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PRZEGLĄD SPAWALNICTWA
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Application of color etching to study the microstructure of TRIP steel after laser remelting

Abstract

TRIP type steels have a multi-phase structure, which includes such phases as: austenite, bainite, ferrite and martensite. The presence of so many co-existing phases creates difficulties in their accurate identification. One of the methods used to identify the components of the microstructure is color metallography. Methods of color metallography in contrary to some methods of microstructure identification (e.g. TEM, EBSD) are simple to use, cheap and not very time-consuming. However, there are still no detailed recommendations on the use of this method. The paper examines the possibilities of application of colored etching methods, to distinguish the components of the microstructure of the as-received material and the welds of the TRIP type steel. Light microscopy methods were used for the study. The obtained results allow for a qualitative distinction of individual components of the microstructure.

Keywords:
TRIP steel; colour etching; welded joint; microstructure

Introduction

The drive of the automotive industry, to increase the passive safety of passenger cars and to reduce their mass, led to the design of new steel grades at the end of the 20th century. The AHSS (Advanced High Strength Steel) group has gained high popularity, which includes such species as: DP, TRIP, CP and MS. Materials from this group are most often steels characterized by a multi-phase structure. The presence of a multi-phase structure allows to obtain properties which until now have not been possible to obtain in steels. Hard phases such as bainite and martensite provide high strength, and soft phases such as ferrite and austenite are responsible for high plasticity. Therefore, AHSS steels are also characterized by high strength parameters (YS and UTS) and good plasticity (A) [1,2].

TRIP (Transformation Induced Plasticity) steel is characterized by a multi-phase structure, which includes: austenite, bainite and martensite distributed in a ferritic matrix. Austenite occurs in the form of free grains or is found between the bainite and/or martensite grains (plates) in the MA and BA isles. The γ phase is very important because it is stable at room temperature (its content can reach up to 20% of its volume). It is responsible for good plasticity and ductility and can undergo martensitic transformation under the influence of plastic deformation. This last feature is very important due to the use in car bodies. Along with the martensitic transformation occurring during plastic deformation (eg car accident), energy is absorbed in favor of this transformation. Therefore, TRIP steel has good strength properties (UTS to 1000 MPa) and good ductility (A over 20%) and thanks to the martensitic transformation in austenite during deformation, it is able to absorb some of the impact energy, which directly affects the increase of passive safety of the car and reduction of its mass [3÷5].

TRIP steel is a very interesting material due to the presence of a very rich multi-phase structure. However, such a structure, or rather its individual components, are difficult to identify using standard methods. Therefore, one of the methods that is to better identify these components is color metallography. Thanks to the use of various metal etching reagents, it is possible to isolate individual components of the microstructure.

Color metallography is not a commonly used method to differentiate the components of a microstructure. This is due to the lack of instructions for its use in Polish and several parameters that make testing difficult and have a direct impact on the end result.

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Research methodology, material for testing

The subject of the research was TRIP690 steel (the minimum UTS value is 690 MPa), whose chemical composition is given in Table I. The chemical composition was determined using the LECO GDS500A chemical composition analyzer.

The material tested was melted with a laser to reproduce changes in the microstructures occurring after the welding process. These steels are commonly laser welded, and then embossed in TWB technology and used as elements of the car body. Therefore, a remelting process was used that was easier to carry out than the welding process.

Table I. Chemical composition of researched steel

Chemical Composition [%]				
C	**Mn**	**Si**	**Al**	**Ni**
0,218	1,65	0,102	1,46	0,02
S	**P**	**V**	**Ti**	**Fe**
0,002	0,02	0,001	0,004	Reszta

The remelting was carried out using a TRUMPF TruDisk 4002 solid-state disk laser. The active laser center is a Yb: YAG garnet crystal. The remelting of the sheet was made with a 2 kW laser with a beam speed of 2.1 m/min. The speed has been selected experimentally, in such a way that full material melting occurs, without inconsistencies and to obtain the smallest possible heat affected zone (HAZ). The beam was focused on the surface of the material. The thickness of the sheet was 1.5 mm.

