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Restoration of Properties of Heat-Resistant Steel After Long-Term Operation in Steam Pipeline Bends of TPP by Heat Treatment

Halyna Krechkovska [1,2], Ivan Tsybailo [1], Ihor Dzioba [3,*], Oleksandra Student [1] and Robert Pała [3]

1 Department of Diagnostics of Materials Corrosion-Hydrogen Degradation, Karpenko Physico-Mechanical Institute of the National Academy of Science of Ukraine, 5 Naukova Str., 79060 Lviv, Ukraine; krechkovskahalyna@gmail.com (H.K.); tsybailo.14@gmail.com (I.T.); oleksandrastudent1@gmail.com (O.S.)
2 Department of Materials Science and Engineering, Lviv Polytechnic National University, Ustyianovycha Str. 5, Building 10, Room 28a, 79013 Lviv, Ukraine
3 Faculty of Mechatronics and Mechanical Engineering, Kielce University of Technology, Av. 1000-An. of Polish State 7, 25-314 Kielce, Poland; rpala@tu.kielce.pl
* Correspondence: pkmid@tu.kielce.pl

Abstract: To improve the microstructure and mechanical properties of heat-resistant 12Kh1MF steel after long-term operation in the stretched bend zone of the main steam pipeline of a thermal power plant, restorative heat treatment (RHT) was proposed. The RHT mode consisted of two normalization stages (from temperatures of 1100 and 960 °C, respectively) followed by tempering at a temperature of 740 °C. The RHT mode, regulated for steel in the initial state, was applied only after its normalization from a significantly higher temperature (1100 °C). It was shown that the proportion of fine grains in the steel structure increased to 55% over the entire pipe wall thickness after using RHT. At the same time, the proportion of large grains in the restored steel decreased significantly (to 10%), while in exploited steel, their proportion reached almost 50%. The proposed RHT mode increased the hardness, strength, plasticity, and resistance to brittle fracture of the restored steel relative to the corresponding characteristics of the operated steel. The maximum positive effect of the RHT was obtained during impact testing. The fractographic features of the exploited and restored steel were studied on fractures of samples tested by tension. The main fractographic feature of the operated steel was nanosized particles at the bottom of large dimples. These tiny particles were considered to be fragments of large carbides formed due to their final decohesion from the matrix during tensile testing. However, such nanosized particles were not found on the samples' fracture surfaces in the steel after restorative heat treatment. In addition, the ductile dimples on the fractures of the restored steel were more prominent, which indicated high energy costs for their formation. Thus, all the obtained research results suggest the possibility of using the proposed RHT mode to extend the service life of long-operated critical elements of a thermal power plant's steam pipelines.

Keywords: heat-resistant steel; restorative heat treatment; microstructure; mechanical properties; fractographic features

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1. Introduction

The physical wear of thermal power equipment is largely due to the exhaustion of its design resources, and sometimes to unscheduled operation. The appearance of damage in the bends of the main steam pipelines of thermal power plants (TPPs), as critical elements of the system for supplying coolant to the turbine, creates a significant danger for personnel and the environment. These components are usually made of low-alloy heat-resistant steels, since they are subjected to high stresses and temperatures over long periods of operation (decades). Recently, researchers have also turned their attention to the high-temperature oxidation of heat-resistant steels as a factor in the deterioration of their properties [1]. The morphology of oxides, their growth rate, phase composition, and structure at high temperatures are analyzed [2,3]. Such conditions promote the creep process, leading to changes in their structure [4,5] and physical and mechanical properties [6–9]. In particular, due to creep, the grain sizes increase, the carbides at their boundaries coagulate, and, finally, due to the separation of carbides from the matrix, pores appear [10,11]. Ultimately, the coalescence of these pores leads to intergranular cracking [12,13]. As a result, the degradation of heat-resistant steels, as well as changes in their substructure, accelerate the creep process. The growth of sub-grain sizes [14,15] contributes to a decrease in the content of alloying elements in the ferrite matrix [16–18] and the redistribution of carbides to the grain boundaries, their separation from the matrix with the formation of pores along the grain boundaries, and, ultimately, the inter-crystalline cracking of steels due to creep [11,19–21]. In particular, the deterioration of the properties of steels operated for a long time at elevated temperatures is associated with structural changes (such as an increase in grain size) and phase transformations (including the precipitation and coagulation of carbides along the grain boundaries) [22–25].

Considering the widespread use of thick-walled pipes in the main steam pipelines of many TPPs, the simultaneous replacement of all damaged pipes with new ones is associated with significant financial costs and is often simply impossible. However, restoring the functionality of the most critical sections of steam pipelines (such as bends and welded joints, where steel degradation occurs much more intensively than on straight sections of pipes) is becoming a completely justified approach. This is particularly relevant in the context of the fight against global warming, since the production of every ton of new pipes makes a significant contribution to CO_2 emissions and, thus, has a negative impact on our atmosphere. One of the possible methods for restoring sections of steam pipelines after long-term operation is restorative heat treatment (RHT), aimed at improving the microstructure and, consequently, the mechanical properties of degraded steel [26–28]. It is noteworthy that significant effects of the restoration of hardness, strength, and plasticity, as well as a noticeable increase in the impact toughness of 25Kh2M1F steel, which worked for 21×10^4 h in the elements connecting parts of the high-pressure rotor housing of a TPP, were achieved after its additional heat treatment [29]. In papers [30,31], RHT was applied to restore the creep resistance of used 14MoV6-3 steel, which yielded promising results. To restore the steel in use, repeated normalization followed by high tempering was also used, which made it possible to achieve a significant improvement in its microstructure, but it was not possible to completely restore its properties [32,33]. In particular, the hardness and strength of the steel increased, but its fatigue limit decreased. This was explained by the presence of small areas with inclusions in the pearlite and ferrite matrix in the steel microstructure, which did not dissolve during heat treatment. Analysis of the results of using RHT to improve the properties of heat-resistant steels after long-term high-temperature operation confirms the relevance of research aimed at developing a promising method for extending the service lives of elements of expensive large-sized structures. This paper introduces a two-stage heat treatment, unlike that from known approaches using RHT, which apply repeated heat treatment to the operated steel according to the mode designated for its initial state. In particular, the process includes additional normalization at a temperature exceeding the recommended level, followed by a regulated heat treatment of the operated steel.

