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THE PHENOMENON OF HYDROGEN EMBRITTLEMENT IN HIGH-STRENGTH BOLTS

The hydrogen embrittlement of metals is caused by the penetration and accumulation of hydrogen atoms inside the metal. The failure of the product due to hydrogen embrittlement is delayed in time and does not occur immediately after its manufacture, but several hours, days, or even weeks later. Therefore, the chances of detecting hydrogen embrittlement when checking the quality of the finished product are very slim. The use of high-strength bolts in industry is associated with the risk of hydrogen embrittlement. This phenomenon poses a threat to the safe use of devices by limiting or completely losing the functionality of the bolt joint. Even a low influence of moisture can trigger failure mechanisms.

The article proposes a method for assessing the risk of hydrogen embrittlement for high-strength bolts in class12.9. For this purpose, bolts made of material grade 32CrB4 were prepared and in a controlled manner the grain flow inconsistency was made, leading in extreme cases to the production of the forging lap. To perform the study, the device proposed by the European Assessment Document (EAD) was adapted to the testing of hydrogen embrittlement of threaded fasteners in concrete. The concrete substrate was replaced with metal spacers that were preloaded with a bolt. The use of the wedge distance under the bolt head led to the generation of two stress states – tensile and compressive, which translated into an increased risk of hydrogen embrittlement. After being tested, the bolts were visually and microscopically inspected to assess potential locations for cracks and hydrogen propagation. As a result of the conducted tests, it was found that the prepared test method allows to assess the resistance or susceptibility of the bolt to threats related to hydrogen embrittlement.

Keywords: cold forging; hydrogen embrittlement; high-strength bolts

1. Introduction

The use of threaded fasteners in industry significantly reduces the costs of manufacturing and operating machines. The possibility of quick disassembly of the machine element, reuse of the structure, and ease of application of threaded fasteners are some of the many advantages of using the bolt [1]. However, merely using the bolt produced by a forging process or machining from a material as delivered is ineffective. The most popular type of fasteners are bolts after heat treatment with a given class of mechanical properties [2]. Heat treatment carries many risks in use, especially the brittleness of type I and II tempering, resulting from incorrect selection of process or material parameters [3]. The process of manufacturing bolts is also not free from threats such as compliance with the grain flow pattern, no notches or defects in the form of lap forgings [4]. Going back to the beginning, attention should be paid to the steel production process, where it is possible to obtain nonmetallic inclusions that may contribute to early destruction of the element [5]. All the above-mentioned threats may not be detected for years and it may turn out that the bolt will work in the assumed time without failure. The factor that may lead to the visualization of the defects mentioned above is the phenomenon of hydrogen embrittlement (HE).

The HE problem has been present in industry, particularly the automotive industry, for years. The risk of sudden loss of functionality by the element poses a huge threat to vehicle users and the environment. For the average user, it is unimaginable that the braking or steering system in a speeding car suddenly fails. The constant development of the industry and the limitations resulting from the care of the environment impose on the constructors the use of steels of higher and higher strength. This leads to the continuous development of the steel grades used, but also to advanced heat treatment. In recent years, many studies have investigated the phenomenon of hydrogen embrittlement in fasteners, sheets, and profiles made of high-strength steel [6-11].

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The HE can be defined as the permanent loss of plasticity in a material that occurs when three factors are met:
– the element that is in danger of HE has a tensile strength of at least 1000 MPa;
– stress has been created in the element;
– a source of atomic hydrogen.

HE is divided into two groups due to the origin of hydrogen:
– internal hydrogen embrittlement (IHE), i.e. residues of residual hydrogen in the material from metallurgical or production processes;
– environmental hydrogen embrittlement (EHE) resulting from the working conditions of the bolt joint and the accompanying corrosion, where hydrogen is released [12-14].

Currently, much work has been done on the basis of the case study of the bolt after failure. Guo-he et al. In their work, present research on the case of a damaged high-strength hub bolt used in aviation. The hub bolt has failed due to a combination of two mechanisms, hydrogen embrittlement and the fatigue effect. The propagation of the crack began with pitting on the surface, which was formed even before assembly. It means errors in the product production process [15]. Ferraz and Oliveira tested two failure cases of the bolt prepared in mechanical property class 10.9 according to ISO 898-1. The complete analysis performed on the samples ruled out many factors, leaving the occurrence of hydrogen embrittlement as a real cause. The point of failure of the bolt that occurs in the radius between the head and the pin is one of the most heavily loaded points in the connection of the loaded bolt [16]. Villaba and Atrens studied the problem of cracking of the rock bolt due to stress corrosion. They compared publications on stress corrosion cracking (SCC) and HE with their research [17]. The tests carried out on bolts prepared in the classes of mechanical properties 10.9 and 12.9 were presented by Ham et al. Hydrogen-charged bolts were subjected to a low strain rate static tensile test. The results obtained on the tensile strength of the hydrogen-charged bolts were lower than before the test [18].

