

## Article

# Effect of fire temperature and exposure time on high-strength steel bolts microstructure and residual mechanical properties

Paweł A. Król <sup>1,\*</sup> and Marcin Wachowski <sup>2</sup>

<sup>1</sup> Warsaw University of Technology, Faculty of Civil Engineering, Institute of Building Engineering, Division of Concrete and Metal Structures, 16 Armii Ludowej Ave., 00-637 Warsaw, Poland; pawel.krol@pw.edu.pl

<sup>2</sup> Military University of Technology, Faculty of Mechanical Engineering, 2 Gen. S. Kaliski St., 00-908 Warsaw, Poland; marcin.wachowski@wat.edu.pl

\* Correspondence: pawel.krol@pw.edu.pl; Tel.: +48222346648

**Abstract:** The article presents results of research consisting in an attempt to assess the influence of temperature, heating time and cooling method on microstructure and residual strength properties of steel previously tempered during the production process. Simulated environmental conditions to which high-strength bolts, commonly used in steel construction, were subjected, were intended to reflect conditions of a natural fire that may occur in public facilities where the obligation to ensure safety of users and rescue teams is of key importance, also required by law. Furthermore, the tests carried out also comprised a simulated effect of a rescue and firefighting operation using shock, rapid cooling of some of the heated bolts. Samples cut out from the tested bolts, after they were properly prepared, were subjected to microstructural tests using light microscopy, scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), phase analysis with the use of an X-ray diffractometer (XRD) and quantitative analysis of the microstructure involving, inter alia, measuring the surface area of grains, their equivalent diameter and mean diameter. As a result of the tests, considerable microstructure changes were identified occurring in the bolt material as a result of exposing it to fire conditions, leading to a change in key, from the point of view of structural safety, mechanical properties. The results of the microstructure tests were compared with the results of previous strength tests, including hardness of the material after the heat treatment and the residual tensile strength of the material  $R_m$ . A conducted comparative analysis showed a significant effect of all such factors as the temperature level of the simulated fire, its duration and the fire-fighting method on the mechanical properties of bolts. Results obtained were provided with required comments and the concept of using the microstructure for the post-fire assessment of steel structures was referred to.

**Keywords:** effect of temperature; exposure time; steel microstructure; residual mechanical properties; high-strength steel bolts; heat treatment of steel; phase transformation; fire; cooling method

## 1. Introduction

The behaviour of steel structures during and after fire has stimulated the imagination of researchers and engineers for decades. Reflections derived from the observation of real fires in steel-structure buildings lead to the conclusion that disasters and collapses of these buildings most often happen not during the fire flashover, but during the fire extinction phase, when structural elements previously subjected to sufficiently long fire exposure lose their stability or shorten, thus generating additional tensile forces that were not previously present. This thesis can be best confirmed by the example of the WTC buildings that collapsed in consequence of a fire that developed in them as a result of ignition of aviation fuel. In such circumstances, internal forces are redistributed or connections between structural components are overloaded. At the same

time, provided that the fire temperature has reached a sufficiently high level, microstructural changes can be seen in the structural material – both in load-bearing members and mechanical connectors. Observation of these structural destruction mechanisms allowed an assumption that high-strength bolts, which were subjected to heat treatment at the production process stage, lose their strength and load-bearing properties much faster than members made of conventional steel. Due to the history of previous heat treatment, they are more sensitive to temperature and thus become the weakest element of load-bearing structures, determining safety of steel constructions.

Brian Kirby is one of precursors of modern research on post-fire behaviour and residual mechanical properties of bolts. In his works [1, 2], he presented results of research carried out on M20-8.8 bolts made in various production processes – using hot and cold forging. He subjected the bolts to pre-heating in the temperature range of 20-800°C, maintained in a given temperature level for 60 minutes, and then naturally cooled them in the air. He observed that hot-forged bolts are more sensitive to temperature changes than cold-forged ones. As a result of his research, he proposed using crystallographic methods to develop a method enabling identification of the maximum temperature that bolts could reach during a fire. He observed that post-fire residual strength of steel can be determined, *inter alia*, by specifying its hardness on a polished surface using the Vickers hardness tests with a load of 30 kgf/294.2 N (HV30) and comparing results obtained with those of a static tensile test, bearing in mind that there is some orderly relationship between the hardness of steel and its tensile strength. He pointed out that if bolt production process parameters are known, this knowledge can be used to assess the temperature reached by a bolt during a fire by comparing its so-called residual hardness with hardness of the material of the bolt in its initial state. At the same time, he demonstrated that if the fire temperature level exceeds the tempering temperature in the production process, the bolt material softens. He showed that knowledge of metallurgical changes occurring in the bolt material can be helpful in diagnosing fire-damaged buildings.

The behaviour of bolts during a fire was also analysed, among others, by Gonzalez et al. [3] who focused their research on tensile tests of grade 10.9 bolts. Tests limited to destructive testing (static tensile test and static shear test) reflecting the behaviour of bolts under natural fire conditions (comprising the cooling phase) were also carried out by Hanus et al. [4]. They confirmed that if the structure is not destroyed during the temperature elevation phase, tensile forces generated in axially-restrained beams during the cooling phase may lead to failure of bolted joints. Unfortunately, they did not conduct any research related to the analysis of material structure changes.

A number of works by Kodur et al. [5, 6] are also worth paying greater attention to. In [5], the authors analyse the effect of temperature on variability of thermal and mechanical properties of high-strength steel bolts. They look on bolt failure mechanisms occurring during the tensile test through the prism of bolt microstructure, having effect on the material ductility and the failure model, as well as the shape of the fracture surface after failure. In [6], apart from routine destructive testing aimed at assessment of residual mechanical properties of grade 8.8 bolts subjected to heating and controlled cooling cycles, they devoted more attention to the issues of crack propagation and bolt failure models, conducting a broader analysis of fracture surface shapes obtained in the context of the target heating temperature level. They made an attempt to explain how cracks propagate depending on microstructure of the bolt material. In [7], Yahyai et al. undertook a similar research as in [6], focusing on bolts with a higher strength grade – 10.9. The effects of a heating temperature, a chemical composition of the charge steel (raw material) and production process parameters on durable mechanical properties were subject to an in-depth analysis. A considerable part of the work was devoted to a detailed analysis of surface of the bolt failure, explaining the shape and form of fractures, and changes occurring in steel microstructure. There can be a couple more similar works found that present results of destructive tests and focus almost exclusively on assessing how strength parameters of bolts exposed to fire temperatures decrease.

Analysis of literature corresponding to the subject matter of this work allows clear distinguishing of two types/groups of bibliographic sources. The first group includes works focusing strictly on the load capacity and strength parameters of bolts and connections, and their authors are usually professionally and scientifically related to the construction industry. The second group – generally referring to steel as one of construction materials – comprises works of people educated in the field of mechanical engineering, fire safety engineering and broadly understood materials engineering. All the works referred to earlier can, in principle, be classified to the first group. Some more interesting works from the borderline of material engineering and fire safety engineering, and related to some extent to the issues discussed in this article, include the paper by Chi & Peng [8], in which the authors present results of steel plate tests, heated to a temperature of 800°C and higher, and then quickly cooled in water. The work was aimed at demonstrating the possibility of reconstructing a fire scenario in a post-fire investigation on the basis of changes in the material microstructure, in a situation where fire development and a level of the temperature reached in a fire were not known. The plates underwent, inter alia, metallographic tests to analyse their composition and microstructure, and results obtained were compared to mechanical parameters of tested members destroyed by fire. The research enabled observation that as a result of the heat treatment, the perlite phase disappeared completely, ferrite was reduced from 80% to 30%, bainite increased its share to 30%, and martensite increased to 40%. A significant increase observed in the martensite phase changed the structure of the steel plate damaged by fire, which was reflected in the change of its material properties. Although yield point and tensile strength showed a growing trend, ductility of the member dropped significantly – from 32.5% to 15%, which may result in a greater likelihood of sudden steel failure in structural elements of a building and translate into a decreased safety of its use. Brittleness of members increases the risk of an abrupt, progressive collapse, caused by inability of the structure to redistribute internal forces by creating local yielding areas. This, in turn, may result in an increase in a threat to lives and health of users of the building – potential victims of a fire and members of rescue teams. The authors draw attention to the importance of this problem, especially in the case of facilities such as shopping centres, galleries, arcades or other public facilities made of steel structures.

Analyses of the effect of microstructure on mechanical properties of post-fire structural steels were also undertaken by Sajid et al. [9]. As part of the research, they analysed samples made of three grades of structural steel, subjected to heating in the temperature range from 500°C to 1000°C for 60 minutes, and then naturally cooled in the air. They presented changes in the microstructure in relation to the heating temperature. The analyses they conducted led to a conclusion that an increase in the share of ferrite fraction and the ferrite grain size leads to a decrease in the residual post-fire yield point and tensile strength, as well as an increase in the ductility of the tested structural steel grades. Based on the obtained results, the authors proposed multivariate linear regression equations to estimate post-fire/residual yield point of steel as a function of ferrite grain size and perlite colony size. In their opinion, results of these tests may be useful for future engineers and can be used to assess the quality and strength parameters of post-fire steel, basing solely on the results of microstructural tests, especially in a situation when information on the temperature level reached in a fire is not available.