The aim of this work Is to study the structure and failure mechanism features of 12Kh1MF steel both after long-term operation in the stretched bend zone of a TPP steam pipeline and after additional restorative heat treatment, followed by determining its strength, plasticity, and brittle fracture resistance characteristics to justify the possibility of extending the high-temperature operation of this steel.

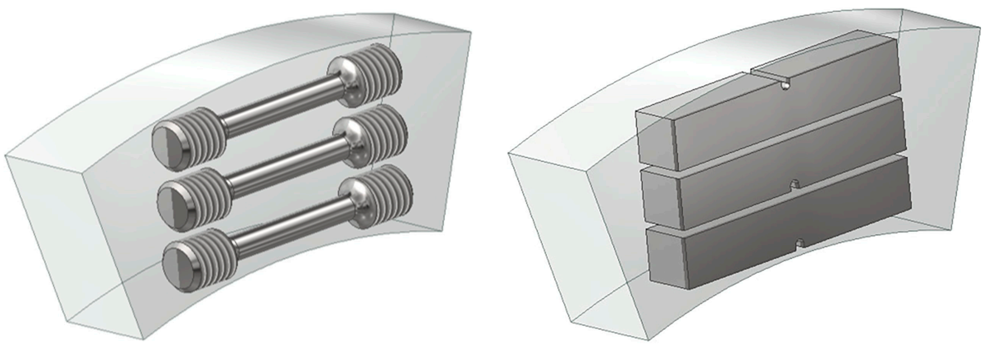
2. Materials and Methods

Heat-resistant 12Kh1MF steel (analogue 14MoV6-3 steel, according to DIN 17175-1979 [34]) was studied after 2.86 × 10^5 h of operation in the vertical bends of the main steam pipelines of a TPP at a temperature of 540 °C and a coolant pressure of 14 MPa. The chemical composition of the steel included the following (wt.%): 0.1 C, 0.22 Si, 0.5 Mn, 0.84 Cr, 0.01 S, 0.0005 P, 0.2 Cu, 0.19 Ni, 0.23 V, 0.28 Mo, Fe—balance. The outer diameter of the pipe on the straight section was 325 mm, and the wall thickness was 38 mm. The bending radius of the studied vertical bends of the steam pipelines was 1370 mm, and the bending angle was 90°. The most favorable force conditions for steel degradation during operation usually occur in the stretched bend zone (SBZ), so the metal of this zone was chosen for the restoration of its properties and structure by heat treatment.

The presence of a gradient of circumferential stresses across the thickness of the pipe wall (especially thermal stresses arising during the process of shutdowns and starting of the unit [35]) also determines a gradient of the mechanical properties of the steel used in the steam pipeline. The mechanical properties of heat-resistant steel after operation in an SBZ are presented in Table 1. They were determined at the following three levels along the pipe wall thickness: near the outer and inner surfaces of the pipe and in the center of its cross-section (Figure 1). Tangential specimens for tensile and impact testing were cut from transverse samples in such a way that their axis passed through the center of the pipe wall section (marked II), placed at a distance of 6–8 mm from the outer and inner surface of the pipe (I and III).

Table 1. The mechanical properties of heat-resistant 12Kh1MF steel after operation in the stretched bend zone of the main steam pipeline of a TPP.

Specimen Location in the Pipe Cross-Section	σ_{UTS}, MPa	σ_{YS}, MPa	Elongation, %	RA, %	KCU, MJ/m^2
Near the outer surface (I)	415	283	13.8	39.3	0.23
In the center of the pipe cross-section (II)	435	293	17.5	50.2	0.65
Near the inner surface (III)	431	303	16.8	48.4	1.13



(**a**) (**b**)

Figure 1. Scheme of cutting tangential samples at three levels of the pipe cross-section in the stretched bend zone for subsequent tensile (**a**) and impact (**b**) tests.