The tested material was cut into small elements. Cut out elements have been mounted and then further grinded and polished, finishing the polishing with the use of a diamond suspension with a granulation of 1 μm.

The test samples were then etched and dyed using the reagents listed in Table II. Each of the samples was pre-etched with nital (Mi1Fe) or pikral (Mi3Fe), and in the next stage dyed (etched) with one of the three reagents: Klemm, LePera and sodium metabisulfite. After each etching, the microstructure of the material was recorded using the NIKON ECLIPSE MA200 light microscope with the NIS Elements BR software.

Macroscopic tests were also carried out, stating the lack of visual welding incompatibilities. In addition, hardness measurements of the as-delivered material and weld were also made. The measurements were made using the Vickers method, with the MMT-X3 microhardness tester, in accordance with PN-EN ISO 6507-2: 2007. The measurement time was 15 s and took place under a load of 200 g (ie 1.961N).

Table II. Chemical reagents for etching metals

Name	Chemical Composition
Nital	4% solution of nitric acid in ethanol
Pikral	4% solution of picric acid in ethanol
Klemm	50 ml ($Na_2S_2O_3$ w H_2O) + 1g $K_2S_2O_5$
LePera reagent	1:1 – pikral + 1% solution $Na_2S_2O_5$ w H_2O
Sodium metabisulfite	10% water solution $Na_2S_2O_5$

Research results and their discussion

This work presents the results of the TRIP steel microstructure and microhardness tests. First of all, images of the microstructure of the as-delivered material, HAZ and the weld were presented. Subsequently, for the purpose of comparison, the results of microhardness measurements of the material were shown.

Native material

Research on the microstructure of the as-received material TRIP steel showed the presence of a fine-grained structure with a directional character that is the effect of rolling the metal sheet. This orientation of the structure is called bandwidth. As a result of nital etching, the separation of various phases against the background of a bright ferritic matrix was observed, which after the use of this reagent are unidentifiable (Fig. 1).

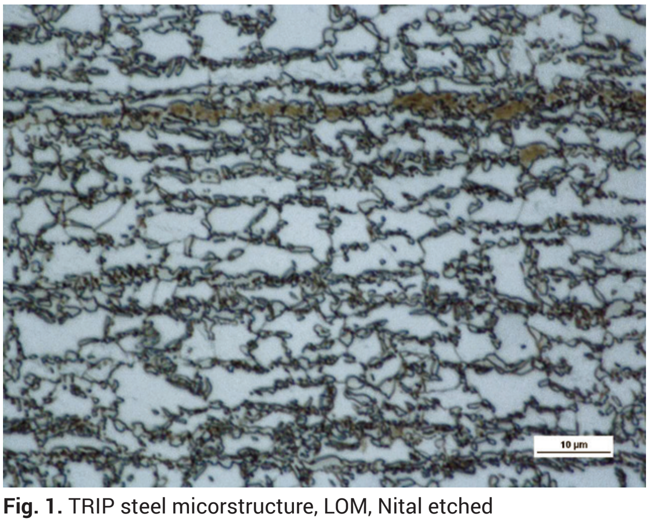

Fig. 1. TRIP steel micorstructure, LOM, Nital etched

As a result of the etching of TRIP steel with nital and Klemm's reagent, the ferritic phase was stained with beige, bainite and martensite remained indistinguishable – dark navy blue and the austenite grains and the MA isles remained white (Fig. 2a) [7,8].

As a result of the use of pikral, the material was etched like in the case of nital etching: there is a bright ferritic matrix and darker separations of the remaining phases (Fig. 2b).

The etching of the as-received material with the pikral and LePera reagent caused the ferrite color to turn into a light beige color, the bainite became dark blue and dark brown and the austenite, martensite and MA isles remained white – austenite and martensite are indistinguishable (Fig. 2c) [7].

Application of pikral and 10% aqueous solution of sodium metabisulfite for etching, allowed for qualitative differentiation of the basic components of the microstructure. The ferrite turned bright blue; bainite, characterized by the presence of carbides in its needles, turned dark blue, martensite (and carbide-free bainitic structures) took the color of straw, and the austenite remained white. Quantitative distinction of individual components of the microstructure is not possible for such a fine-grained steel as TRIP steel. This is due to the limitations of the light microscope, as a result of which it is impossible to obtain the magnifications necessary for quantitative evaluation of individual phases, whose size was often below 1μm (Fig. 2d) [6,9].