Based on the literature review, it was found that more intensive testing of bolt products was necessary due to HE. Currently, research performed on bolts focus on the mechanical properties and geometry of the product. The authors propose a method to assess the reliability of a threaded connection (bolt) under HE conditions at the time of bolt exploation. The article describes a test that allows one to actually verify the correctness of technological assumptions and the production process in conditions that favor HE.

2. Research methodology

2.1. Sample preparation

The bolts with a flange head according to the DIN 6921 standard were made of 32CrB4 material. The material for production was prepared from wire rod, which was processed by pickling, annealing, zinc phosphate coating and wire drawing. The bolts were prepared in two variants of settings in the multi-operational forging process. A Sacma forging machine was used to forge the products. In the case of the tested bolt, the following steps were performed:
– cutting the wire into sections of fixed length,
– operation I, initial upsetting of the material in the area of the bolt head and reduction of the pin diameter,
– operation II, forming a cylinder by upsetting in the area of the bolt head,
– operation III, forming an initial outline of the hexagonal geometry of the bolt head,
– operation IV, shaping the hexagonal head of the bolt with a collar and marking,
– tapping the pin by trimming operation,
– thread rolling.

The variations of the samples differed by the change of the constant volume principle for one of them, and thus by the change of the pattern of the fiber arrangement in the part of the bolt head (Fig. 1). In one variant, in the first forging operation, the material was upset from a too small part of the volume of the prepared wire semi-product. More material was left in the bolt pin. As a consequence, in the following operations, the bolt material was forced into the bolt head, leading to the phenomenon of lap forging on the bearing surface of the bolt flange (Fig. 1c).

2.2. Material

The prepared samples were heat treated on CAN-ENG continuous furnaces. The material was heated to a temperature

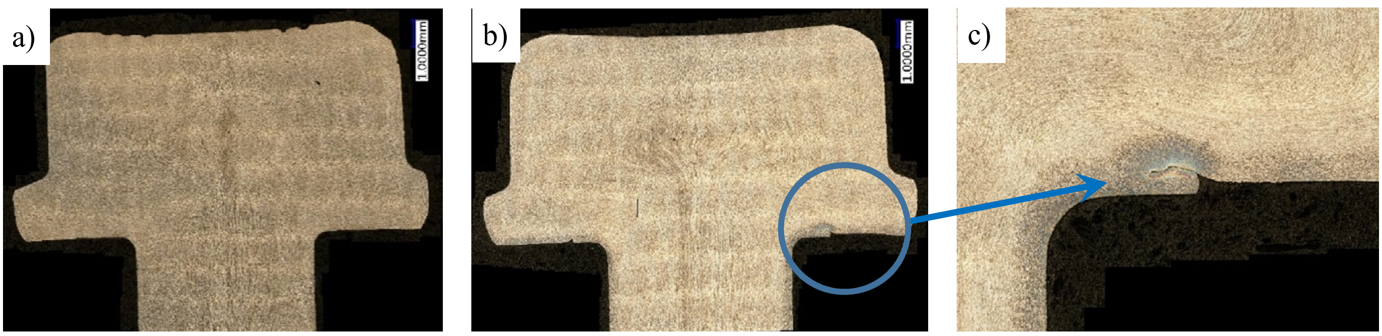


Fig. 1. Samples of polished longitudinal sections of DIN 6921 M6x20 bolts after the forging process with the grain flow pattern. a) variant compatible with the technology used, b) variant with a deliberate defect of lap forging the bearing surface, c) the lap forging of the bearing surface

of 880°C and then quenched in oil. In a further step of the line, a tempering was performed at 460°C. The process was conducted in an endothermic atmosphere with a neutral potential. After metallographic evaluation, the bolts did not have heat treatment defects, such as surface carburization or oxidation, or a change in the hardness of the thread surface relative to the bolt core greater than 20 HV. Bolts made of 32CrB4 material (TABLE 1) have been upgraded to class 12.9. The results of the mechanical properties are presented in the the TABLE 2. Hardness and tensile strength were checked according to the ISO 898-1 standard. Thus, the first condition for the existence of HE was met, that is, material strength greater than 1000 MPa and hardness greater than 32 HRC.

