects and imperfections, the focus is primarily on discontinuities, i.e.: cracks, slagging, non-metallic inclusions, and micro-inclusions.

Table 3

Chemical composition of Adler and Nital reagents according to existing standards

Standard	ASTM E407[3]		PN-61/H-04502 [4]	PN-61/H-04503[5]
Etching reagent	Adler	Nital	Adler	Nital
Chemical composition	9 g $(NH_4)_2CuCl_4$; 75 ml of water; 150 ml of HCl; 45 g of $FeCl_3$	1–5 ml of HNO_3; 100 ml of ethanol (95%) or methanol (95%)	3 g $(NH_4)_2CuCl_4$; 25 ml of water; 50 ml of HCl; 15 g of $FeCl_3$	1–5 ml of HNO_3; 100 ml of ethanol
Etching time	few seconds	from a few seconds to one minute	few seconds	from a few seconds to a few minutes
Application	etching of stainless steel and Hastelloy alloys	revealing the overall structure in iron alloys	weld examination – clear revealing of weld and welded layers	universal etching reagent for iron alloys

[Own study].

The etching of steel containing alloyed martensite is not possible with nitric acid. For this reason, the $FeCl_3$ reagent (a solution of $FeCl_3$ in hydrochloric acid and ethanol) is most commonly used. This reagent etches the grain boundaries and forms deposits on the grain surface, however, the use of the Adler reagent alone, which contains $FeCl_3$, will provide more information about the microstructure (compare Figures 4 and 5).

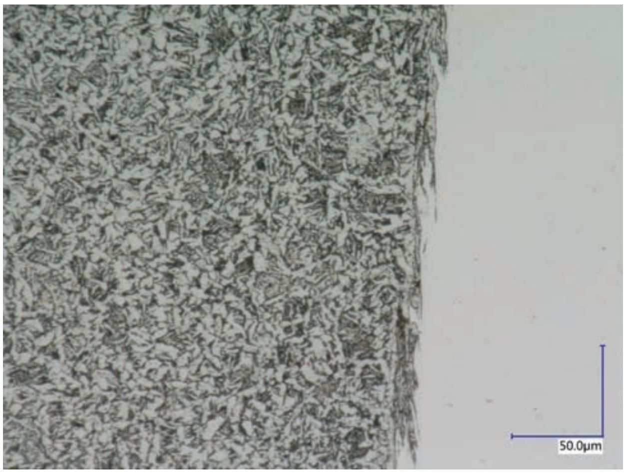

Fig. 4. Specimen after etching with 4% Nital; ferritic-perlitic microstructure revealed on the reinforcing steel side. The surface on the martensitic steel side remains smooth, as the alloyed martensite is not etched under the influence of nitric acid, which is the main component of Nital [photo: M. Ostromęcka]

Fig. 5. Structure revealed on the martensitic steel side after etching using only Adler's reagent; on the left, a fine-grained martensitic thermo-plastic zone close to the weld line [photo. M. Ostromęcka]

3. Possibilities of evaluating the imperfections of welded joints on the basis of macro- and microscopic examinations

As metallographic examinations are time-consuming and also often costly methods, they should be optimised before proceeding. For this reason, it is beneficial to know how the characteristics of the joint can be evaluated after the application of a given method (Table 4). According to the provisions of PN-EN ISO 6520-1:2007 [8], for fusion welding processes, and PN-EN ISO 6520-2:2005 [9] for welding, welding imperfections are classified as follows:

- Group 1 – Cracks; (No 100 for fusion welding processes P 100 for welding);
- Group 2 – Pores and cavities; (No 200 for fusion welding processes P 200 for welding);
- Group 3 – Inclusions; (No 300 for fusion welding processes P 300 for welding);
- Group 4 – Lack of fusion, incomplete penetration; (No 400 for fusion welding processes P 400 for welding);
- Group 5 – Shape imperfections; (No 500 for fusion welding processes P 500 for welding);
- Group 6 – Miscellaneous imperfections. (No 600 for fusion welding processes P 600 for welding).

4. Conclusion

The correct preparation of the metallographic specimen is the most important step in microscopic and macroscopic examination. In the preparation of a specimen of a dissimilar welded joint, it is extremely important that the whole sample is pressed uniformly against the grinding wheel during grinding. In the case of mechanised grinding, the pressure of the sample against the grinding wheel is most often applied pointwise, so that the sample may be grinded non-uniformly. Uniform pressure can be ensured by using grinding holders with lateral clamping of the samples to the holder, however, in many cases, the sample preparation process at the Railway Research Institute is carried out manually due to the large size of the examined samples. This requires a lot of dexterity and experience and sometimes has to be repeated several times. Samples with surfaces of varying hardness are prepared manually each time, which requires particular focus during preparation.

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Table 4

Possibilities for the evaluation of characteristics in macroscopic and microscopic metallographic examination

Description of inconsistencies	Welding incompatibility according to PN-EN ISO 6520-2	Macroscopic examination without etching	Macroscopic examinations with etching	Microscopic examination without etching	Microscopic examinations with etching
Hot crack	P 100	X	X	X	X
Cold crack	P100	X	X	X	X
Lamellar crack	P 100	X	X	X	X
Pores and cavities	P 200	X	X	X	X
Inclusions	P 300	X	X	X	X
Lack of fusion, incomplete penetration	P 400	X	X	X	X
Imperfections in shape	P 500	X	X	–	–
Heat affected zone	P 600	–	X	–	X
Seams and layers	P 600	–	X	–	X?
Grain boundaries	P 600	–		X?	X
Grain structure	P 600	–			X
Primary structure	P 600	–	X		X
Preparation of edges for welding	P 600	X?	X	X?	X
Rolling direction	P 600	–	X	–	X
Structure directivity, texture	P 600	–	X	–	X
Segregation	P 600	–	X	–	X
Separations	P 600	–		–	X
Repair welds tracs and imperfections	P 600	X?	X	X?	X
Thermo-mechanical effects	P 600	–	X	–	X
X – characteristic revealed with certainty					
X? – characteristic that may or may not be revealed					

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