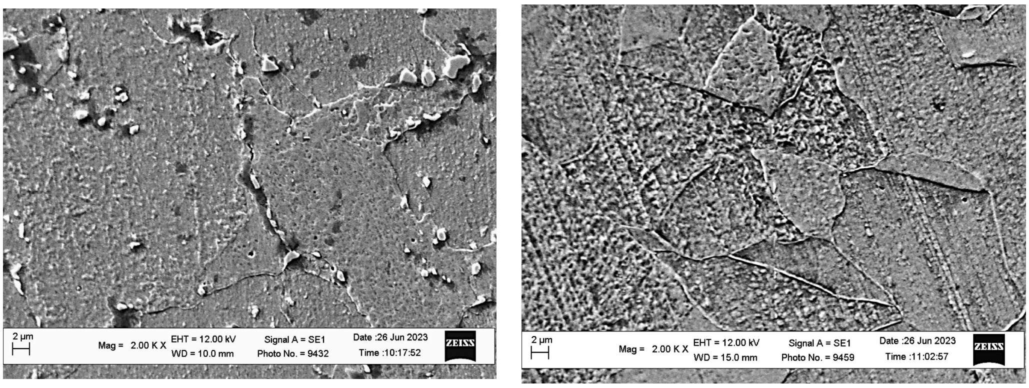
To improve the microstructure and increase the mechanical properties of the long-term-operated 12Kh1MF steel, the RHT mode (two-stage normalization followed by tempering) was proposed. The RHT mode was optimized by varying the holding times of the samples at an austenitization temperature of 1100 °C at the first stage of processing from 30 to 630 min [36]. After all, the holding duration of samples of exploited steel above the critical point Ac_3, where diffusion processes are intensified, predetermines the redistribution of elements in their structures. It is obvious that the homogenization of austenite in terms of carbon and alloying element content also occurs due to diffusion, and this process occurs over time. Therefore, it is believed that the duration of austenitization has a significant effect on the uniformity of the distribution of elements in the matrix and on the sizes of the structural components of the steel structure. For further research, the steel in use was selected, which was treated at a temperature of 1100 °C for 150 min. The samples subjected to the following heat treatment were used in further studies of the effect of RHT on the properties of the operated steel. At the first stage of RHT, the samples of operated steel were held at a temperature of 1100 °C for 150 min and then cooled in air. This was the optimal holding time at a higher austenitizing temperature, which was justified using grain size. Previously, based on metallographic studies and an analysis of the dependence of grain size changes on the duration of the austenitization process of operated steel, the optimal holding time for samples at the austenitization temperature was substantiated [36]. After the first stage of the RHT, the samples were subjected to repeated normalization in accordance with the heat treatment mode recommended for 12Kh1MF steel in the initial state (TU 14-3-460-2009, Ukraine). According to these requirements, at the second stage of the RHT, the samples were normalized by cooling in air after holding them for τ = 30 min at a lower austenitization temperature of 960 °C compared to the first stage of the RHT, and they were then tempered after holding for τ = 180 min at a temperature of 740 °C. The restoration of steel previously operated for a long time in the stretched zones of steam pipeline bends [24] was carried out according to a justified RHT mode on tangentially oriented templates with a thickness of 12 mm. Both steel variants (after operation and additional heat treatment) were compared in terms of hardness HB, impact toughness KCU, strength characteristics (ultimate tensile strength σ_{UTS} and yield strength σ_{YS}), and ductility (elongation and reduction in area RA). The hardness of both steel variants was determined on sections with plane-parallel planes along the pipe wall thickness in the SBZ as an average value of at least 10 measurements at one level. To determine the impact toughness, Menage-type samples (10 mm × 10 mm × 55 mm) with a U-shaped notch 2 mm deep and 1 mm in radius were used. Impact tests on the samples were carried out on an IO-5003 pendulum impact tester following ISO 148-1:2022 [37]. The mechanical properties of the steel under tension were determined in cylindrical samples with a diameter of 5 mm and a working length of 25 mm. Tensile tests were carried out on an UME-10T testing machine in accordance with ISO 6892-1:2019 [38], ISO 7500-1:2018-02 [39], ISO 9513 [40]. The deformation rate of the samples was 10^{-3} s^{-1}. All samples were carefully polished before testing using polishing materials and pastes of varying grain sizes. All mechanical properties were determined by averaging the test results of at least three samples of each category, and their deviations from the average value did not exceed 3–5%.

A microstructure analysis of the steel after long-term operation and RHT was carried out. For its detection, multiple alternating polishing and etching procedures for the specimens were used, with a 2–4% solution of nitric acid in alcohol. The grain size distribution was utilized as an indicator of the structural states of the two steel variants (operated and restored using the proposed heat treatment mode). They were determined by available computer processing methods applied to SEM images of the steel structures obtained at various levels along the pipe wall thickness. Fractographic images of the fracture surfaces of the tensile-tested specimens, as well as microstructure images of both steel variants, were used to determine the number of grains of a certain size category near the outer and inner surfaces of the pipe, as well as in the center of its cross-section, and were obtained using an EVO 40XVP (ZEISS, Oberkochen, Germany) scanning electron microscope.

3. Results

3.1. Distribution of Grains with Different Sizes Along the Pipe Wall for Both Steel Variants

The changes in the number of grains of a certain size in the structure of the 12Kh1MF steel, long-term-operated in the SBZ of the steam pipeline and after RHT, were analyzed (Figure 2). All analyzed grains were conditionally divided into the following three size categories: small (<10 μm), medium (10 to 30 μm), and large (>30 μm). The number of grains in each category was determined by averaging the grain size values measured in the structure images at 20 points at each level in the pipe wall cross-section.



(**a**) (**b**)

Figure 2. Typical microstructure of 12Kh1MF steel (**a**) after long-term operation in the stretched bend zone and (**b**) after restorative heat treatment.