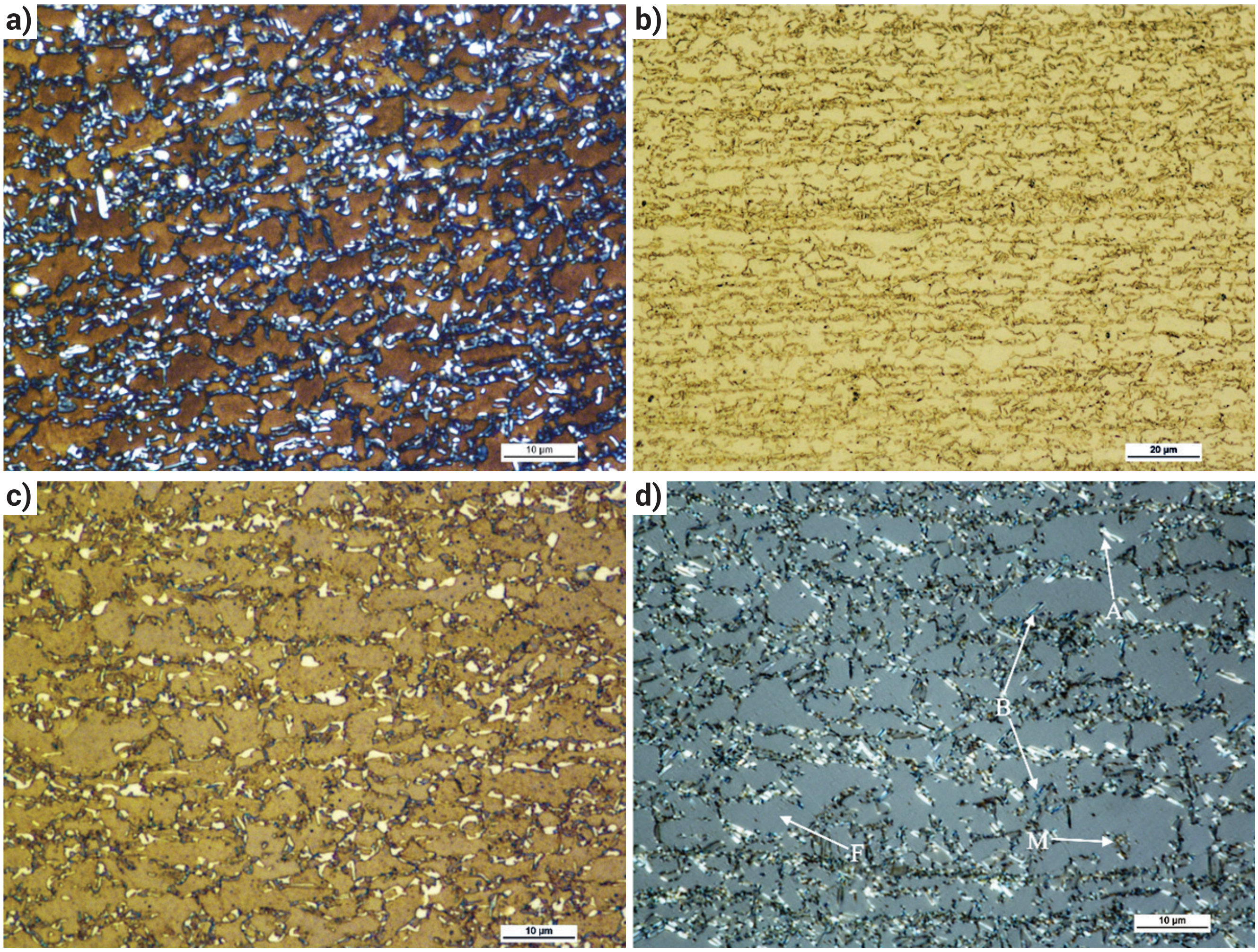

Fig. 2. TRIP steel micorstructure, LOM: a) Nital + Klemm reagent etched, b) Pikral etched, c) Pikral + LePera reagent etchant etched, d) Pikral + sodium metabisulfite etched

