AST Advances in Science and Technology Research Journal

Advances in Science and Technology Research Journal 2022, 16(3), 306–315 https://doi.org/10.12913/22998624/150564 ISSN 2299–8624, License CC-BY 4.0 Received: 2022.04.30 Accepted: 2022.05.14 Published: 2022.06.01

Studies on the Mechanical Properties of C45 Steel with Martensitic Structure after a High Tempering Process

Waldemar Woźniak¹, Michał Sąsiadek¹, Tomasz Jachowicz^{2*}, Milan Edl³, Paweł Zając⁴

- ¹ University of Zielona Góra, Faculty of Mechanical Engineering, ul. Prof. Szafran 4, 65-516 Zielona Góra, Poland
- ² Lublin University of Technology, Faculty of Mechanical Engineering, ul. Nadbystrzycka 38, 20-618 Lublin, Poland
- ³ University of West Bohemia, Faculty of Mechanical Engineering, Department of Industrial Engineering and Management, 8 Univerzitni Str., 30614 Plzen, Czech Republic
- ⁴ Wroclaw University of Science and Technology, Faculty of Mechanical and Power Engineering, Wybrzeze Wyspianskiego 27, 50-370 Wroclaw, Poland
- * Corresponding author's e-mail: t.jachowicz@pollub.pl

ABSTRACT

This article describes the studies of the mechanical properties of the martensitic structure of C45 steel, obtained as a result of heat treatment. This steel was subjected to high tempering, within the temperature range of $500\div700^{\circ}$ C (every 50° C) and for various exposure times, from 15 minutes to 23 hours. Moreover, martensitic steel was subjected to tests by quenching at a temperature of 850° C for 20 minutes and then tempering it for 1 hour, within the temperature range of $50\div800^{\circ}$ C (every 50° C). The resulting steel samples were subjected to strength tests, that is, to tensile and hardness tests and also to tests on the micro-structure. The results of these studies are presented and discussed, in detail, in terms of their practical application.

Keywords: steel, heat treatment, quenching, tempering, steel structure, martensitic structure.

INTRODUCTION

Machine parts made of medium-carbon steel by cavity processing, such as by forging or casting, are very often subjected to pre-thermal or thermo-chemical treatment. in order to improve their performance properties. Pre-heat treatment consists of several stages. Usually the first of these is quenching or isothermal quenching, which results in various steel structures such as martensite, bainite, or perlite with ferrite; this possibly tolerates a mixture of these structural components [1, 2]. Most often, the next stage of the heat treatment operation is tempering which can be performed at different temperature ranges, namely, low, medium and high tempering. Manufacturers of heat-treated, steel parts strive to reduce heat treatment, time and hence energy consumption costs. It is well known that heat

treatment has a significant impact on production costs. The best combination of steel properties, in terms of ductility and strength, is achieved as a result of "thermal improvement", so-called, which is a combination of the two operations of quenching and high tempering.

The development of metal science, especially the development of new research tools, used in metallographic research, provides all the best cognitive capabilities that are used for production management. Information, obtained as a result of research, can be translated into the parameters of the production process of machine parts, carried out using heat treatment. The purpose of such research is very often to reduce the cost of production, by reducing processing time and reducing energy consumption, along with the use of new media in production thus improving the performance of the products and/or the materials [3, 4]. This improves the competitive position of the parts manufacturer and gives it a market advantage.

Modern science, dominated by new developments in the field of composite materials, nanotechnology, etc., allows C45 steel to be looked at as a material that, after thermal improvement, can be considered as a composite, consisting of a base, in the form of soft ferrite, reinforced with granules of hard cementite. This expression can also describe the ferritic-perlitic structure of this steel, after such as hot rolling, after normalisation, with the exception that, in this case, it will be ferrite, reinforced with a lamellar form of cementite. These two forms of cementite, present in C45 steel, have long been known about and recognised and are a classic, metallurgical problem. It is known that the tile form of cementite gives the worst performance, whereas the ball form is better [5]. At present, we are more interested in considering how the release of cementite, obtained after tempering, affects its usefulness. Their sizes vary, depending on the temperature at which the tempering is conducted. Such information is not available in the literature and therefore an attempt has been made to obtain it through experiments, the results of which are presented in this article. For this article, grade C45 steel was selected for the study with only those changes in the structure and properties being analysed that would appear in the steel under study when the technological parameters were changed, during the quenching and tempering processes. The applied, empirical research method consisted in conducting a series of heat treatments under laboratory conditions and describing the effect of the changes in the parameters of these processes on the structure and properties of the material under study.