The percentages of grains belonging to certain size categories in the structure of the operated steel varied unevenly along the pipe wall thickness (Figure 3a). In particular, the proportions of small and medium grains gradually and slightly increased from the outer to the inner surface of the pipe. Thus, the number of medium-sized grains in percentage terms was the smallest near the outer surface of the pipe (44%) and the largest near its inner surface (53%). In the center of the pipe section, the number of grains with sizes from 10 to 30 μm reached 49%. As for large grains, their amount changed ambiguously. Their amount in percentage terms was the largest near the outer surface of the pipe (48%), compared with their amount in the center of the pipe wall (39%) and near its inner surface (44%). Such an ambiguous change in the number of grains with the maximum size (D > 30 μm) along the thickness of the pipe wall in the operational state indicated that these grains were responsible for the increased tendency of the operated steel to creep. After all, the force conditions for creep were also most favorable near the outer surface of the pipe. In addition, the energy capacity of dislocation pileups at their boundaries increased with an increase in grain size. This facilitated the penetration of dislocations through these boundaries into neighboring grains.

After the RHT of the operated steel, the proportion of small grains increased with a simultaneous decrease in the proportion of medium and large grains throughout the entire thickness of the pipe wall (Figure 3b). The proportions of grains with the smallest size (D < 10 μm) were 55%, 56%, and 51% near the outer and inner surfaces of the pipe and in the center of its cross-section, respectively. The percentage of medium grains (10 to 30 μm) near the outer and inner surfaces of the pipe was slightly lower (36 and 35%, respectively) than in the center of the pipe cross-section (39%). The proportion of large grains after RHT remained virtually unchanged across the pipe wall thickness (varying within 9–10%). It was expected that such a significant increase in the amount of the smallest grains across the pipe wall thickness following RHT had a positive impact on the steel's mechanical properties.

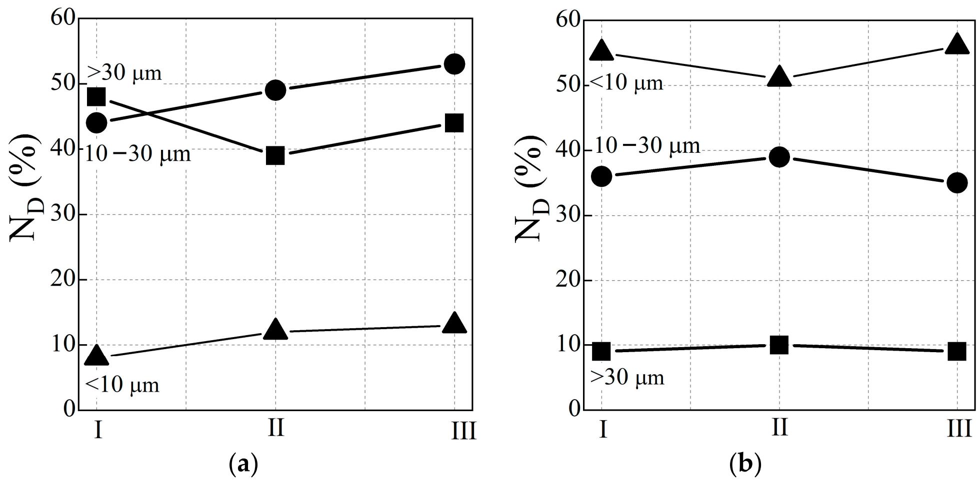


Figure 3. The proportion of grains N_D of a certain size category relative to their total number in the structure of 12Kh1MF steel from the stretched bend zone of a pipe after its long-term (2.86 × 10^5 h) operation (**a**) and restorative heat treatment, according to the optimal mode (**b**) determined near the outer (I) and the inner (III) surfaces of the pipe, as well as in the center of its cross-section (II).

Since the proportion of fine grains, as a key structural indicator of steel condition, increased after the RHT, a significant rise in hardness was also expected. Hardness measurements showed that, after operation, its value in the center of the pipe section did not exceed 130 HB, while near both its surfaces, the hardness decreased to 120 HB (Figure 4a). Neither of the two hardness values obtained for the long-term service steel met the standard industry requirements [41]. At the same time, the application of the optimal mode of RHT to this steel made it possible to increase its hardness to 170 HB, indicating the restoration of its serviceability for this indicator (Figure 4a).

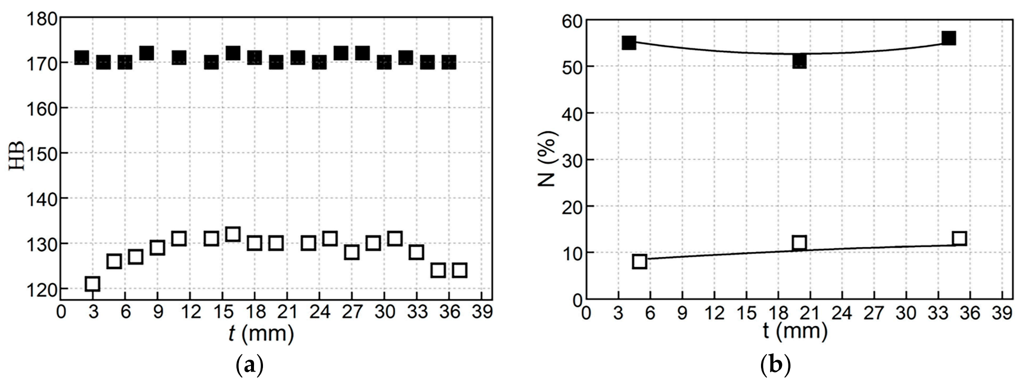


Figure 4. Change in the hardness HB (**a**) and the proportion of fine (≤15 μm) grains of N in the structure of 12Kh1MF steel (**b**) across the pipe wall thickness t of the stretched bend zone after its operation for 2.86 × 10^5 h on the main steam pipeline of a TPP (□) and after additional application of restorative heat treatment to it according to the optimal mode (■).