To increase the susceptibility of the bolts to the phenomenon of HE, the electroplating process was carried out on the production line, reaching the thickness of the coating from 8 to 11 microns.

2.3. Research stand

The tests were carried out on equipment adapted to the requirements of the European Assessment Document (EAD) to test the effect of hydrogen embrittlement on threaded fasteners installed in concrete (Fig. 2). The first modification was the change in substrate; the loaded bolt assembly and the solution with the electrodes were placed directly in the container, ignoring the influence of the concrete environment on the experiment (Fig. 3). The exchange of substrate in the study had no significant influence on the obtained results. The use of a concrete foundation in a standard test is used to anchor the tested fastener. By anchoring, it is possible to create a tensile stress in the fastener. One of the factors that is required for the appearance of HE is the generation of tensile stress. The influence of the substrate on the pH of the solution in the standard test was also negligible, because

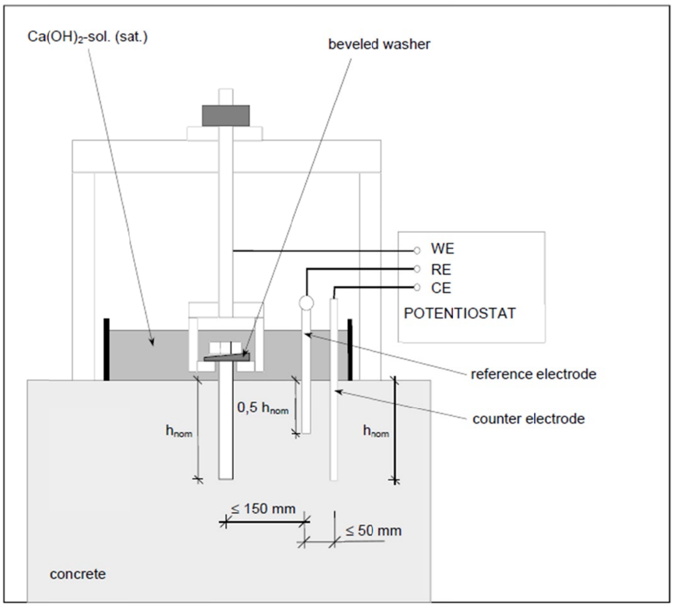


Fig. 2. Scheme of the hydrogen embrittlement test according to European Assessment Document (EAD)

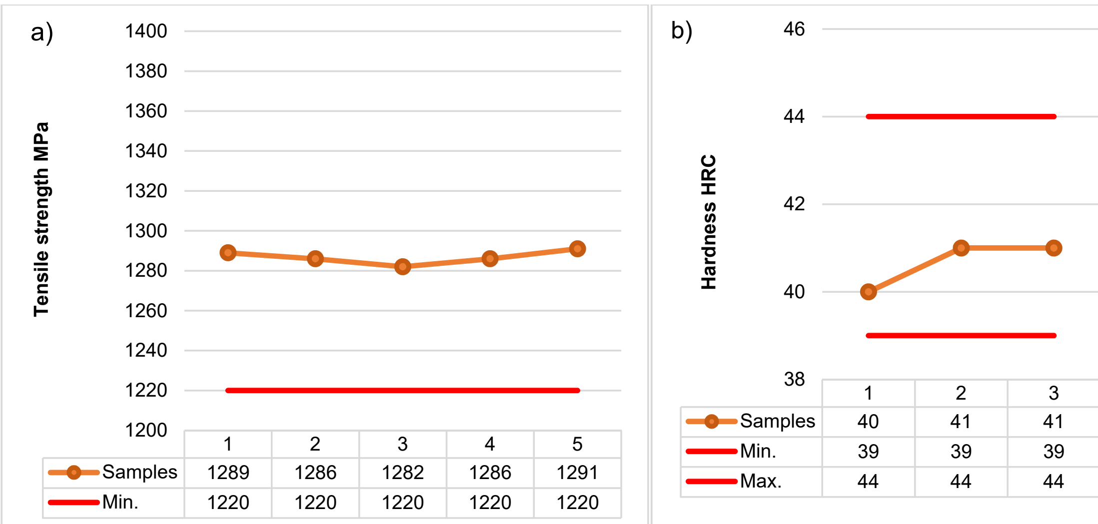
TABLE 1

Chemical composition of the 32CrB4 grade of the wire used for bolt production

		C [%]	Si [%]	Mn [%]	P [%]	S [%]	Cr [%]	Cu [%]	B [%]
32CrB4	min.	0.300	—	0.600	—	—	0.900	—	0.0008
	max.	0.340	0.300	0.900	0.015	0.015	1.200	0.250	0.005