When the correct method of cooling was chosen, the martensitic structure was obtained [6]. Additionally, it was subjected to high tempering during the various parameters of the process, with any samples, prepared in this manner, being examined both with regard to structure and to strength, using a scanning microscope for the static, tensile testing and for measuring micro-hardness. Steel testing can involve many aspects. The most common of these are mechanical studies, metallographic or structural studies, as well as studies on anti-corrosion and other properties. In the literature, a large area of research is devoted to the strength testing of welded, steel joints. A unified description of the hardness of martensitic steels for a wide range of carbon content has been presented in [7].

These concern, among other things, tests for the strength of welded joints, obtained by a fibre laser at various welding speeds [8]. In [9] steels with 12% CR, enhanced with Z phase, were tested to determine the dependence between corrosion and its resistance to creeping. In [10], the mechanical properties, that is, the stretching and elongation, of ferritic and martensitic steel were tested, depending on the different shares of C, N and W. In another aspect [11], it was proposed to develop ultra-high-strength steels, intended for cold stamping in the automotive industry, by using special alloy additions. In [12] presents the results of tests on the strength of steel as a result of the repeated quenching-partitioning-tempering process and replacement of 1.5% (wt.%) of Si with a 2% (wt.%) Al addition. Also, in [13, 14], martensitic devices, designed to work in ultra supercritical- parameters, $(T = 620 \div 650^{\circ}C, P =$ 25÷30 MPa) were subjected to constant tests, obtained as a result of the normalisation process at temperatures above A3 and tempering at temperatures below A1. In turn, the effect of thermal ageing on mechanical properties and micro-structure, in low-activation martensitic steel, has been developed [15]. Strength tests of low-activation martensitic steel, subjected to the ageing process at 550°C over 20,000 h, are presented in [16]. The ageing process has also been the subject of work [17]. In [18], micro-structural changes resulting from shock loads and their influence on the mechanical properties of steel were analysed. The study of the influence of the quenching process on low-carbon martensitic steel and on micro-structural, tensile properties, as well as the influence of both susceptibility and bending, were the subjects of research described in [19]. The quenching of martensitic steel with chromium, at high temperature and the effect of this process on tensile strength and the fracture mechanism, were the subject of the research, presented in [20]. Studies of the influence of the evolution of the microstructure of steel (16 wt.% Cr; 4.5 wt.% Ni; 1.6 wt.% MO; 0.9 wt.% B; 0.6 wt.% Mn and 0.12 wt.% C) on the improvement of resistance to corrosion and impact strength, is described in [21]. In [22], the frictional behavior of low-alloy martensitic steel with silicon nitride was investigated. The process of the quenching and tempering of martensitic steels was the subject of research in [23], where the influence of the carbides on

strength properties was determined. Issues related to the tempering of martensite are presented in [24]. Other studies focus on the micro-structure, where the formation of crystal plasticity, due to temperature changes, was studied for martensitic steels [25]. Other studies [26] considered the effect of Ti, as a low-activation, alloying element on the micro-structure and on mechanical properties.

In the literature, studies of the tribological properties of products made of perlitic and martensitic steel [27] can also be found. In turn, an overview is presented in [28], of the micro-structures and the mechanisms by which they are formed by tempering, patched martensite, with low and medium carbon content. High-strength martensitic steel was subjected to heat treatment by quenching-partitioning-tempering (Q-P-T). The mechanism that improves both the plasticity and stability of austenite at high temperatures, has been studied [29]. A new, hybrid approach for describing and simulating the creeping behavior of improved, martensitic steels is presented in [30, 31]. In [32], the mechanical characteristics of five, low-carbon martensitic steels, tempered over a wide temperature and time range are presented and the relationship between the mechanical properties, hardness and the tempering conditions were further analysed. Quenched martensite, obtained from four different tempering modes, was characterized in [33, 34].

In the context of the literature review in question, the aim of this work is to determine the characteristics and properties of C45 steel (1.0503) subjected to martensitic hardening based on its tempering at elevated temperatures for varying times. Furthermore, an additional study was carried out by hardening the steel in a polymer solution after heating the samples to 850 °C and holding them at this temperature for 20 minutes. In the characterised process, the following parameters were studied: strength, microhardness and microstructure of steel samples obtained in different process variants of tempering of martensitic steel.