In our opinion, the established increase in steel hardness was due to a significant increase in the proportion of small grains relative to large ones in the steel structure. Our measurements showed that, after RHT, the proportion of small grains increased over the entire thickness of the pipe wall by more than five times (Figure 4b). The energy costs for the penetration of dislocation pileups through grain boundaries directly depend on both their size and the number of such boundaries. Therefore, such a significant decrease in grain size and, as a consequence, an increase in the length of their boundaries, which dominated in the steel structure after RHT, were considered as an argument for the validity of positive expectations for steel recovery.

Since both grain size and hardness values can be measured directly on the outer surfaces of pipes during scheduled inspections, monitoring the changes in these parameters during operation will make it possible to solve some important problems in ensuring the operability of the steam pipeline system. Firstly, such measurements create a basis for the non-destructive monitoring of the current technical condition of the heat-resistant steels used long-term in steam pipelines of TPPs. Secondly, such measurements make it possible to identify critically degraded elements of steam pipelines that require restorative heat treatment. Thirdly, these measurements make it possible to verify the effectiveness of the proposed RHT mode during pilot industrial tests using industrial frequency currents of 200 Hz to heat the pipes. Finally, such an approach will provide an accessible method for monitoring the stability of the state of steam pipeline elements restored by the proposed RHT method during their further operation, which is important for confirming the validity of the RHT application.

3.2. Comparison of Mechanical Properties of Long-Term-Operated 12Kh1MF Steel Before and After RHT

Tensile tests of samples from restored and operated steel revealed increases in both strength characteristics (σ_{UTS} and σ_{YS}) after RHT (Figure 5). Moreover, the effect of RHT on σ_{UTS} was even greater than that on σ_{YS}. The increases in these indicators were observed at all three levels of analysis by pipe wall thickness. In particular, the maximum increases in the σ_{UTS} and σ_{YS} characteristics after RHT reached 18.5 and 11%, respectively. These were recorded on samples cut near the outer surface of the pipe. Thus, the application of the optimal RHT mode confirmed its effectiveness in the restoration of 12Kh1MF steel for both strength indicators. It should be noted that, near the outer surface of the pipe, there was a barely noticeable tendency for greater increases in both strength indicators compared to near its inner surface. This is important, since the most favorable conditions for creep and, as a consequence, the most intense degradation of steel usually occur near the outer surface of the pipe. However, these negative consequences of the long-term operation of steel in a steam pipeline were successfully overcome after the use of RHT. This may be a weighty argument considering the question of the feasibility of extending the service life of steel in critical elements of the main steam pipelines of TPPs.

As for the plasticity characteristics (elongation and RA) of the long-term operated 12Kh1MF steel, an even more pronounced gradient of both characteristics across the pipe wall thickness was revealed after steel restoration (Figure 5). The maximum increases in both indicators after RHT were also recorded near the outer surface of the pipe. In particular, the value of elongation increased to 50% and RA reached 60% compared to the corresponding characteristics of the operated steel. The RHT effect in the center of the pipe cross-section and near its inner surface was significantly smaller. The increases in elongation and RA were only 28 and 38%, respectively. Based on this, it was concluded that the RHT largely eliminated the negative consequences of the long-term operational degradation of 12Kh1MF steel in the SBZ of the steam pipeline. This increases confidence in the validity of the proposed processing mode as an effective method for increasing the plasticity characteristics of operated steel. This concerns zones with the most favorable creep conditions, since a sufficient reserve of steel plasticity is a necessary condition for extending the service lives of long-operated steam pipeline elements.

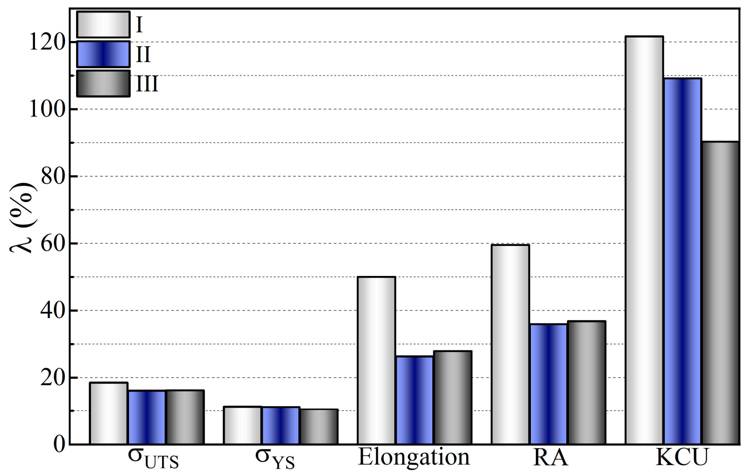


Figure 5. Change in mechanical properties (in percentage terms λ) for 12Kh1MF steel at different levels along the pipe wall thickness (I and III—near the outer and inner surfaces of the pipe; II—in the center of its cross-section) after its restorative heat treatment in comparison with the corresponding properties of this steel after 2.86×10^5 h of operation in the stretched bend zone of a TPP steam pipeline.