TABLE 2

Mechanical properties of bolt products made of 32CrB4 material after heat treatment for mechanical property class 12.9 according to ISO 898-1. a) Tensile strength, and b) core hardness



a saturated solution based on calcium hydroxide was used with a pH value of 12.6, which is similar to the pH of concrete. Exchange of the substrate did not affect the emerging electropotential between the liquid and the tested material. The use of the actuator generating a constant load of the connector was abandoned and replaced with a sleeve and a nut (Fig. 4 and 5). In this way, three test stands connected to a potentiostat were prepared.

An environment conducive to the evolution of hydrogen on the bolt surface was created via

– preparation of a saturated solution of calcium hydroxide, which was used as an electrolyte;
– constant electromechanical conditions, a constant potential of –955 mV and a normal hydrogen electrode (NHE) were used;
– potential control by the control electrode.

2.4. Stress arrangement

A wedge washer (Fig. 4a) with a surface inclination angle of 4 degrees relative to the base was used to differentiate a stress state. The angle value was adopted according to the guidelines of the wedge strength test of ISO 898-1. As a compression element, a specially designed sleeve was used for the test (Fig. 4b) with technical holes made, allowing the free flow of the solution towards the bearing surface and the bolt thread. Both elements were made of structural alloy steel with a hardness of 40-41 HRC.

The bolts were compressed with a controlled tightening torque (TABLE 3). ŁT-43 grease was used in the assembly of the bolts, whose task was to stabilize the friction coefficient. Grease was applied to the thread and the bearing surface of the nut. Experimental tests on a Kistler tribological tester determined the value of the total coefficient of friction of the ŁT-43 lubricant at 0.10. The assembly procedure was prepared according to the EN 16047 standard [19]. The bolts were mounted using a torque wrench set for the torque shown in TABLE 3. Thus, the value of 0.85 FpC was obtained. The FpC value is related to the clamp force generated in the joint. DIN 934 class 12 nuts were used for assembling the bolts. After 1 hour, the bolts were tightened to the required torque to limit the effect of relaxation on the loss of clamping force. The assembled set shown in Fig. 5 guaranteed constant bolt load throughout the test.

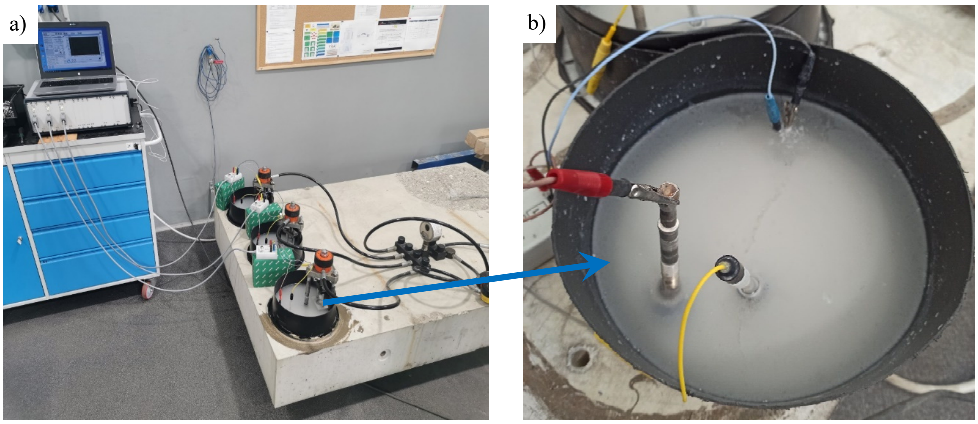


Fig. 3. a) Test stand for the effect of hydrogen embrittlement on fasteners with a common potentiostat, and b) test stand after adaptation for testing high-strength bolts