Works by Haiko et al. [10] and Xie et al. [11] are also worth mentioning, however since they do not relate directly to the issues addressed in this article, they will not be broadly discussed. None of the cited works directly referred to the fact that post-fire properties of the bolt material heat-treated during the production process noticeably differ from properties of typical structural steels, whose strength parameters result from a suitable selection of alloying elements. The difference results precisely from the history of heat treatment, therefore applying the relations describing mechanical properties of carbon steels, available in design standards, e.g. [12-13], to the assessment of load capacity of bolted connections, may result in dangerous estimates.

This article presents results of research on the effect of various thermal and environmental conditions, typical for a real fire situation, on changes in mechanical properties of high-strength construction bolts, resulting from their microstructural transformation. M20-8.8 bolts were used for the research, as they are commonly used in prestressed butt joints and friction lap joints, as well as more and more commonly – due to their universal properties – used in regular non-prestressed joints. During the preliminary phase of testing, the bolts were subjected to thermal effects corresponding to selected conditions of a simulated fire, by heating them in batches in an electric furnace for the time specified in [14], corresponding to fire safety requirements adopted within the EU, established by law in relation to structural elements of buildings and building structures. Wording of national legal acts [14-15] directly implements provisions of Regulation (EU) of the European Parliament and of the Council of 9 March 2011 laying down harmonised conditions for the marketing of construction products. Their purpose is to ensure that basic requirements, such as required load-bearing capacity and structural stability, fire safety, health and safety of use, are met. Following the exposure to high temperature, some bolts were let aside to cool down naturally. The intention was to recreate conditions of a spontaneous, natural end of a fire, resulting either from a shortage of combustible substances or an insufficient amount of oxygen. The other part of the bolts was shock-cooled by immersion in water, which corresponded to a simulated firefighting operation carried out by rescue and firefighting teams.

A comprehensive approach to the analysis of the effect of simulated natural fire conditions on microstructural changes in the material of construction bolts and, consequently, also on their key strength properties, taking into account the effect of thermal conditions, exposure time and cooling method, which is presented in the paper, cannot be found in available literature. In this context, this work brings a completely new value to the state of knowledge in this area and may really contribute to the progress of work on methods of post-fire assessment of quality and reliability of structures. The results obtained can be helpful both for the purpose of assessing structures that have survived a fire without any major damage – in the context of the possibility of reusing selected elements, as well as those that have been damaged by fire. In the latter case, the results can be used to recreate a fire development scenario and to estimate maximum values of fire temperatures in a situation where they have not been measured by firefighting and rescue services.

2. Materials and Methods

Samples for microstructural tests were cut from a series of bolts previously subjected to fire exposure in various thermal conditions and – alternatively – also a simulated firefighting operation. The bolts were subjected to heating/tempering processes at various temperatures (400°C, 600°C, 800°C and 1000°C). In the case of the first series of samples, cooling was carried out naturally in the air (air-cooling/sample symbol: AC), letting them to cool down slowly, while in the case of the second series – in an accelerated way, by immersion in water (water-cooling/sample symbol: WC). In each of the cases, two different heating times were applied – respectively 60 minutes and 240 minutes, corresponding to the selected requirements resulting from [14]. A list of samples is presented in Table 1. Apart from the reference sample in the initial state (IS), the samples were labelled according to the X/Y/Z principle, where X shows the cooling method, Y – the heating temperature and Z – the heating time at a given temperature. Microstructural tests and hardness measurements were carried out using samples cut perpendicularly to the bolt shank axis, the area of which corresponded to the shank cross-section in the threadless place.

Table 1. List of tested samples.

Labels of samples prepared for testing
IS – reference sample
AC/400/60

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AC/400/240
AC/600/60
AC/600/240
AC/800/60
AC/800/240
AC/1000/60
AC/1000/240
WC/400/60
WC/400/240
WC/600/60
WC/600/240
WC/800/60
WC/800/240
WC/1000/60
WC/1000/240

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The bolts were made of an alloyed steel with an addition of boron, designated as 32CrB3, whose chemical composition is provided according to the manufacturer's quality certificate in Table 2.

**Table 2.** Chemical composition of the 32CrB3 bolt steel, according to the manufacturer's quality certificate.

Steel designation	Chemical composition [%]										
32CrB3	C	Mn	Si	P	S	Cr	Ni	Cu	Al	Mo	Sn
	0.31	0.84	0.13	0.012	0.013	0.74	0.08	0.15	0.025	0.018	0.010

In the production process, the bolts were made of smooth wire rod in the hot-forging process and then subjected to thermal improvement by quenching at a temperature of approx. 850-860°C and tempering at a temperature of approx. 550°C, which resulted in obtaining of expected mechanical properties, corresponding to grade 8.8.

Cut out samples for microstructural testing were hot-mounted in phenolic resin with Struers Multifast graphite filler and then ground using grinding wheels of 320, 600, 800 and 1200 gradation. The samples prepared in this way were polished using a diamond suspension with a grain diameter of 3 and 1 µm. Owing to such processing method adopted, high polishing accuracy was achieved and, at the same time, thermal effects on the top layer of the analysed surface, occurring during cutting, were eliminated. The microstructure of the steel was revealed by etching with a 2% solution of nitric acid in ethanol (2% NITAL).

In order to illustrate the microstructural changes resulting, inter alia, from phase transformations, the research material was analysed using the OLYMPUS LEXT OLS4100 digital light microscope and the JOEL scanning electron microscope (SEM), model JSM-6610, equipped with a secondary electron detector (SE) and a backscattered electron detector.

The light microscopy tests were performed on the sample series shown in Table 3.

**Table 3.** List of samples tested with the use of light microscopy.

Labels of samples tested with the use of light microscopy
IS – reference sample
AC/600/60
AC/600/240
AC/800/60
AC/800/240
AC/1000/60

AC/1000/240
WC/400/60
WC/400/240
WC/600/60
WC/600/240
WC/800/60
WC/800/240
WC/1000/60
WC/1000/240

At the stage of microstructural tests, samples heated at the temperature of 400°C were eliminated, since the results of the static tensile test and the hardness test did not reveal any noticeable changes in relation to the initial state IS, which can be explained by the fact that this temperature is significantly lower than the tempering temperature used during the production process and the phase transformation temperature of steel, A<sub>1</sub>. In the case of each of the samples, photos were taken at several points along the shank width, corresponding to their distance from the cross-sectional edge, representing 1, 3, 5, 7 and 9 mm, respectively. For each of the points, two photos were taken at different magnifications, x50 and x100, respectively, to obtain a more complete picture of changes occurring in the material microstructure. A scanning electron microscope (SEM) was used to test the initial state sample, with an accelerating voltage of 20 kV. The microscopic observations were supplemented with surface microanalysis of the chemical composition by means of the Oxford X-Max energy dispersive X-ray spectrometer (EDS), the results of which were generated in the form of an X-ray spectrum. In order to demonstrate the presence of carbides in the material structure, phase analysis was performed with the use of X-ray diffraction (XRD).

Images of the bolt microstructure in the form of photos obtained with the use of a digital light microscope at the magnification of x100 were used for a quantitative analysis. Photos taken at a distance of 3 and 7 mm from sample edges were selected for this purpose in order to capture differences, if any, in the microstructure along the width of the bolt shank. These images were used to outline grains of two basic phases, namely ferrite and perlite, observed in the material microstructure. The quantitative analysis was performed using the MountainMap program. For each of the phases such parameters as the number of grains visible in the photo, their density, grain surface or the mean equivalent grain diameter were determined. Types of samples for which this type of analysis was performed are presented in Table 4. Considering the size of this article, results of the quantitative analysis were not presented in detail and only some of them were shown in Table 13 and Fig.3, included further in the work.

**Table 4.** List of samples tested for the purpose of the quantitative analysis.

Labels of tested samples
AC/800/60
AC/800/240
AC/1000/240
WC/800/60

The Vickers HV hardness tests were carried out with a load of 30 kgf (294.2 N) in the same places along the shank width and compared with the breaking strength values obtained from the previously performed static tensile test, in order to confirm the correlation relationship between hardness HV and tensile strength R<sub>m</sub>. For structural steels, it is presented in various literature sources in the form of the following relationship:

$$R_m \text{ [MPa]} \approx (3.2\div3.5)\cdot HV \tag{1}$$

In order to eliminate a so-called human error, the hardness tests were carried out in an automated manner using the NEXUS 4300 stationary hardness tester in several places along the width of the bolt shank, in order to capture any differences. Due to the lack of differentiation of the measurement results across the width of the sample, the mean value was taken as representative for further considerations.