It was established that the resistance to brittle fracture of the steel was found to be the most sensitive to changes in the condition of the material after its restoration (Figure 5). The positive effect of applying RHT to the operated steel exceeded 120% near the outer surface of the pipe and decreased to 90% near its inner surface. Thus, it is obvious that RHT did not eliminate the gradient of resistance to brittle fracture across the pipe wall thickness. As a consequence, a noticeable decrease (up to 30%) in the positive effect of steel restoration from the outer to the inner surface of the pipe was also observed for this indicator. A possible explanation for this result may be the very low value of the impact toughness of long-term operated steel (not corresponding to the requirements of the current regulatory document), relative to which, the positive effect of restoration was actually assessed. This value was more than four times lower near the inner surface of the pipe and over two times lower in the center of its section, as shown in Table 1. Thus, a more than two-fold increase in the brittle fracture resistance of the restored steel was obtained throughout the entire thickness of the pipe wall. However, the increase in its KCU values relative to the corresponding values for operated steel at each level in the pipe cross-section was greatest near the outer surface of the pipe, where the steel degradation was highest. In any case, a significant reduction in the sensitivity of the operated steel to impact loads after its restoration ensured compliance with regulatory requirements, which provides prospects for the expanded use of RHT in the practice of servicing the steam pipelines of TPPs.

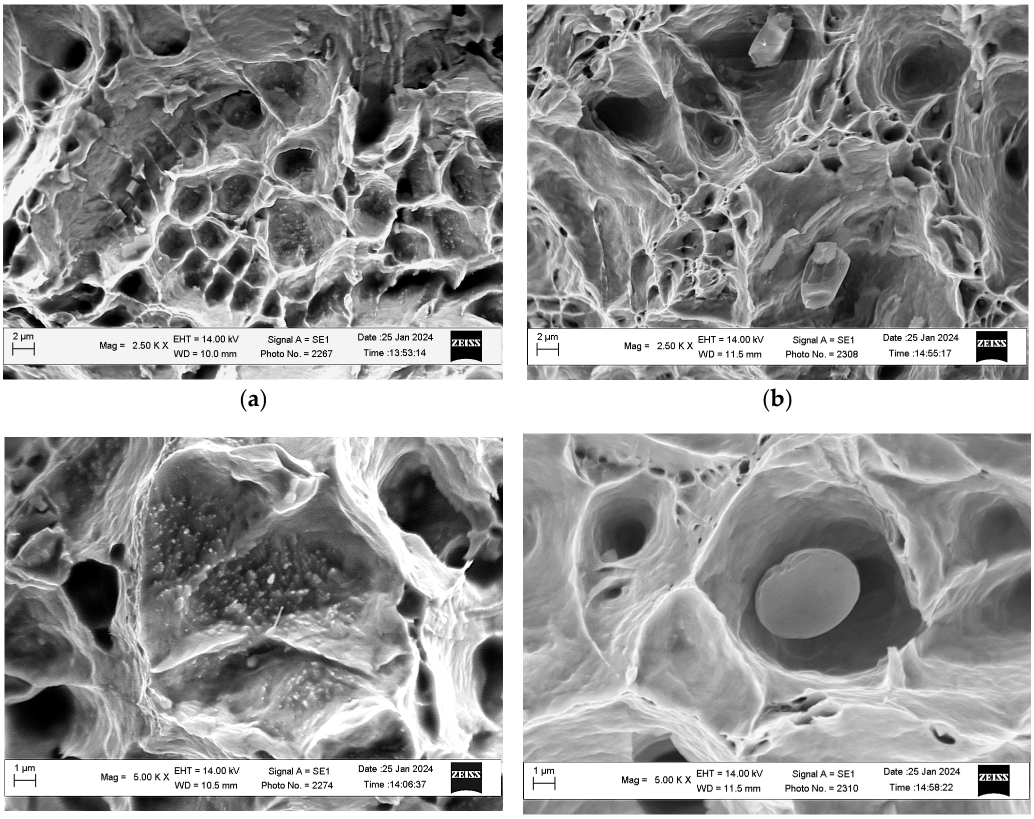
Thus, by applying RHT to the exploited steel, it was possible to effectively improve the microstructure of the steel (by reducing the size of the dominant grains) and increase its mechanical properties (hardness, strength, and plasticity characteristics, as well as resistance to brittle fracture). All the described signs of improvement in the operated steel after RHT justify the legitimacy of using the proposed mode for the restoration of operated steel. In a case where the hardness of long-operated steel, measured on the outer surface of a pipe, approaches unacceptable values, the use of the proposed RHT method will make it possible to extend the service lives of elements made of this steel.

3.3. Fractographic Features of Samples Made of Operated and Restored 12Kh1MF Steel Tested for Tension

An analysis was carried out on fractures of tangentially oriented samples cut near the outer and inner surfaces of the SBZ of the main steam pipeline of the TPP made of

12Kh1MF steel. The fracture features of steel that had worked on the steam pipeline for about 2.86 × 10^5 h and that had undergone RHT after operation under the same conditions were compared.