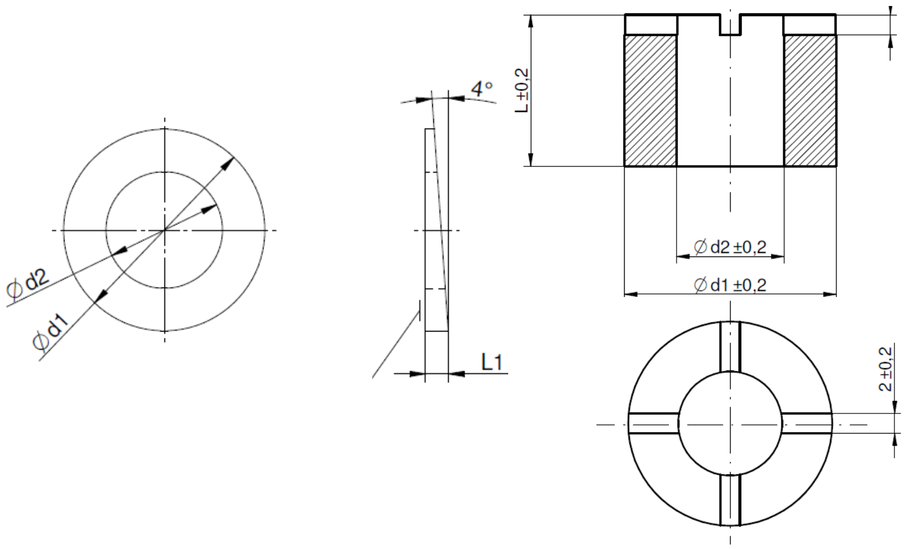


Fig. 4. a) The wedge washer with an inclination angle of 4 degrees, and b) the distance sleeve with holes

TABLE 3

Assembly parameters of a bolted connection

Mechanical property class	Cross-sectional area of the bolt, mm^2	Actual tensile strength, MPa	Clamping force required for installation at 0,85FpC, kN	Required tightening torque, Nm
12.9	36.6	1300	40.4	52

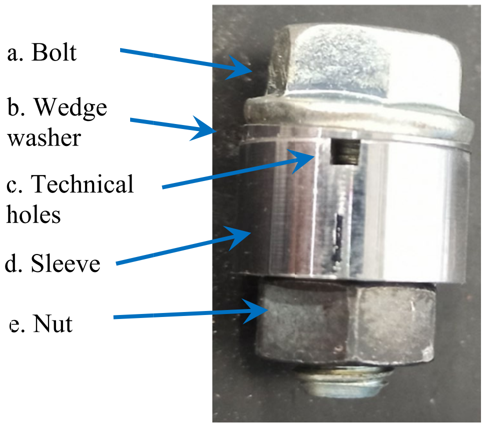


Fig. 5. Bolt set after assembly before performing the HE test

2.5. Research plan

Four variants were planned for testing (TABLE 4). The influence of the environment and surface defects on the possibility of causing failure or nucleation of cracks in the sample was taken into account. Adequate material hardness and bolt assembly stresses have been considered necessary for the test and are present in each of the variants.

TABLE 4

Variants of bolt tests in the HE test

	Material	Stress	Environment	Surface defect
Variant 1	+	+	+	+
Variant 2	+	+	–	+
Variant 3	+	+	+	–
Variant 4	+	+	–	–

The duration of the test was designed to be 100 hours. During the test, readings were taken for the potential value. The assembled bolt sets were completely immersed in the solution. For the most optimal test conditions, one test stand was used for one bolt set.

3. Procedure

The test sets were removed from the solution after 100 hours. The evaluation of the samples was carried out according to the following steps:

A. Evaluation of the prestressing of the assembly, whether the stiffness of the connection has been maintained. The evaluation was based on an attempt to disassemble the connection with the hand. If the bolt tears during the test, the clamping force would be deducted. Such a situation would lead to a reduction in the loosening torque and the possibility of easy bolt disassembly.
B. Visual inspection of the surface discontinuity in the transition radius area and the thread were inspected. The bolts were initially evaluated with the zinc coating applied earlier. Subsequently, the coating was removed, and the macroscopic evaluation of the surface was performed.
C. Microscopic evaluation of the longitudinal run of the sample in the places specified in point B. The polished sample was prepared taking into account the performance of the wedge. The cutting was made through the longitudinal section where the greatest tensile stresses occurred in the bolt. Phenol-formaldehyde resin was used to prepare the metallographic specimen. The samples were polished, the last polishing was done on a 3 μm pad. The evaluation of the samples was carried out at a magnification of 500×.
D. For samples that do not meet the joint stiffness (no prestress), visual assessment, in the next step, making a breakthrough and assessing the scrap fracture. The breakthrough was made by locking the bolt in a vice, grasping the end of the bolt thread. Then, by a hammer it was hit against the bolt head (flange) from the side of the impact of the wedge on the stresses.