3. Results

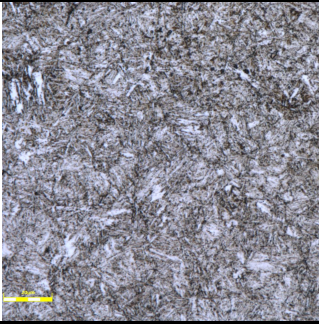
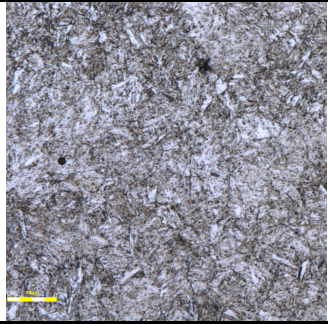
3.1. Microstructural analysis

The analysis was aimed at investigating and assessing the effect of the secondary heat treatment resulting from exposure to various thermal conditions that may occur during a fire and the accompanying firefighting operation, on the microstructure and mechanical properties of bolt steel, previously quenched and tempered during production processes.

The material in the initial state IS is characterised by a martensite structure ( $\alpha$  phase – dark phase/areas in the photos of the IS sample), with a small quantity of residual austenite ( $\gamma$  phase – light areas in the photos of the IS sample), Table 5.

Analysis of the photos included in Table 5 does not show any significant differences between the microstructure image seen in the photo taken at a distance of 3 and 7 mm, respectively, from the shank edge. The presence of the martensite structure is related to the quenching process previously carried out, typical of steel bolt products, in particular those with increased strength. The martensite structure occurs throughout the cross-section of the tested element – along the entire width of the bolt shank.

Table 5. Microstructure of steel in its initial state.

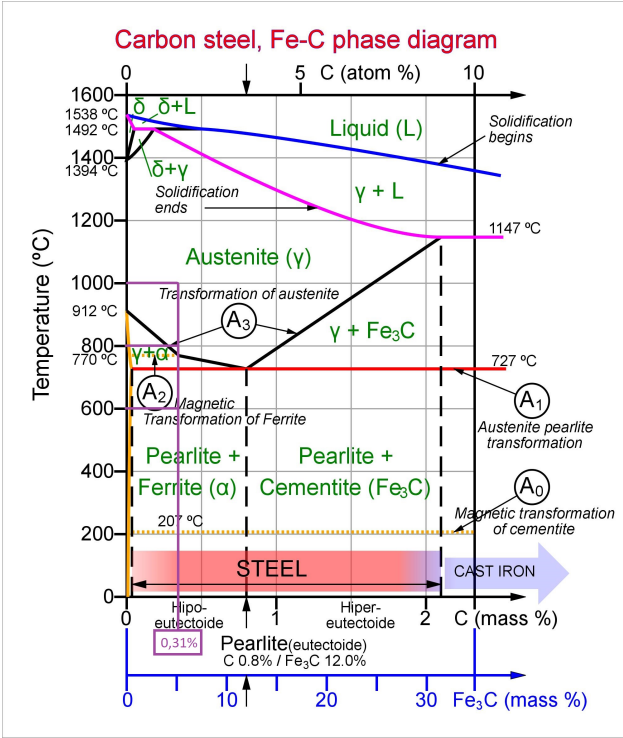
Sample label	Image at a distance of 3 mm from the sample edge (scale mark 20 $\mu$ m)	Image at a distance of 7 mm from the sample edge (scale mark 20 $\mu$ m)
IS – initial state (reference sample)		

Due to the 0.3% carbon content, the steel covered by the research is referred to in the literature as hypoeutectoid steel and in its unquenched state, it is characterised by a ferrite and perlite structure. Heat treatment of steel in the quenched state is called tempering and consists in heating up of steel to a temperature below  $A_1=727^{\circ}\text{C}$  – read out from the Fe-C phase diagram (Fig.1), leaving it at this temperature and slowly cooling to ambient temperature. Depending on the heating and cooling rate as well as the amount and type of alloy elements, the critical temperatures shown in Fig.1. may somewhat vary. The purple line in Fig.1. marks the carbon content in 32CrB3 steel (0.31%). Heat treatment below temperature  $A_1$  does not lead to the formation of austenite ( $\gamma$  phase), whereas annealing above  $A_1$  takes place when the ferrite ( $\alpha$  phase) and austenite ( $\gamma$  phase) states coexist. Processes that occur during tempering are closely related to the phenomenon of diffusion of carbon and alloying elements, therefore they depend on both the temperature level and the heat treatment time. The purpose of the annealing process is to obtain a more fine-grained and more plastic structure, which is desirable in the context of a more predictable behaviour and a non-brittle model of structural failure. Fine graining leads to an increase in the yield point, as a denser mesh of grain boundaries is shaped in

the structure of the material, which may slow down the formation of dislocations in the steel crystal lattice. According to the Hall-Petch formula (2), [17], the yield point is proportional to the square root of the mean grain size, which confirms the previously formulated thesis:

$$f_y \sim 1/(d)^{0.5} \tag{2}$$

As was also confirmed by authors' own observations, the value of the yield point remains practically unchanged in the temperature range up to approx.  $600 \pm 50^\circ\text{C}$ , while in higher temperatures, grains start to grow, which reduces the density of mesh of grain boundaries, thus leading to a reduced yield of steel.

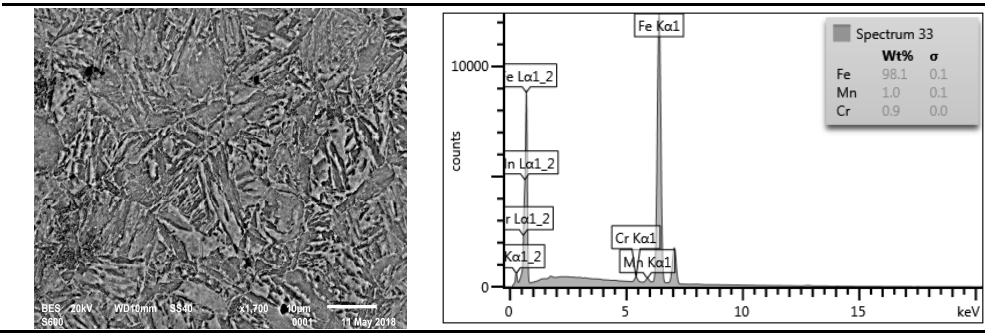


**Figure 1.** Carbon steel, Fe-C phase diagram  
[source: [https://commons.wikimedia.org/wiki/File:Steel\\_Fe-C\\_phase\\_diagram-en.png](https://commons.wikimedia.org/wiki/File:Steel_Fe-C_phase_diagram-en.png) - freely reused under the permission of Wikimedia.org open license; access: 22-04-2021]

In order to confirm a chemical composition of steel (Table 2), an EDS analysis was performed using a scanning electron microscope. The analysis showed the presence of the following alloy elements: carbon, iron, manganese and chromium. The content of manganese (1.0 wt.%) and chromium (0.9 wt.%), Table 6, turned out to be slightly higher than the content specified in the metallurgical certificate (Table 2).

**Table 6.** Microstructure of steel in its initial state, along with the results of the EDS analysis

Microstructure of steel in the initial state	Results of the EDS analysis
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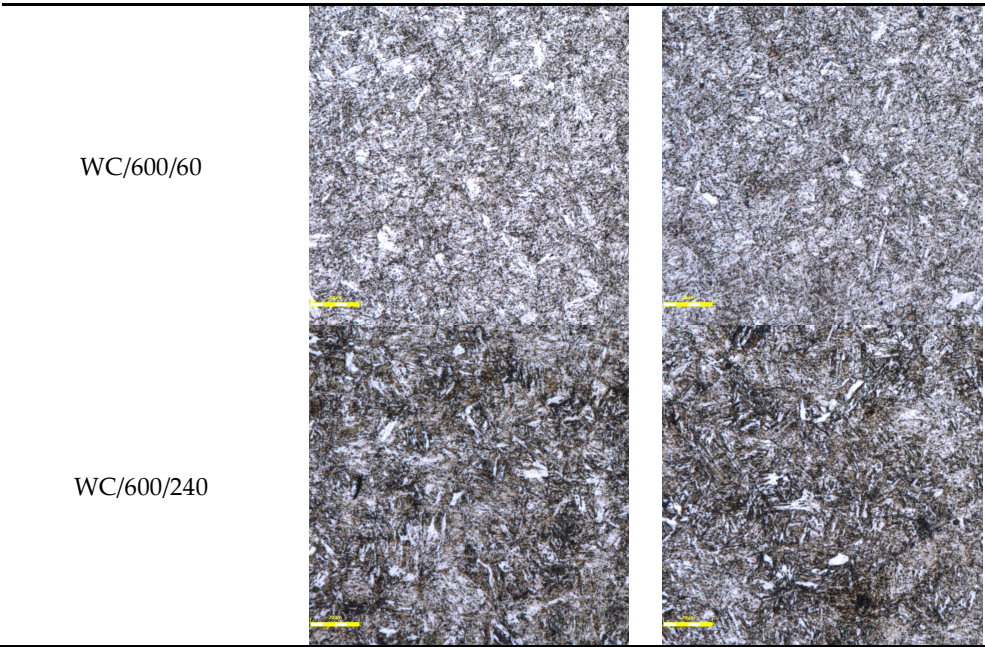


3.1.1. Microstructural analysis of samples heated at 600°C

Tempering at a temperature of 600°C with both air- and water-cooling does not noticeably change the microstructure of steel. This temperature, although it exceeds the nominal tempering temperature used during the production process of this grade of bolts, turned out to be still too low to initiate the phase transformation, consisting in the decomposition of martensite and the release of austenite, Fig.1 and Table 7. Such an excess in comparison to the nominal tempering temperature is probably the result of presence of alloying elements that may cause certain disturbances in technological parameters in the case of repeated thermal treatment.