HAZ and the weld

Investigations of the heat affected zone and welds of TRIP steel after laser remelting showed the presence of a significantly different structure, from the structure of the as-delivered material. As a result of nital etching, the image of the HAZ microstructure revealed the presence of Widmanstätten's coarse structure, lath martensite packets and the structure of ferrite needles inclined to one another at the angles of 60° and 120° (Fig. 3a). The low carbon content in the structure (less than 0.1%) and the rapid cooling that occurs after the bonding process favors the creation of the Widmanstätten's structure.

Analysis of the chemical composition showed a carbon content of 0.22% (Table I). This carbon content favors the formation of a lath martensite structure after the hardening process (or after bonding processes), which is mostly found in the joint of the tested material. The local increase in carbon content in austenite was the reason for the formation of plate martensite or bainite, the presence of which indicates the arrangement of the plates under characteristic angles. In the joint a ferritic structure was observed, with a characteristic morphology, which should be identified with the feathery bainite (Fig. 3b) [10].

The analysis of the microstructure with the use of nital and Klemm's reagent for etching revealed the presence of a characteristic plate structure with different crystallographic orientations (Fig. 3c). The morphology of the structure would, in the first place, indicate the presence of a pearlitic structure. However, careful analysis suggests the existence of a plate-like structure of the upper bainite. The following facts support the occurrence of the upper bainite: the thickness of the observed bainite plates is about 0.5 µm, which is confirmed by the scientific literature [10]; the chemical composition of the steel (CTPi curve) is not conducive to the passage through the perlite occurrence area, in the case of high cooling rates that occur during bonding processes; low carbon content, amounting to approx. 0.22%, is not conducive to the occurrence of pearlitic structure, especially at such high hardness in the weld. Etching with nital and Klemm's reagent resulted in revealing the structure of the lath martensite in light beige color. The dark beige color may suggest the occurrence of lower bainite, whose morphology is almost identical to the martensite morphology, however, it is etched stronger (darker) than martensite, due to the presence of the second phase – carbides. Nevertheless, due to the fact that the structure is etched with Klemm's reagent, the supposition may be wrong. Upper bainite was revealed in the area of characteristic white plates (Fig. 3c) [10÷13].

The use of pikral to etch heat affected zone and weld allowed to reveal the dendritic structure present in the weld. The area of occurrence of the dendritic structure clearly separates the HAZ, in which the as-received material has normalized and overheated, from the weld, where the material has been remelted (Fig. 3d).

The principle of pikral's activity is different from nital. Nital etches ferrite and grain boundaries, while pikral etches carbides and interfaces between ferrite and carbides.