4. Analysis

The assessment of the tested samples was divided according to the variants from TABLE 4.

4.1. First tested variant

In this variant, in addition to the three factors mentioned, for the occurrence of HE, the parameter of the lap forging defect on the bolt bearing surface was added. The results obtained in the tests gave inconsistent results for one of the three bolts in step A of the assessment. The nut was removed from the bolt by hand, there was no tension that indicated deformation of the bolt, elongation of the loaded part due to fracture. The other two research sets were disassembled using a torque wrench. Three pieces were initially visually inspected, then the zinc coating was removed with hydrochloric acid and a full macroscopic analysis of the surface discontinuity was performed at 4 times magnification. Parameters were assessed according to the ISO 6157-3 standard [20], with particular emphasis on the areas most exposed to cracks. The bolt threads, the first thread (thread starting from the bolt pin with a larger radius at the bottom than the rest of the thread) and the radius between the bearing surface and the pin were checked. In samples 1 and 3, no additional cracks were found, apart from the lap forging defects generated intentionally in the forging process (Fig. 6). Longitudinal sections of the bolts for metallographic evaluation were prepared. The analysis showed no new crack nuclei. The lap forging length of the bolt was measured and compared with the samples immediately after the forging process (Fig. 7). The measurement was to show whether the lap forging defect propagated, the end of which can be defined as a notch during the HE test. There was no significant increase in the length of the defect. The difference obtained in favor of the samples after the test could have resulted from chance and depended on the variability of the forging process, high strain rates, tool heating, and production speed reaching 180 pcs/min. In the future, for the remaining samples prepared for HE tests, it is planned to extend the test to 200 hours to assess the potential increase in the length of the lap forging defect due to the diffusion action of hydrogen atoms.

A breakthrough was made in sample 2 of variant one (Fig. 8). Macroscopic and microscopic analysis allowed us to classify the type of scrap as brittle. The crack after evaluating the polished sample of the bolt thread was defined as an intercrystalline crack. There are no places where plastic scrap occurs, this is due to the fact that the tension in the bolt dropped after the crack had occurred. The clamping force was reduced by increasing the clamping length on the bolt due to a break.

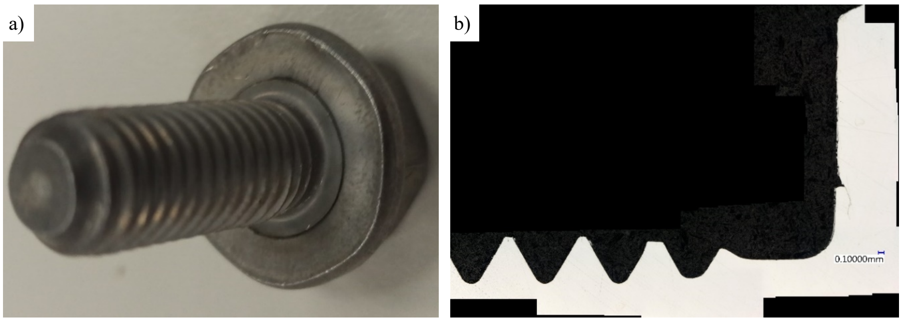


Fig. 6. Evaluation of sample 1 of variant 1 after the test. a) assessment of surface discontinuities, b) assessment of the bolt longitudinal section