**Table 7.** Microstructure of steel after heating at 600°C for 60 and 240 minutes with air- and water-cooling.

Sample label	Image at a distance of 3 mm from the sample edge (scale mark 20 μm)	Image at a distance of 7 mm from the sample edge (scale mark 20 μm)
AC/600/60		
AC/600/240		



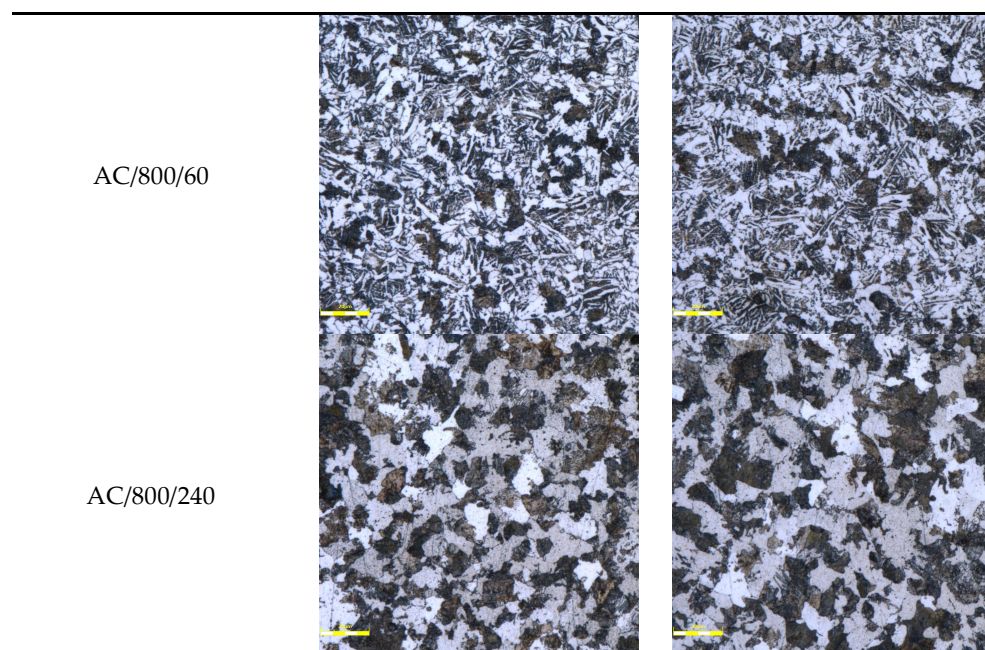
Previously carried out strength destructive tests showed that heating at this temperature did not lead to a significant reduction of residual strength properties, such as e.g. breaking or shear capacity, especially in the case of the heating time shorter than 60 minutes. The analysis of the photos presented in Table 7 does not reveal any significant differences in the microstructure, neither along the width of the shank, nor between the samples with different heating time or cooling method. Although there are no visual differences in the microstructure between the IS, AC/600 and WC/600 samples, both in the case of the air-cooled and water-cooled bolts, the value of residual tensile strength after heating for 60 minutes turned out to be lower than the initial value of the IS bolts by approx. 12%, and in the case of bolts heated for 240 minutes – by as much as 22%. These tests showed the effect of the heating time on the value of residual strength properties of fire-exposed bolts.

3.1.2. Microstructural analysis of samples heated at 800°C

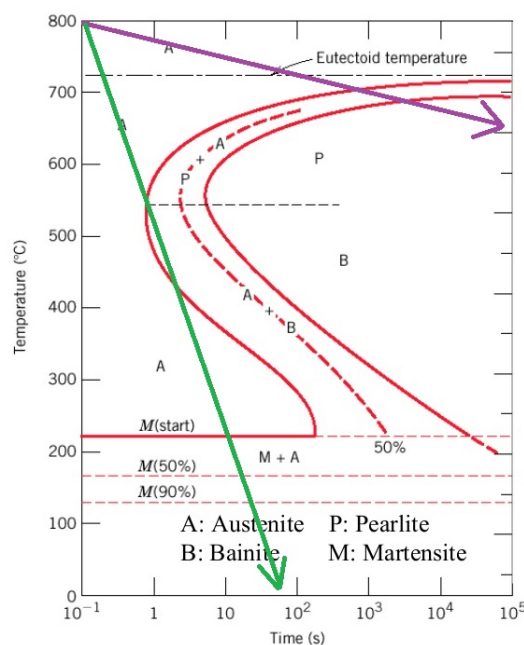
Tempering at the temperature of 800°C with air cooling (AC/800/60 and AC/800/240 samples) leads to the martensite → austenite phase transformation. Then, during the cooling phase, ferrite is released (light areas) at the boundaries of austenite grains (Table 8). The temperature of 800°C is above the A<sub>1</sub> line on the Fe-C phase diagram, therefore martensite austenitizes at this temperature (Fig.1).

**Table 8.** Microstructure of steel after heating at 800°C for 60 and 240 minutes with air-cooling.

Sample label	Image at a distance of 3 mm from the sample edge (scale mark 20 μm)	Image at a distance of 7 mm from the sample edge (scale mark 20 μm)
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The austenite transformation during tempering of alloy steels is influenced by the content of elements dissolved during austenitization. Chromium, being an alloying element of the tested steel, strongly increases its transformation temperature, as a result of which the amount of ferrite released during the heating at 800°C is small, but it increases along with growing temperature and heating time. When analysing the photos of the microstructure shown in Table 8, it can be easily noticed that the microstructure of the steel heated at 800°C for 240 minutes is significantly different from that heated for 60 minutes only. The grain size increases, the mesh of boundaries between respective phases loosens, which results in a further reduction of the residual tensile strength and hardness of the material, which becomes more plastic. Chromium, present in the tested steel as one of the alloying elements in the range up to 1%, slightly reduces the hardness of ferrite and considerably increases its impact strength, with a simultaneous decrease in hardness as compared to the reference value at room temperature (HV = 324). Moreover, it increases the amount of plastic residual austenite in the quenching process. Since the concentration of carbon in ferrite is lower than in the austenite from which it is released, the carbon content in austenite increases along with heating. After reaching the critical value, i.e. after exceeding the limit level of the solubility of carbon in austenite, the perlite transformation begins, leading to the transformation of the remaining austenite into perlite (dark areas), which results in obtaining a plastic, soft ferrite-perlite structure. The phase transformation is presented in the TTT-Diagram shown in Fig.2 (purple line).



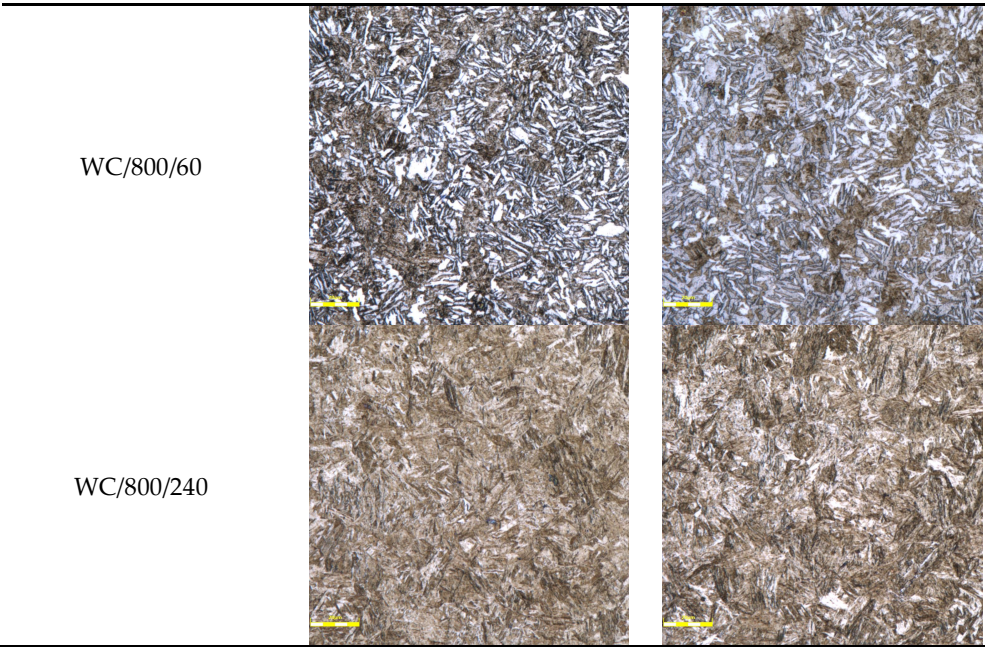
**Figure 2.** TTT-Diagram with marked phase transformation for the temperature of 800°C, with air cooling (purple line) and water cooling (green line) [source of a TTT-Diagram: <https://slideplayer.com/slide/13534682/>; access: 22-04-2021]