In the central parts of the fractures of the samples, where the initiation of their failure under uniaxial tension usually occurs, ductile dimple relief prevailed, and this mechanism dominated regardless of the location of the samples near the outer or inner surface of the pipe in the SBZ. However, on fractures of samples cut near the outer surface of the pipe, dimples grouped into conglomerates were quite often observed. Ridges with signs of a shear mechanism were formed along the perimeters of these areas (Figure 6a). The presence of such shear ridges indicated the formation of voids on carbides and non-metallic inclusions near the outer surface of the pipe even before tensile testing. The bridges between such individual voids were destroyed during the tensile testing of the samples due to the mechanism of their stretching until they broke, thereby forming a larger cavity. Merging with similar cavities formed by other nearby void conglomerates occurred due to a shear in the bridge between them. In contrast, the fractures of samples cut near the inner surface of the pipe had a uniform ductile relief characterized by uniform equiaxed dimples (Figure 6b). Similar results for steels exposed to high temperatures were obtained in other works [42].



(**a**) (**b**)

(**c**) (**d**)

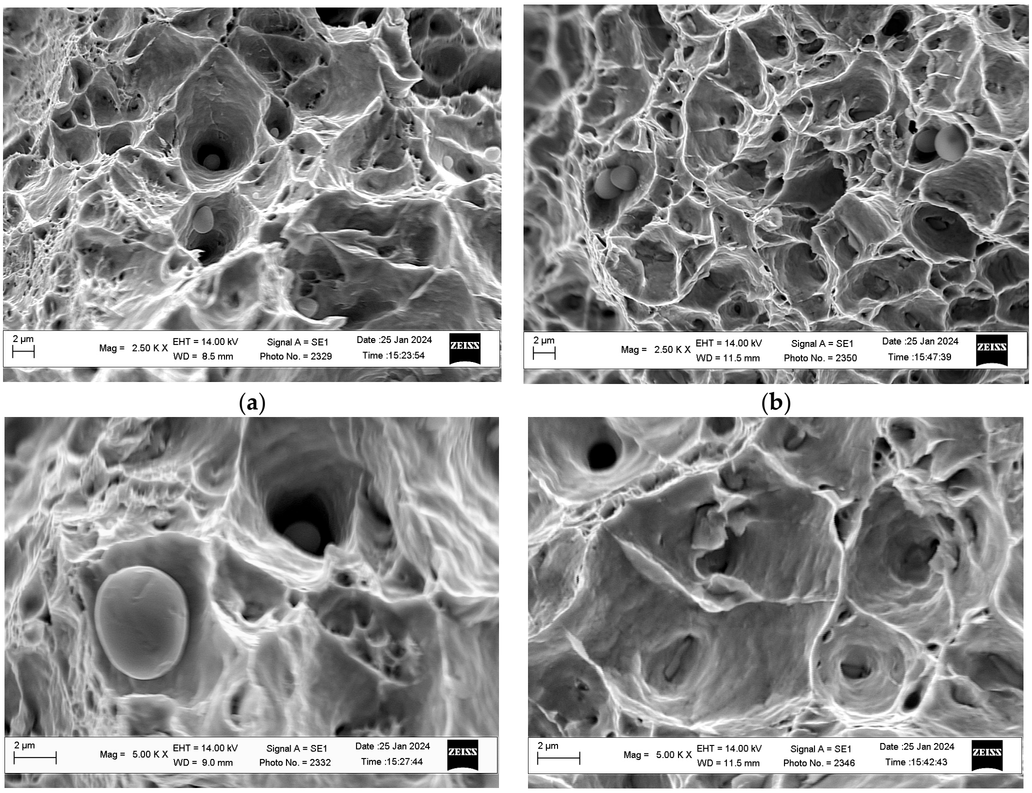
Figure 6. Microfractograms of the central part of the fractures of tangentially oriented tensile specimens taken near the outer (**a**,**c**) and inner (**b**,**d**) surfaces of a pipe made of 12Kh1MF steel that operated for 2.86 × 10^5 h in the stretched bend zone of the main steam pipeline of a TPP.

Non-metallic inclusions or carbides were typically detected at the bottom of most dimples on the ductile fracture surfaces of non-exploited steels. Similar inclusions (as a source of microvoids) were also found in the fractures of operated steels. The stretching of the bridges between such voids until their destruction led to the formation of ridges at the fractures, separating adjacent dimples from each other. Most carbides or non-metallic inclusions in the non-exploited steels usually remained cohesive with the matrix and were easily detected at the bottom of most dimples after the tensile testing of the specimens. However, during the long-term high-temperature operation of the steels, when favorable conditions for creep existed, inclusions and carbides in the steel structure gradually lost cohesion with the surrounding matrix. As a result, the force holding them at the bottom of the dimples disappeared, and the probability of their detection at the fractures of tensile-tested samples also decreased. For the first time, this feature of the high-temperature degradation of steels was identified based on a comparison of fractures of weld metal samples both in the initial state and after long-term operation on a steam pipeline of a TPP [43].

Fractographic signs confirming this phenomenon were also found on fractures of the used 12Kh1MF steel. In particular, at the bottom of the dimples on the fractures of samples cut near the outer surface of the pipe, a significantly smaller number of inclusions were found than on samples from the vicinity of its inner surface. It was believed that this fractographic feature indicated a more intense degradation of steel near the outer surface of the pipe due to the loss of cohesion of carbides and especially large non-metallic inclusions with the surrounding matrix (Figure 6). Additionally, at the tops of the ridges along the boundaries of the large dimples on the fractures of the steel operated near the outer surface of the pipe, there were no much smaller dimples with signs of the stretching of the bridges between them (Figure 6a,c). On the contrary, on the fractures of samples from the same pipe but cut near its inner surface, a noticeable feature of relief was chains of small equiaxed dimples. They were formed under the influence of normal tensile forces and located along the tops of the ridges, outlining larger dimples (Figure 6b,d). This, in itself, indicated a greater plasticity in the steel near the inner surface of the pipe. After all, the bridges between large dimples at least partially retained the ability to stretch during the tensile tests, instead of breaking due to shear.