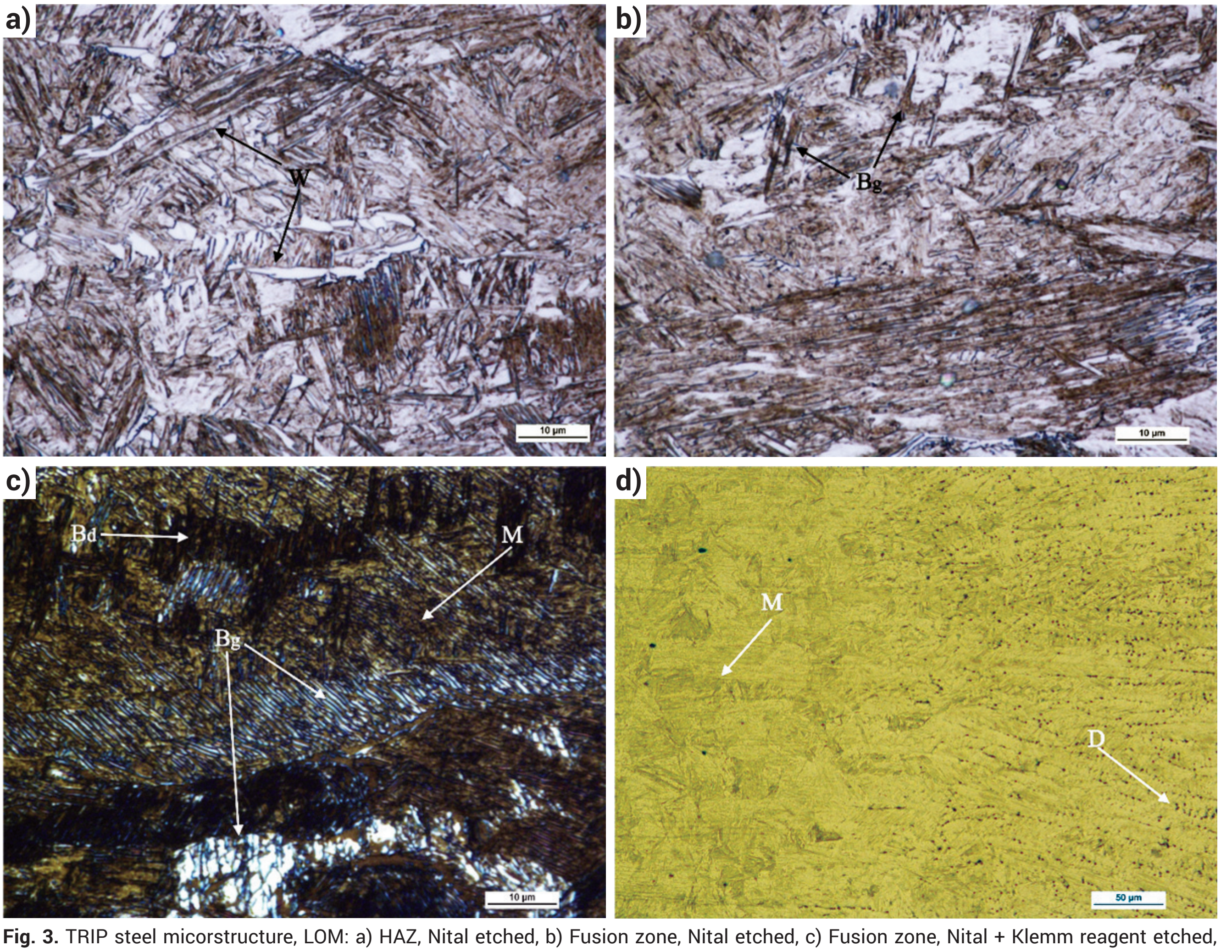

Fig. 3. TRIP steel micorstructure, LOM: a) HAZ, Nital etched, b) Fusion zone, Nital etched, c) Fusion zone, Nital + Klemm reagent etched, d) Fusion zone and HAZ, Pikral etched

Therefore, the use of pikral is justified in the etching of steels in which carbides are present. In TRIP steels, fine carbides are found on the boundaries of austenite grains, therefore it is possible to reveal this phase. The use of pikral is also justified in the case of etching of bainitic structures that contain carbides. The use of pikral to etch steel welds of the TRIP type allowed the separation of bainitic structures containing carbides. These structures should be identified with bainite or bainitic ferrite and Widmanstätten's irregular needles (Fig. 4a) [11, 12].

The etching of the structure with the help of pikral and LePera reagent and pikral with 10% aqueous solution of sodium metabisulfite allowed to reveal the areas of bainite occurrence. Referring to the publication no. 9, the use of reagents containing sodium metabisulfite and pikral for etching leads to the coloration of carbide-containing structures in the dark-blue color.

As a result of observation of the microstructure, it was found that there are upper bainite areas whose morphology has a plate-like character (Fig. 4b, Fig. 4c). As expected, the bainitic structures grow from the grain boundaries of the former austenite. The remaining volume of the material showed the occurrence of martensite (beige color). In addition, the areas of clearly visible lath martensite packs and upper bainite with feathery morphology were observed (Fig. 4b). The microstructure images also include light (white) zones with a feather shape, which should be identified with bainitic ferrite. Bainitic ferrite (probainitic) is a structure depleted or containing no carbides in relation to bainite and is formed as a result of the failure to fully bainitic transformation [9,10].