The transformation into ferrite-perlite structure is demonstrated by a reduction in the tensile strength ( $R_m$ ) from the initial state value equal to 1001 MPa (breaking force  $F_m=245$  kN) to the level of  $R_m=595$  MPa (maximum tensile force  $F_m=146$  kN), with HV=200 – for the heating time of 60 minutes and  $R_m=585$  MPa (maximum tensile force  $F_m=143$  kN), with HV=196 – for the heating time of 240 minutes. It is worth noting here that in the case of samples heated at the temperature of 800°C and air-cooled, despite the noticeably different microstructure (Table 8), the heating time does not have a significant effect on the differences in the value of residual tensile strength – in both cases it remains on an almost identical level. In the case of a longer heating time, recrystallization of the defective ferritic matrix was observed, which translated into a slightly greater decrease in the  $R_m$  value.

Tempering at the temperature of 800°C combined with subsequent water-cooling (WC/800/60 and WC/800/240 samples) did not result in the martensite  $\rightarrow$  ferrite + perlite phase transformation, despite reaching the heating temperature above  $A_1$ . As can be seen in the photos of the microstructure presented in Table 9, the material in this case still shows a typical martensite structure, similar to the original one in the IS state. The cooling method applied, characterised by a high speed of thermal energy reception, had a significant impact on inhibition of the phase transformation.

**Table 9.** Microstructure of steel after heating at 800°C for 60 and 240 minutes with water cooling.

Sample label	Image at a distance of 3 mm from the sample edge (scale mark 20 $\mu$ m)	Image at a distance of 7 mm from the sample edge (scale mark 20 $\mu$ m)
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Rapid water cooling of the steel heated up to the austenization temperature made it harden again (Fig.2 – green line). The degree of hardening turned out to be clearly dependent on the heating time. The martensite transformation did not fully take place within 60 minutes (residual austenite is still visible), which resulted in the value of  $R_m=1064$  MPa (maximum tensile force  $F_m=261$  kN – higher than the reference value for the sample in the initial state), with a simultaneous significant increase in hardness to  $HV=361$ . Heating for 240 minutes resulted in austenization of the whole microstructure and then the martensite transformation in the entire volume of the material, which translated into the value of  $R_m=1064$  MPa (maximum tensile force  $F_m=261$  kN – analogous as in the case of the heating time of 60 minutes), with another significant increase in hardness to the level of  $HV=543$ .

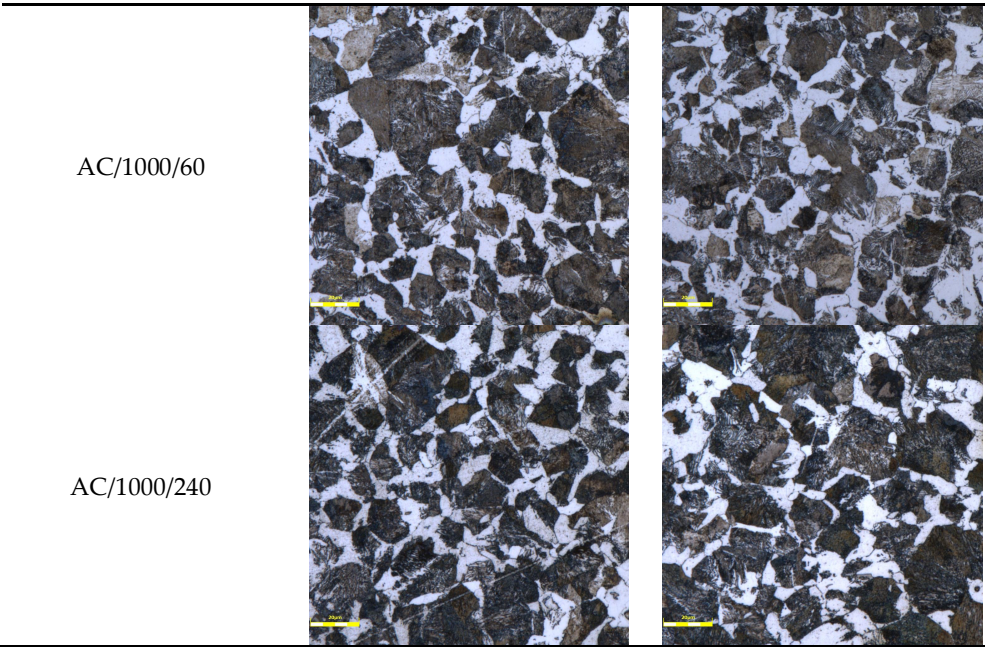
This stage of research showed that in the case of shock water-cooled samples, the duration of the heating time had a significant effect on increased hardness of the material and the fact of rapid cooling itself significantly influenced the value of residual tensile strength, which in this case exceeded the reference value obtained for bolts in the initial state IS ( $R_m=1001$  MPa).

3.1.3. Microstructural analysis of samples heated at 1000°C

Tempering at 1000°C with air cooling (AC/1000/60 and AC/1000/240 samples – Table 10), similarly to the heat treatment at 800°C, caused the martensite → austenite phase transformation during the heating process and then the release of ferrite and perlite within the boundaries of austenite grains in the phase of slow, free cooling.

**Table 10.** Microstructure of steel after heating at 1000°C for 60 and 240 minutes with air cooling.

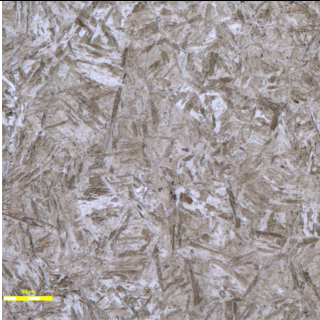
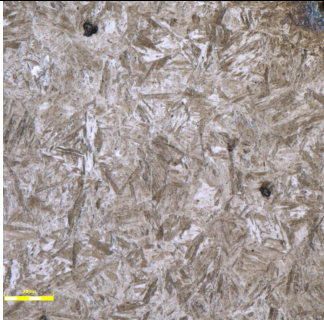
Sample label	Image at a distance of 3 mm from the sample edge (scale mark 20 μm)	Image at a distance of 7 mm from the sample edge (scale mark 20 μm)
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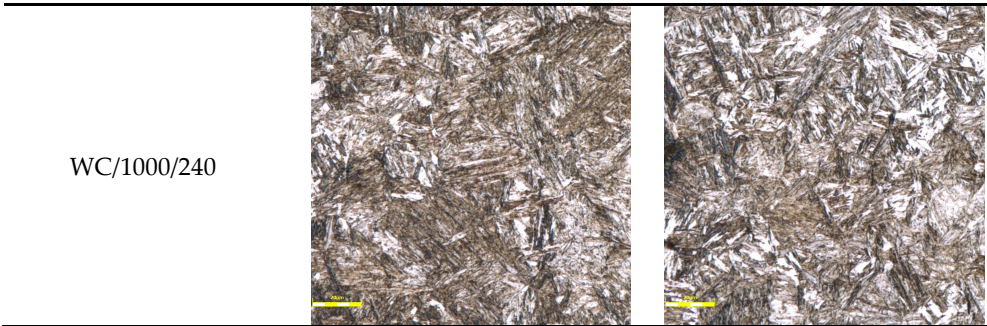


The share of ferrite (light areas) decreased noticeably, whereas the share of perlite (dark areas) increased compared to samples heated at 800°C and air-cooled (AC/800/60 and AC/800/240). A significant grain growth and recrystallization of the defective ferritic matrix were observed, which in turn resulted in a decrease in the tensile strength to the level of  $R_m=619$  MPa (maximum tensile force  $F_m=152$  kN), with  $HV=204$  – for the heating time of 60 minutes and, respectively,  $R_m=576$  MPa (maximum tensile force  $F_m=142$  kN) with a noticeably lower  $HV=153$  – for the heating time of 240 minutes. Attention should be paid to a visible difference in hardness, depending on the applied bolt heating time. In the case of samples cooling down naturally, an increased heating time results in a reduced value of residual tensile strength and hardness of the material. The same trend was also observed in the case of the AC/600 and AC/800 series samples, which may confirm that this trend is of a structured nature.