MATERIALS AND METHODS

Steel C45 (1.0503), in accordance with EN 10277-2-2008, refers to high-quality, unalloyed

steels for heat treatment. The C45 steel is used for machinery and equipment components with a medium load, such as spindles, non-hardened gears, axles, shafts, motor shafts, levers, conventional knives, disks, bolts, corkscrews, wheel hubs, rollers, pump rotors and rods. This steel is easily subjected to hot and cold plastic processing and belongs to difficult-to-weld steels. The C45 steel studied was analysed to identify the elements that make up its composition. The designated chemical composition (obtained by chemical testing) is shown in Table 1.

Samples, subjected to martensitic quenching, were heated to the austenitisation temperature (850°C) and kept at this temperature for 20 minutes and then cooled in an aqueous polymer solution. For manufactured, martensitic, steel structures, high tempering was undertaken in the temperature range: 500÷700°C (every 50°C). The tempering time for each temperature variant was: 15 min, 1 h, 3 h, 9 h, and 23 h. Three samples were allocated for each variant of the processing time period. For each sample, a tensile test, a hardness test and a structure test were carried out after tempering. An additional experiment was carried out on the initial structure of the martensitic steel obtained. The test material was tempered to 850°C and kept at this temperature for 20 minutes. Quenching in the range of 16 temperature options (50, 100, 150, 200, 250, 300, 350, 400, 450, 500, 550, 600, 650, 700, 750 and 800°C) was conducted for 1 hour.

Suitably prepared specimens were tested using following equipment:

- tensile tests and, based on them, determination of the strength properties were carried out on an INSTRON 8202 testing machine,
- hardness tests were carried out using a Zwick / Roell ZHV 10
- microstructure tests were carried out on a JEOL JSM-6400 Scanning Microscope.

RESULTS

Structures after the application of heat treatments

After cooling at different speeds and after tempering at different temperatures and times, the

Table 1. Chemical composition of steel C45 under study

Stool grada	Chemical composition, %								
Steel grade	С	Mn	Si	Р	S	Cr	Ni	Cu	Al
C45	0.48	0.71	0.25	0.013	0.023	0.10	0.09	0.18	0.023



Figure 1. Spheroidite structures after the high tempering of martensite (magnification 5000×)

Parameter	Temperature,°C	Tempering time							
		15 min	1 h	3 h	9 h	23 h			
Re (MPa)	500	1225	1035	1000	980	950			
	550	960	910	900	830	760			
	600	900	780	760	700	660			
	650	770	660	660	600	560			
	700	605	590	570	535	470			

Table 2. Tensile test results for parameter Re – martensitic structure

Table 3. Tensile test results for the Rm parameter – martensitic structure

Parameter	Tomporature °C	Tempering time							
	Temperature, C	15 min	1 h	3 h	9 h	23 h			
R _m (MPa)	500	1289	1085	1045	1021	996			
	550	1030	968	953	878	819			
	600	957	839	829	747	714			
	650	810	715	723	646	617			
	700	738	636	630	586	546			

structures are now shown on the summary boards in Figure 1. Structures after heat treatment were presented at various magnifications, whereas structures after high tempering were shown at the standard magnification of 5000×.

The results of tensile tests

In the tensile test, the main strength parameters (Rm, Re, A%) characterising the material under study, were determined, i.e. yield strength Re, tensile strength Rm and elongation – A. The tensile test was performed on a Hegewald & Peschke test machine, model: Inspekt Table 100 in a company working for the automotive industry. The results of the tensile tests for the specified yield strength, tensile strength, and elongation limits for the martensitic structure are shown in Tables 2, 3 and 4. The dependence of the average

Table 4. Tensile test results for the elongation parameter A – martensitic structure

Parameter	Tomporaturo °C	Tempering time							
		15 min	1.5 h	3 h	4.5 h	12 h	23 h		
A%	500	12.2	12	11.7	12.2	10	9		
	550	13.5	11.4	13	13.2	13.5	13.2		
	600	17.1	12.6	13	12.8	14	15.4		
	650	15.7	17.9	13.5	19.5	18	17.6		
	700	20.2	21.1	21.5	22.1	21.2	20.1		



Figure 2. The 3D graph of the relation between the mean yield point and the tempering time and the temperature for a martensitic structure