The white areas between the bainite plates/needles can also be an austenitic phase, the color of which matches the assumptions. The presence of residual austenite in the TRIP steel weld, after laser welding, has been proven in other studies, using magnetic methods and XRD. Austenite can also occur between martensite/bainite needles and be imperceptible when using light microscopy methods (Fig. 3c, Fig. 4d) [11,14].

Microhardness

Hardness measurements were carried out to determine the hardness distribution in the material structure, after the bonding process and to confirm the observed microstructures (Fig. 5, where "s" is the distance from the center of the weld in mm).

The hardness of the as-received material of the tested TRIP steel was 224 HV0.2. The hardness in the weld was characterized by a value greater than approx. 200 HV and reached the value of 450 HV0.1. Such hardness is not achieved by pearlitic structures with this carbon content. In addition, martensitic structures with this carbon content are characterized by a hardness of 50 HRC, which is a value greater than that achieved. Therefore, the achieved hardness values confirm the occurrence of bainitic structures in the weld. In addition, an increase in the hardness of the material

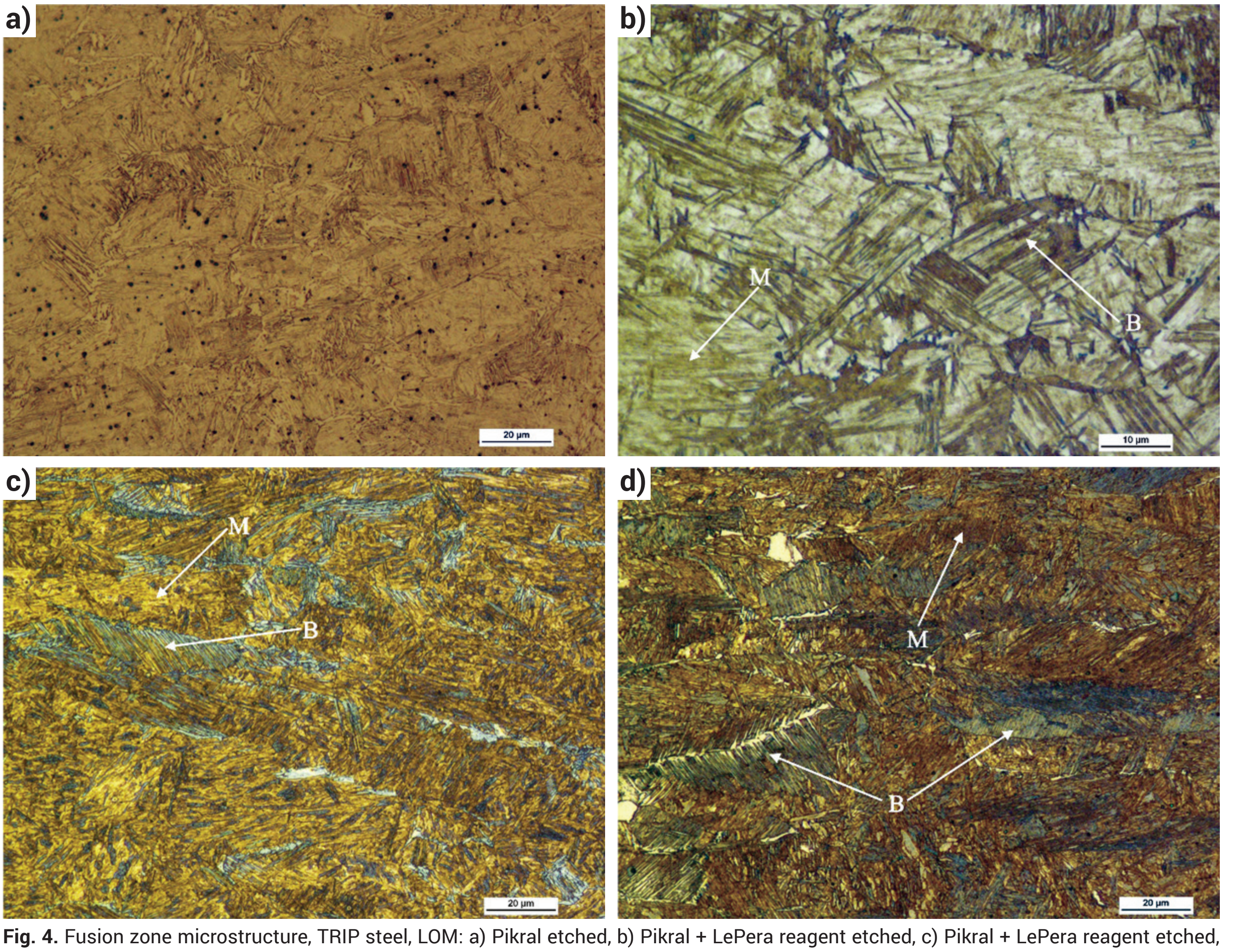

Fig. 4. Fusion zone microstructure, TRIP steel, LOM: a) Pikral etched, b) Pikral + LePera reagent etched, c) Pikral + LePera reagent etched, d) Pikral + sodium metabisulfite etched

in the heat affected zone was noted at a distance of 0.6 mm from the center of the weld. Such an increase should be interpreted due to the presence of a fine-grained structure occurring at the location of the HAZ, in relation to the coarse-grained structure occurring in the place of material overheating (s = 0.4 mm). The metallographic tests carried out confirmed the differences in grain size of the joint tested in the above-mentioned places [12,14].

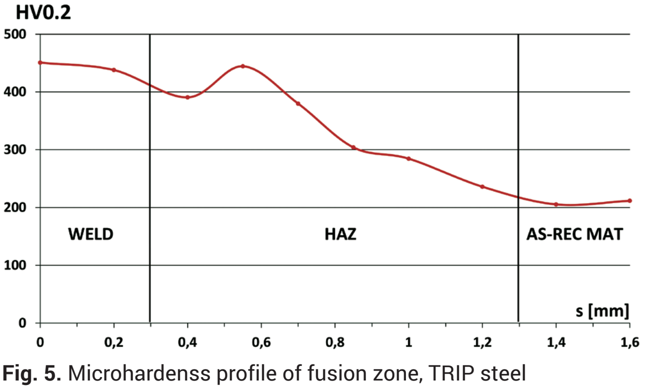

Fig. 5. Microhardenss profile of fusion zone, TRIP steel

Summary and Conclusions

1. The conducted tests confirm the validity of the application of colored metallography for revealing the components of the microstructure of the as-received material and the weld of the TRIP type steel. Further works are planned using color metallography, for testing AHSS steel and others.