Heat treatment at the temperature of 1000°C with water-cooling (WC/1000/60 and WC/1000/240 samples), just as in the case of heat treatment at 800°C, did not lead to the martensite → ferrite + perlite phase transformation, although a temperature considerably higher than  $A_1$  was reached. The high cooling rate made the steel harden again, so that the material still had the martensite structure similar to the initial one IS (Table 11).

**Table 11.** Microstructure of steel after heating at 1000°C for 60 and 240 minutes with water cooling.

Sample label	Image at a distance of 3 mm from the sample edge (scale mark 20 μm)	Image at a distance of 7 mm from the sample edge (scale mark 20 μm)
WC/1000/60		



In turn, the high temperature of heat treatment contributed to the formation of coarse-grained martensite. The degree of hardening turned out to be significantly dependent on the heating time. Within 60 minutes, complete austenitization took place and coarse-grained martensite was obtained in the entire volume, which resulted in a significant increase in the tensile strength value to the level of  $R_m=1178$  MPa (maximum tensile force  $F_m=289$  kN), exceeding the reference value for the sample in the initial state, with  $HV=540$ . Heating for 240 minutes turned out to be too long and made the grains grow, which translated into a decrease in the  $R_m$  value to the level of 869 MPa (maximum tensile force  $F_m=213$  kN) with a simultaneous considerable decrease in hardness to  $HV=210$ .

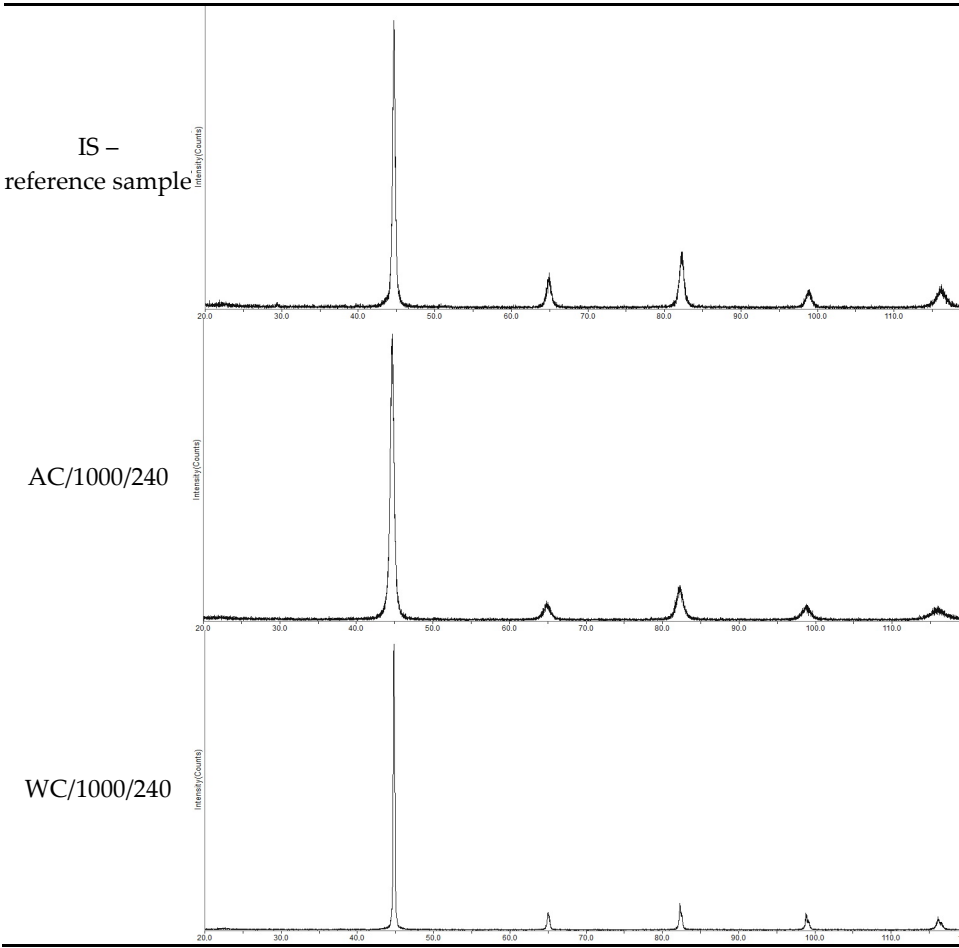
3.2. Testing with the use of X-Ray diffraction (XRD)

32CrB3 steel contains 0.74% of chromium in its chemical composition. Chromium increases the temperature of the austenite transformation, slightly decreases ferrite hardness, increases the amount of residual austenite in the quenching process and increases impact strength. Heat treatment of steel, in which chromium is the alloying element, may contribute to the formation of carbides, which in the end have a noticeable effect on mechanical properties of steel. Carbides formed in steel are a hard and brittle phase. They are formed as a result of the solubility level of carbon in austenite and ferrite changing along with the temperature. The presence of carbides in steel most often increases its hardness, yield point and tensile strength. The carbides are also responsible for the secondary hardness effect, i.e. an increase in steel hardness during tempering. At the same time, their presence can have an adverse effect on impact strength, ductility and fracture resistance of steel.

Increased hardness and residual tensile strength in the case of the majority of samples heated at the temperature of 800°C and 1000°C might lead to a presumption that the presence of carbides in the material structure could be responsible for some of these changes. In order to exclude the presence of carbides in the steel structure before and after the heating process at the temperature of 1000°C, XRD tests were carried out. The tests were carried out both on samples cooled naturally in air – AC, and those shock-cooled – in water – WC. An analysis of the XRD results (Table 12) showed that diffractograms do not differ in the number of peaks, but only in their intensity, which confirms the fact that there are no carbides present in the steel structure before and after the heating process, both in the case of air-cooled and water-cooled samples.

Table 12. Results of the XRD analysis.

Sample label	Diffractogram for a respective sample
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3.3. Quantitative analysis of the microstructure

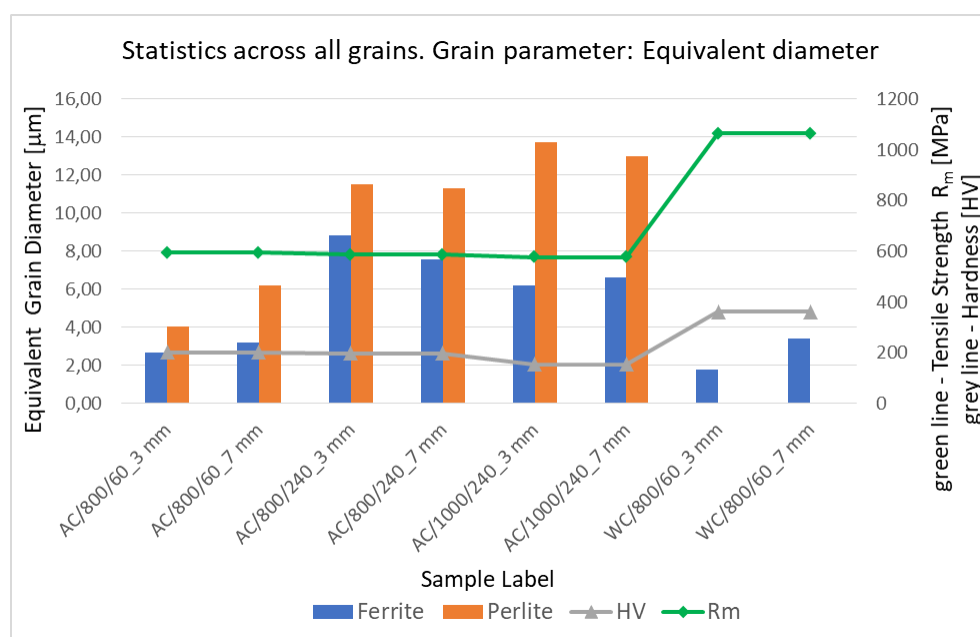
In respect of each of the phases shown in the pictures of the bolt microstructure, taken with the use of a digital light microscope (at the magnification of x100) of the samples specified in Table 4, a quantitative analysis was carried out in order to determine the number of grains visible in the photo, their density, mean grain area, mean and equivalent grain diameter. The analysis was performed separately for the ferrite and perlite phases. The tests were aimed at confirming the previously observed qualitative change in the grain size also in quantitative terms and linking these changes with changes in residual mechanical properties of the analysed samples. Due to limited funds, the analysis was performed only in respect of selected samples.

Collective results showing the dependence of microstructure indices, i.e. mean diameters of ferrite and perlite grains, HV hardness and tensile strength, on heat treatment parameters, are summarised in Table 13 and Fig.3.

**Table 13.** Results of measurements of the mean equivalent diameter of ferrite and perlite grains, hardness and strength of respective series of samples.