Figure 3. The 3D graph of the relation between the tensile strength and the tempering time and the temperature for a martensitic structure

Temperature, °C	Tempering time							
	15 min	1 h	3 h	9 h	23 h			
500	380	351	346	323	318			
550	336	311	296	289	268			
600	302	278	257	244	237			
650	274	249	224	212	205			
700	237	223	211	193	185			

Table 5. Average values of HV 0.5 micro-hardness measurements after tempering - martensitic structure



Figure 4. The 3D graph of the relation between the average hardness value and the tempering time and the temperature for a martensitic structure

hardness value, on the time and temperature of tempering, is shown in Figure 2. The dependence of the average tensile strength, on the time and temperature of tempering is shown in Figure 3.

Results of micro-hardness measurements

The results of the micro-hardness measurements for the martensitic structure after tempering are presented in Table 5. The dependence of the average hardness value, on the time and temperature of tempering, is shown in Figure 4.

Results of additional experiment

In an additional experiment, tempering for all temperatures was carried out for 1 hour. Quenching was carried out in a polymer solution after heating the samples to 850°C and maintaining this temperature for 20 minutes. The micro-hardness HV 0.5 of the material before and after the quenching of the martensitic structure is shown in Table 6. The micro-hardness of the quenched material, after tempering, is shown in Table 7. The average values were calculated from 5 measurements, after rejecting maximum and minimum values.

RESULTS INTERPRETATION

Changes in particle size when tempering various source structures

The micro-structure studies after quenching, undertaken with a scanning microscope, showed that the variability in the size of the globular particles of Fe_3C cementite, is in the range from 8 to 1000 nm.

The effect of the structure of the output on the properties

The subject of observations and analyses were, among other things, initial structures: martensite, bainite and nanoperlite, with an 8% admixture of peredeutectoid ferrite. The highest Rm value was obtained for martensite (1196 MPa), while a lower value was found for bainite (936 MPa) with the lowest value of all being found for nanoperlite (919 MPa).

A comparison of mechanical properties (Re and A) indicates that bainitic and nanoperlytic structures have the lowest properties and that a martensitic structure, with a low A parameter

Table 6. Average	values of HV 0.	.5 micro-hardness	measurements	before a	and after	quenching	– martensitic
structure							

Variant	Measurement results HV 0.5	Average HV 0.5
Before quenching	308, 288, 312, 306, 301	305
After quenching in an aqueous polymer solution	654, 656, 663, 651, 660	657

Table 7. Values of HV 0.5 micro-hardness measurements after tempering - martensitic structure

Temperature, °C	1	2	3	4	5	Average
50	649	641	646	640	646	644
100	641	661	647	640	655	648
150	591	589	584	593	587	589
200	565	584	563	560	570	566
250	510	512	510	509	511	510
300	471	465	478	480	473	474
350	405	418	422	420	421	420
400	391	391	395	400	392	393
450	383	382	386	380	379	381
500	359	359	359	360	355	359
550	316	314	315	318	315	315
600	288	285	290	284	289	287
650	252	250	256	251	254	252
700	215	233	235	234	229	232
750	223	224	226	226	225	228
800	215	216	214	214	213	214

value, indicates that the material is very brittle. The change in the characteristics of the material occurs after high tempering. From martensite, which is a form of ferrite, supersaturated with carbon, globular particles of cementite are released. Their size increases as a result of coagulation with increasing time and the temperature of the tempering. The distribution of cementite particles over the entire volume can be estimated as homogeneous.

An indicator of changes in properties and structure, that is, the size of sections that strengthen steel, results from measurements of microhardness. Studies show that the temperature increase has a stronger effect on the hyperplasia of cementite particles during coagulation than does the time of release. Therefore, from the point of view of controlling the tempering process, it is better to regulate the properties by selecting the appropriate temperature of the tempering and secondly, by selecting the time for the duration of the tempering. Manufacturers of heat-treated parts usually seek to reduce the time given over to heat treatment. With the data obtained, the necessary temperature and time parameters can be selected. However, it is necessary not to overdo the time reduction element, so as not to enter the

range of unstable properties that occur with very short treatment times.

Effect of tempering conditions on the final properties of steel with different output structures

The influence of the tempering conditions and initial structures on the final properties, indicates that the best mechanical properties were obtained for the martensite structure over short times and low temperatures of tempering. As the temperature of the tempering increases, differences in the output structures had less and less of an influence on the levels of Re, Rm and HV 0.5 micro-hardness obtained – due to the fact that the structure of the material has reached a state of equilibrium.