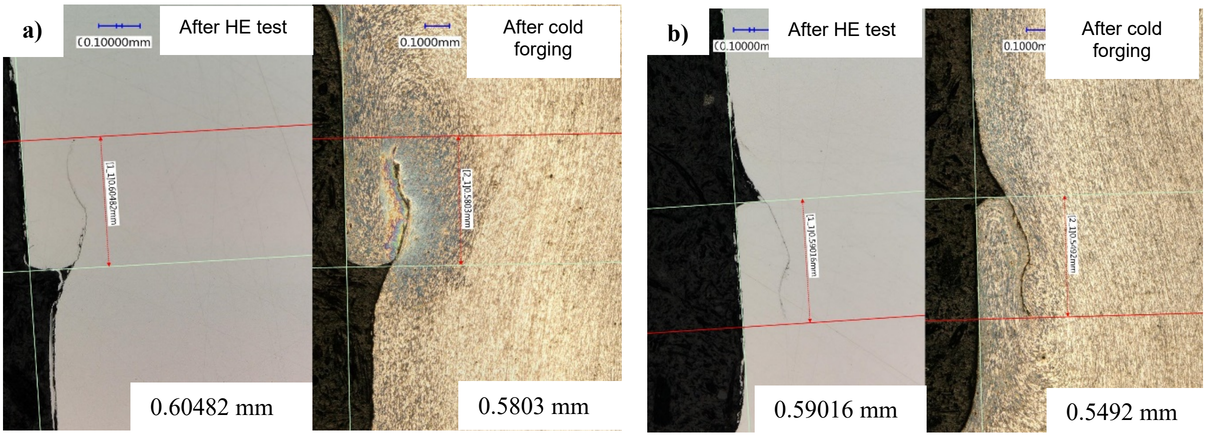


Fig. 7. List of the clamping length for the bolt after the HE test and after the forging process (before heat treatment), a) right side of the bolt, b) left side of the bolt

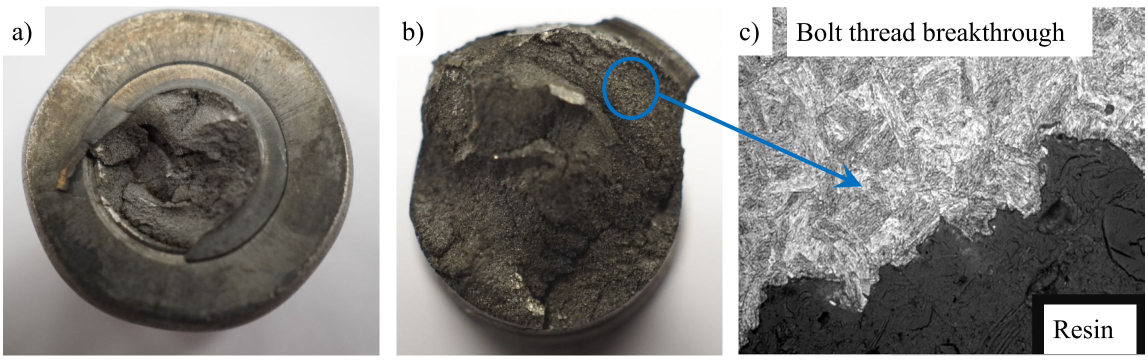


Fig. 8. Assessment of breakthrough of the bolt after failure, a) bolt head, b) threaded bolt pin, c) cross cut from the threaded bolt pin

4.2. The second and fourth tested variants

Variants without environmental impact were tested with positive results. Both the A, B and C tests did not indicate any defects. The mere load of high-strength bolts for 100 hours, despite the stress concentration with the use of a wedge, did not cause cracking. The environment in which the test was carried out was characterized by a humidity not exceeding 60%, at a temperature of 20 to 25°C. For this purpose, the laboratory room was used to assemble the sets and store them during the test. The room is constantly monitored and it was possible to maintain constant test conditions. It should be noted here that such restrictive test conditions are far from the actual bolt operation conditions. Threaded fasteners are used in virtually all industries and in various environments. You can also take into account the long-term impact of the environment and the exposure of the bolts to its negative impact. Likewise, the environment in which the bolts are used can change and the conditions in the hall, installation, or outdoors can deteriorate.

4.3. Third tested variant

The verification of bolts manufactured in accordance with factory quality standards under hydrogen diffusion conditions was to answer whether there was a reasonable risk of bolt failure or nucleation of a crack. The A and B tests for three samples gave a positive result. There was no visible effect of hydrogen embrittlement on the surface of the product. Test C allowed the location of the nucleus of the fracture on the side of the bolt exposed to the wedge, of increased tensile stress. The nucleus of the crack was located in the radius between the bearing surface and the bolt pin. The stress point in the loaded bolt. The first microscopic assessment did not give a clear answer as to whether the image shows a crack germ, there was a risk of confusion with a defect in the sample polishing process. The sample was polished again, which confirmed the presence of a defect in the same place. The length of the crack nucleus (Fig. 9b) is 0.03336 mm, it was created as a result of a notch on the surface. The notch could have resulted from the random sticking of semi-product material particles to the die.

5. Discussion

The results of individual tests with regard to the individual assessment steps are presented in the TABLE 5. Variant 1 for sample 2 shows ifluences HE at all assessment points. In turn, variant 3 in sample 1 shows the influence of HE on the score from point C, i.e. the microscopic assessment. The appearance of a crack nucleus in the notch area may result from the concentration of hydrogen atoms there. The obtained results were incompatible for the samples where the environment was created favoring the phenomenon of hydrogen embrittlement. Note that according to industry standard fasteners and ISO 6137-3, one nonconforming sample from a production batch indicates that the entire batch is non-compliant [20]. On this basis, it was assumed that variants 1 and 3 are not compatible.