Sample label	Equivalent diameter [μm]		Hardness	R <sub>m</sub>
	Ferrite	Perlite	[HV]	[MPa]

AC/800/60_3 mm	2.67	4.01	200.41	594.53
AC/800/60_7 mm	3.19	6.20	200.41	594.53
AC/800/240_3 mm	8.85	11.50	196.46	585.47
AC/800/240_7 mm	7.55	11.30	196.46	585.47
AC/1000/240_3 mm	6.19	13.70	152.98	575.88
AC/1000/240_7 mm	6.62	13.00	152.98	575.88
WC/800/60_3 mm	1.77	not measured	361.26	1063.90
WC/800/60_7 mm	3.39	not measured	361.26	1063.90



**Figure 3.** Diagram presenting dependence of microstructural indices, i.e. mean equivalent diameters of ferrite and perlite grains, HV hardness and tensile strength, on heat treatment parameters

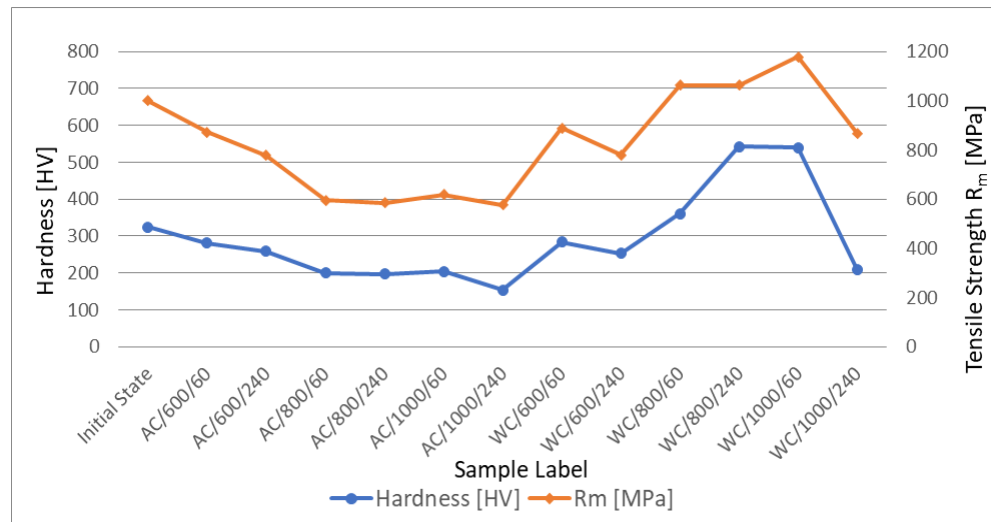
The measurements showed that in the case of air-cooled samples, the size of the perlite grains increased along with an increase in the heating temperature as well as the time of exposure to respective thermal conditions, however the grain growth was not uniform across the entire width of the bolt shank. In the case of ferrite, the grain growth was clearly noticeable along with an increase in the heating temperature to the level of 800°C, but at 1000°C, the grain size slightly decreased. In the case of water-cooled samples, the ferrite grain size is clearly smaller than in the case of air-cooled samples corresponding to them in terms of heat treatment conditions. This confirms the previous observations made on the basis of the visual analysis of the pictures included in Table 11. The water shock cooling of the samples prevented the phase transformation of austenite into perlite and inhibited the growth of ferrite grains. The impact of the shock cooling on the size of ferrite grains was noticeable – near the outer walls of the sample shank, the grain diameter is almost half the size of those near the bolt axis. In the case of air-cooled

samples, along with an increase in temperature and heat-exposure time, a slight downward trend in the value of the residual tensile strength and the hardness of the bolt material was also clearly visible. The water shock cooling re-hardened the bolts and was followed by a sharp increase in both the  $R_m$  and HV values.

#### *3.4. Analysis of correlation between hardness and residual tensile strength*

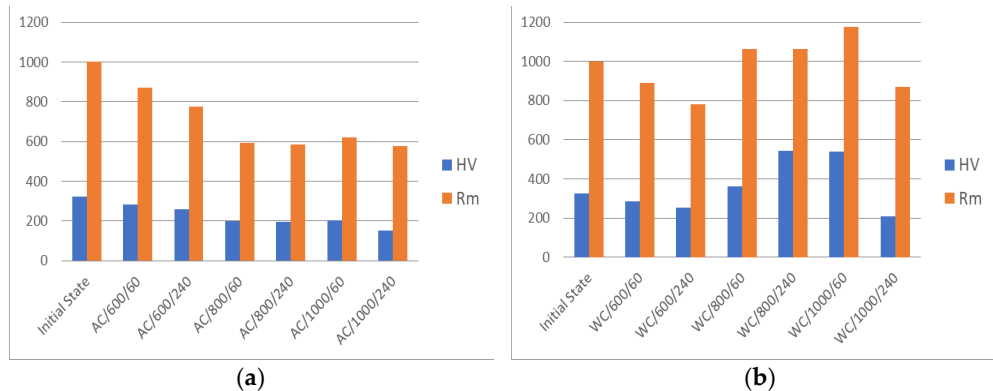
The purpose of the analysis was to investigate veracity of the linear correlation described by the formula (1) between tensile strength  $R_m$  and hardness HV of steel subjected to heat treatment during the production process and then subjected to secondary thermal treatment, e.g. as a result of exposure to thermal effects of a fire. The relationship (1) has been confirmed so far by numerous tests carried out almost exclusively on samples of commonly used structural steels, working in normal thermal conditions. The available literature does not provide any information proving its correctness also in relation to the value of residual tensile strength characteristic of the material of high-strength steel bolts after the fire exposure.

In order to better illustrate the effect of secondary heat treatment parameters on the relationship between hardness HV and residual tensile strength of the bolt steel, a comprehensive diagram has been presented in Fig.4. It shows a clear linear relationship between the values of hardness and tensile strength, but it meets the criterion described by the scaling factor  $3.2\div3.5$  not in the entire domain of determinacy. In the case of air-cooled samples, the value of this factor obtained in the tests fluctuates in the range of  $3.0\div3.8$ , and in the case of water-cooled samples – in the range of  $2.0\div4.1$ , respectively. In the case of air-cooled samples, attention should be paid to a noticeable trend of a decrease in strength (in relation to the reference value, characteristic of the material in its initial state), accompanying a temperature and heating time increase. The hardness parameter also shows a similar trend. In the case of shock water-cooled samples, this trend, characteristic of air-cooled samples, is reversed. Along with an increase in the temperature and heating time – values of the residual tensile strength and hardness of the bolt steels increase. Some regularity of this trend is locally disturbed only in the case of the WC/600/240 and WC/1000/240 samples, which may, however, be caused by a systematic error due to a small size of the sample. Confirmation of this thesis would nevertheless require further research. In the case of water shock cooling of samples (simulation of a rescue and firefighting operation), both residual tensile strength and hardness of the bolt material increase significantly and under certain conditions even exceed the reference values characteristic of the tested high-strength bolts in their initial state. On the one hand, it can be perceived as a desirable effect, due to significant strengthening of the material, and on the other hand – as negative, due to an increase in its brittleness, which translates into the possibility of an abrupt form of failure in the event of overloading the bolts in the joint.



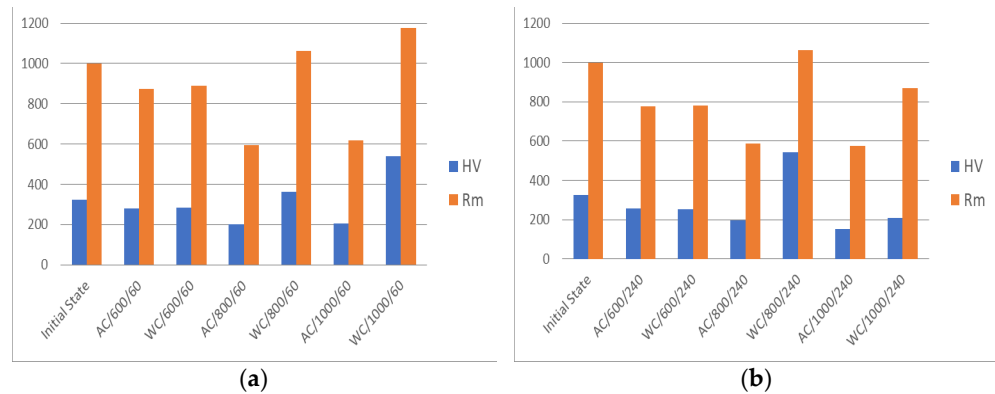
**Figure 4.** Diagram of correlations between hardness HV and residual tensile strength of the bolt steel after secondary heat treatment, depending on parameters of this treatment

In order to determine the effect of the cooling method, temperature level and heating time on the disturbance of the relationship between the hardness of the bolt material and its residual tensile strength, the relationships between these two values in different configurations are shown in Fig.5-Fig.6.



**Figure 5.** Diagram of dependencies between bolt steel hardness and bolt residual tensile strength on heating time, for samples: (a) naturally air-cooled; (b) shock water-cooled.