The phenomena that occur during the tempering of C45 steel

A summary of the phenomena that occur when steel, quenched for martensite is being tempered, has been provided in a graph, based on an additional experiment discussed in additional experiment. Samples, with a martensitic structure were kept for 1 hour at various temperatures (at



Figure 5. Graph of the influence of temperature on the value of micro-hardness HV 0.5.

 50° C intervals) in the range of $50 \div 800^{\circ}$ C, after which their micro-hardness was measured.

The resulting curve shows a decrease in hardness with an increase in temperature; this decrease is not monotonic as the curve is undulatory, in character. This is due to the many phenomena that overlap when the temperature rises. These phenomena can be observed using dilatometric measurements. They are as follows (Figure 5):

- during the initial heating period, carbon segregation occurs,
- the secretion of metastable carbide begins above temperatures of approximately 70÷80 °C,
- above 210°C, the process of cementite Fe₃C secretion begins and continues until approximately 410°C. The size of Fe₃C secretions obtained in this temperature range does not exceed approximately 10 nm,
- in the range of approximately 220÷300°C, which is superimposed on the range of cementite secretion, the residual austenite is converted into martensite,
- above 300°C, the recovery period of the quenched structure begins and then turns into polygonisation, which take place in the range of up to approximately 600°C,
- above approximately 410°C, all the carbon is almost completely secreted and is present only as cementite with a spherical shape, which, with the increasing temperature of tempering, assumes ever increasing sizes as a result of the coagulation process. Since the coagulation process is a diffusive process, increasing

the temperature contributes to the growth of particles,

- above 600°C, the phenomenon of recrystallisation occurs, after which the micro-hardness is the lowest possible and is comparable to the micro-hardness of normalised material. This phenomenon explains why the impact strength on Figure 5 falls above 630°C,
- the above temperatures are indicative values, since the temperatures at the beginning and at the end of each secretion process and the changes occurring in the material depend on the heating rate.

The undulatory nature of the tempering curve, shown in Figure 5 deviates from the simplified versions, presented in the literature as a monotonic flow. The results of the measurements of micro-hardness in the framework of the "*additional experiment*" seem closer to reality and better reflect the complex nature of many phenomena that occur during tempering. Therefore, the literature data on the hardening curve should be considered as fairly indicative information.

CONCLUSIONS

The significance of the thermal improvement parameters is as follows:

- 1. In the quenching range:
- The austenitisation temperature (before quenching) should be higher than A₃.

- For each steel grade, the A₃ temperature can be read from the Fe-Fe₃ C graph. If it is high, it results in faster austenite homogenisation. However, the disadvantage that then occurs is the coarse-grained nature of the structure. Hence, it is best to strive for the lowest temperature, just above A₃. In practice, the temperatures used are higher by 30÷50°C than the temperature of A₃. The carbon solubility in austenite is very high and the carbon diffusion coefficient is also high in the austenitic range, so the austenitisation temperature should not be too high. It is beneficial if austenite is homogeneous.
- The austenitisation time is the second factor that affects the production of homogeneous austenite. This parameter depends very much on the cross-section size of the parts undergoing quenching and must be selected individually for each type of work.
- The cooling speed. After austenitisation, it is beneficial to conduct cooling at the maximum possible speed allowed for each steel (in the range of 200 degrees per second and more), greater than the critical cooling rate and depending on the type of cooling medium. When selecting quenching baths, the principle should be used to ensure that the cooling rate is sufficient to induce the planned structural changes. However, too high a speed can lead to quenching cracks as a result of thermal stresses and the stresses caused by structural transition during cooling.
- 2. In the tempering range:
- Using various, pairing combinations of temperature / time, the same or very similar properties can be obtained. The tempering temperature has a stronger influence on the change in mechanical properties than does the tempering time. Tempering time a very commonly used tempering time is about 2 hours; this can be reduced, but only if the quenching temperature increases.
- The application of thermal improvement processes, using the knowledge obtained in the research characterised in this work, has been implemented in two case studies of forgings, produced for the automotive industry. In each of these cases, the general characteristics of the steel, from which these parts are made, are known, but the parameters of the thermal improvement process were selected individually and differed in details from the literature data.

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