In the case of variant 1, the appearance of a crack was already visible at the time of disassembly. This is the most consist-

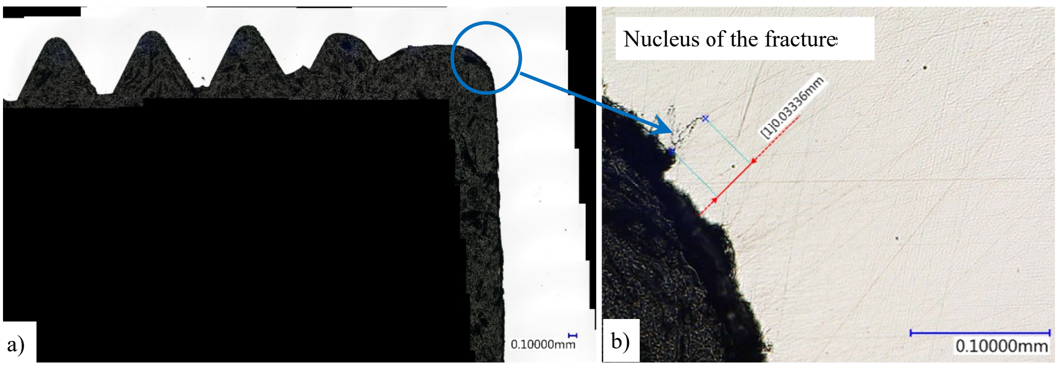


Fig. 9. Microscopic evaluation of a polished longitudinal sample, a) evaluation of surface discontinuities in places exposed to cracking, and b) crack nucleus on the radius

TABLE 5

Results of HE influence tests on high-strength bolts

Tests \ Samples	Variant 1			Variant 2			Variant 3			Variant 4		
	1	2	3	1	2	3	1	2	3	1	2	3
A	OK	**NOK**	OK	OK	OK	OK	OK	OK	OK	OK	OK	OK
B	OK	NOK	OK	OK	OK	OK	OK	OK	OK	OK	OK	OK
C	OK	X	OK	OK	OK	OK	**NOK**	OK	OK	OK	OK	OK
D	NA	NOK	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Results	**NOK**			OK			**NOK**			OK		
OK-Compliance, NOK-Not Compliance, NA-Not Applicable												

ent with the cases of sudden failures during the use of fasteners [15,16]. Variant 3 revealed the surface discontinuity only after making a sample of the longitudinal section of the bolt. The method of such assessment allows for a more precise verification of the appearance of the crack nuclei [18]. The conducted assessment took into account the areas with the highest stresses in the bolt connection, i.e. the radius between the head and pins, the first incomplete thread turn and the first loaded thread.

6. Conclusions

The method analyzed to assess the effect of hydrogen embrittlement on threaded fasteners for concrete applications allows the adaptation of the EAD test standard for use in the case of high-strength bolts. The main conclusions from the conducted experiments are as follows:

1. The developed method allows for the assessment of screw products in terms of the potential risk of hydrogen embrittlement and to indicate places where such failure may occur.
2. High-strength bolts in mechanical property class 12.9 subjected to the hydrogen embrittlement test showed susceptibility to this phenomenon.
3. Defects after the HE test appeared in the radius (between the head and the bolt pin), which confirms that it is one of the most exposed places to damage.
4. The cracking mechanism with hydrogen embrittlement is a random phenomenon and does not occur on all samples despite the same test conditions.
5. Product defects, i.e. material defects, defects in heat treatment, lap forging, folds, wrinkles, and notches favor cracking triggered by hydrogen embrittlement.
6. Testing bolt products for hydrogen embrittlement is a very sensitive test of product quality and should be widely used for high-strength fasteners with unusual shapes of heads and abutment surfaces, such as those used in automotive (dodecagon) and construction (anchors and bolts). Not only IHE should be taken into account, but also EHE (stress corrosion cracking, intercrystalline corrosion).
7. For hydrogen embrittlement (product failure), three conditions must be met (environment, stress, material).

The experiment carried out is the beginning of a wide series of studies on the evaluation of high-strength bolts in an environment that allows hydrogen diffusion. The developed test procedure allows to effectively verify the operation of the bolt joint under constant load conditions.

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