When analysing the diagrams shown in Fig.5, it can be noticed that a negative effect of long heating of particular series of samples is clearly visible – both in the value of residual tensile strength  $R_m$  and hardness HV. The same trend showing the effect of the heating time on stability of the analysed material properties is visible for each temperature level. Excessively long heating results in a decrease in residual strength. In consequence, material hardness is also reduced.



**Figure 6.** Diagram of dependencies between bolt steel hardness and bolt residual tensile strength on heating time, for samples subjected to secondary heat treatment for the time of: (a) 60 minutes; (b) 240 minutes.

The analysis of Fig.6 shows that in the temperature range up to 600°C, both in the case of air-cooled bolts and those water-cooled, no effect of the heating time on stability of the analysed mechanical properties of the samples has been identified. Corresponding pairs of graphs for the AC/600/60 and WC/600/60 and AC/600/240 and WC/600/240 samples do not differ from each other. The only difference between the bars of the graphs shown in Fig.5a and Fig.5b is in the  $R_m$  and HV values. Even the  $R_m$ /HV interrelationships for the corresponding pairs of samples are almost identical. This confirms the previous observations made on the basis of the analysis of microstructure pictures, described earlier in section 3.1.1 of this article.

An increase in the heating temperature to 800°C and higher makes the differences between the air-cooled and water-cooled samples clearly visible. Under similar thermal conditions, water shock cooling results in achieving a much higher residual tensile strength of the sample material –  $R_m$  and hardness HV compared to the sample freely cooling in the air. This results from the fact that the samples are re-hardened and the processes of phase transformations are stopped.

#### 4. Discussion, conclusions and recommendations

The microstructural tests confirmed that under environmental conditions corresponding to a simulated fire situation and an accompanying firefighting operation, significant structural changes occur in the material of bolts, strongly dependent on both the temperature reached, the time of exposure to fire conditions, and the method of cooling. These changes result in modifications of residual strength properties, crucial from the point of view of structural safety, in particular – tensile strength and correlated hardness. Microstructural changes significantly affect how the bolt material behaves, which usually determines how the structure will fail in the event of a potential collapse. The bolts heated to a temperature exceeding 600°C and cooled naturally in the air, as a result of fire exposure, undergo a martensite → austenite phase transformation during the heating process and then ferrite and perlite are released within the boundaries of austenite grains in their structure. This structural modification makes steel softer and bolt load-bearing capacity considerably lower, however, as a rule, at the expense of increased plasticity, therefore an overloaded bolt does not rapidly fail. Bolts that underwent such a heating cycle permanently lose their original strength properties. Bolts heated to a temperature exceeding 600°C and shock cooled with water (e.g. during firefighting operation in a real fire) retain a martensite structure, similar to the original one, because they are re-hardened through rapid cooling. Due to the sudden reception of thermal energy, they do not undergo the microstructural change characteristic of the martensite → ferrite + perlite phase transformation, despite reaching a temperature significantly higher than  $A_1$ . Steel of the bolt hardens and the temporary load capacity of bolts can as a result of the re-hardening even exceed the load capacity that the bolt had in its initial

state or be very close to this value. This is done at the expense of increased brittleness of the bolt, so that in the event of overload, the bolt can be expected to fail rapidly.

Considering the foregoing, the method of carrying out a rescue and firefighting operation may be of key importance for safety of the structure and people staying in a building in fire. In the case of structures subject to dynamic loads, water cooling of bolts should be avoided, as this will make them more susceptible to brittle fracture. In the case of fire-damaged structures that have already undergone significant deformations, potential gains and losses should be assessed somewhat on a real-time basis. If members have not been deformed by a fire and there is a real chance of renovating and re-using – then the risk of cooling the bolts may be taken in order to increase their load capacity in real time. Of course, during the reconstruction or renovation of the structure, such bolts must be replaced with new ones with predictable strength properties. If the structure cannot be saved, its stability is compromised or it has undergone significant deformations preventing it from being reused, bolts should rather not be hardened to allow their slow plastic failure at the moment of being overloaded.

The conducted research confirmed the possibility of using microstructural tests for a post-fire assessment of steel structures and an attempt to reconstruct fire scenarios. However, unless an as precise as possible fire temperature is known, an assessment based solely on microstructural tests may not be sufficient or may be flawed with a significant error. This may be the case, for example, when the image of the bolt material microstructure indicates a martensite structure. In the case of high-strength bolts, it is typical for bolts in their initial state, bolts heated to a temperature lower than 600°C, and bolts heated to a much higher temperature and rapidly cooled with water. In this case, a reliable assessment cannot be made without conducting additional destructive strength testing. This example shows that the post-fire assessment of bolts should be done in an extremely reasonable and careful way.

The undertaken research and promising results obtained indicate that this work should be continued, also with focus on development of detailed guidelines for designing bolted joints, taking into account fire effects. There are still no precise guidelines in this respect in standards applicable to design of structures [12-13].

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## References

1. Kirby B.R. The behaviour of high-strength grade 8.8 bolts in fire. Technical report SL/HED/R/S1792/1/92/D. British Steel Technical Swinden Laboratories. 1992
2. Kirby B.R. The behavior of high-strength grade 8.8 bolts in fire. *J. Construct. Steel Research* **1995**, 33, pp. 3-38.
3. González F.; Lange J. Behaviour of high strength grade 10.9 bolts under fire conditions, Proceedings of the International Conference on Application of Structural Fire Design, Prague, Czech Republic, 19-20 Feb. 2009; Publisher: Prague, Czech Republic, 2009, pp. 392-397

4. Hanus F.; Zilli G.; Franssen J.-M. Behaviour of grade 8.8 bolts under natural fire conditions – Tests and model. *J. Construct. Steel Research* **2011**, 67, pp. 1292-1298; doi:10.1016/j.jcsr.2011.03.012
5. Kodur V.; Kand S.; Khaliq W. Effect of temperature on thermal and mechanical properties of steel bolts. *J. Mater. Civ. Eng.* **2012**, 24(6), pp. 765-774; doi: 10.1061/(ASCE)MT.1943-5533.0000445.
6. Kodur V.; Yahyai M.; Rezaeian A.; Eslami M.; Poormohamadi A. Residual mechanical properties of high strength steel bolts subjected to heating-cooling cycle. *J. Construct. Steel Research* **2017**, 131, pp. 122-131; doi:10.1016/j.jcsr.2017.01.007
7. Yahyai M.; Kodur V.; Rezaeian A. Residual mechanical properties of high strength steel bolts after exposure to elevated temperature. *J. Mater. Civ. Eng.* **2018**, 30(10); doi: 10.1061/(ASCE)MT.1943-5533.0002416.
8. Chi J.-H.; Peng P.-C. Using the microstructure and mechanical behavior of steel materials to develop a new fire investigation technology. *Fire and Materials*. **2017**, 41, pp. 864-870; doi: 10.1002/fam.2438
9. Sajid H.U.; Kiran R.; Naik D. Microstructure-mechanical property relationships for post-fire structural steels. *J. Mater. Civ. Eng.* **2020**, 32(6); doi: 10.1061/(ASCE)MT.1943-5533.0003190.
10. Haiko O.; Kaijalainen A.; Pallasburo S.; Hannula J.; Porter D.; Liimatainen T.; Kömi J. The effect of tempering on the microstructure and mechanical properties of a novel 0.4C press-hardening steel. *Appl. Sci.* **2019**, 9, 4231; doi:10.3390/app9204231.
11. Xie Z.; Song Z.; Chen K.; Jiang M.; Tao Y.; Wang X.; Shang C. Study of nanometer-sized precipitation and properties of fire resistant hot-rolled steel. *Metals*. **2019**, 9, 1230; doi:10.3390/met9111230
12. EN 1993-1-2 Eurocode 3: Design of steel structures – Part 1-2: General rules – Structural Fire design. European Committee for Standardization, Brussels
13. EN 1994-1-2 Eurocode 4: Design of composite steel and concrete structures – Part 1-2: General rules – Structural Fire design. European Committee for Standardization, Brussels
14. Regulation of the Minister of Infrastructure of 12 April 2002 on the technical requirements to be met by buildings and their location, Dz.U. (*Journal of Laws of the Republic of Poland*) 2019, item 1065; in Polish
15. Construction Law Act of 7 July 1994, Dz.U. (*Journal of Laws of the Republic of Poland*) 2020, item 1333; in Polish
16. Regulation (EU) No 305/2011 of the European Parliament and of the Council of 9 March 2011 laying down harmonised conditions for the marketing of construction products and repealing Council Directive 89/106/EEC (Text with EEA relevance), Official Journal of the European Union L 88/5, <http://data.europa.eu/eli/reg/2011/305/oj>.
17. Bhadeshia H.; Honeycombe R. Steels: Microstructure and Properties, 3rd Edition, Butterworth-Heinemann, ISBN 9780080462929