2. The study of as-received material from TRIP type steel, using a variety of etched reagents, allowed revealing all the basic components of the microstructure. Each time the ferrite was colored in a bright color. The Klemm's reagent allowed the isolation of austenite, LePera reagent to isolate bainite. The use of pikral and 10% aqueous solution of sodium metabisulfite allowed to isolate, in the image of the microstructure: austenite, bainite ferrite and martensite.

3. The weld and heat affected zone tests allowed to isolate areas of the upper plate-type bainite from the microstructure, in which the packs of lath martensite prevailed. Usually conducted research using nital does not allow for such a thorough analysis. The occurrence of plate martensite, upper bainite with feathery morphology, probainitic ferrite, dendritic structure and Widmanstätten was also observed. The possibility of residual austenite and lower bainite presence in the weld and HAZ structure was marked.
4. Microhardness tests confirmed the occurrence of previously observed microstructures. An increase in hardness of approx. 200 HV was observed in relation to the as-received material (224 HV0.2). Such a high increase in hardness may be the reason of cracking of the weld under certain conditions. The total change in the microstructure of the material, as confirmed by the results of hardness measurements, led to a change in the properties of the TRIP steel, which will be characterized by lower plasticity and lack of residual austenite, which consequently leads to a local loss of energy absorption capacity and can be a sensitive point of construction.
5. The obtained test results may be application-oriented and may lead to the creation of more detailed instructions on the use of color metallography. The conducted research allowed for a qualitative assessment of individual components of the microstructure. However, such studies also require confirmation by more advanced methods like XRD and EBSD or electron microscopy. Such tests will allow to perform the quantitative assessment.

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