It was also noted that a characteristic feature of the fractures, indicating the more intense degradation of the 12Kh1MF steel near the outer surface of the steam pipe, was the absence of large inclusions at the bottom of large dimples. Instead, only clusters of very small (nanosized) particles (no more than 300 nm) were observed at the bottom of the large dimples (Figure 6a,c). It was assumed that these clusters were formed by micro fragments of large carbides and inclusions that remained at the fractures after the final separation of large particles from the matrix during the tensile testing of the samples. In contrast, on the fractures of samples cut near the pipe's inner surface, traditional inclusions and carbides of various shapes and sizes were found at the bottom of the dimples (Figure 6b,d). It was suggested that a significant portion of the large carbides that were visible and coagulated at the grain boundaries during long-term high-temperature steel operation still retained at least partial cohesion with the ferrite matrix. It was believed that the nanoscale fragments at the bottom of the large dimples on the fractures of samples cut near the outer surface of the pipe were traces of their final decohesion. Their final separation from each other occurred during the tensile testing of the samples. As a result, against the background of a generally typical ductile fracture relief, nanosized particles in the form of fragments of large carbides were preserved at the bottom of large dimples. It was noted that they could be visualized only at a high magnification.

As for the fractographic features of the restored steel, conglomerates of dimples united by common shear ridges, characteristic of exploited steel, were not found in its fractures. In addition, the relief of ductile dimples at the fracture surface of the restored steel was higher and more pronounced than on the fracture surface of the exploited steel. This indicated that more energy was spent on their formation. Thus, the distinctive feature of steel degradation under operating conditions (the presence of large dimples with nanosized particles at the bottom, separated from large carbides during the tensile testing of the samples) was not detected at the fractures after using the proposed RHT mode (Figure 7).



(**a**) (**b**)

(**c**) (**d**)

Figure 7. Microfractograms of the central part of fractures of tangentially oriented samples of 12Kh1MF steel cut near the outer (**a**,**c**) and inner (**b**,**d**) surfaces of an operating pipe and tested for tension after applying restorative heat treatment according to the proposed mode.

On the fracture surface of steel subjected to RHT, small equiaxed dimples predominated. They were formed by the mechanism of the stretching of bridges between adjacent voids up to their rupture. It was believed that the small (tenths of a micron) carbides observed at their bottom were precipitated during the high-temperature tempering of steel at the final stage of its RHT. The predominance of dimples on the fractures of the restored steel, formed due to the mechanism of the stretching of bridges between adjacent voids before their rupture, indicated that the role of the shear mechanism (which played a significant role in the formation of the dimple relief on the fractures of the operated steel) was practically levelled out after RHT. Additionally, clusters of small dimples caused by the destruction of finely dispersed pearlite were found in the structure of the restored steel (Figure 7). Small particles of cementite within the pearlite grains were responsible for the nucleation of these small dimples directly during the tensile testing of samples. On the contrary, large and deep dimples were found inside the ferrite grains, visible against the background of small dimples surrounding them. The presence of large dimples on the fractures of the restored steel was associated with inclusions that did not dissolve at the initial stage of RHT (normalization from 1100 °C). Their preservation in the structure of the restored steel was due to a significant (and often complete) loss of cohesion of large carbides (or non-metallic inclusions) to the matrix due to the creep of the steel during long-term high-temperature operation in a steam pipeline. As a result, their dissolution became difficult (due to an insufficient austenitization temperature) or even impossible (due to the loss of diffusion paths). The defects formed due to the decohesion of the matrix and large carbides at the grain boundaries remained virtually unchanged after the RHT of the steel. However, due to the recrystallization of the steel during high-temperature austenitization, most of them were already located inside the newly formed grains and not along their boundaries. The tensile testing of a sample was accompanied by the plastic deformation of the matrix surrounding operational defects. Large inclusions inside them either remained at their bottom if they were deep enough (Figure 7c,d) or were absent there if the depth of the defects was insufficient to retain the inclusion during deformation.

Thus, all the obtained results showed that, even after the long-term use of 12Kh1MF steel, it was possible to extend its service life by applying RHT. Considering the differences in the strength of the steel near the outer and inner surfaces of the SBZ of the steam pipeline of the TPP, as well as the corresponding difference in the degree of the degradation of steel due to different creep conditions, it can be hoped that the use of RHT can be useful for the restoration of steel at different stages of its operation in steam pipelines. Its application will be especially effective for steels whose structural transformations have not yet reached a critical level and whose complete decohesion matrix and carbide coagulation along the grain boundaries have not yet occurred. In addition, the results obtained from the application of the proposed RHT mode to operated steel give hope for extending the service lives of the elements of steam pipelines of TPPs and show the possibility of delaying their final decommissioning. However, such a perspective requires a much broader experimental justification for the feasibility of this approach. The results of our experimental research only demonstrate that the window of opportunity is open. However, for the final implementation of the demonstrated positive effect of RHT in the practice of servicing TPP equipment, multilateral checks performed on steels with different degrees of degradation and using various research methods will be required.

4. Conclusions

To substantiate the applicability of the restorative heat treatment of heat-resistant 12Kh1MF steel after 2.86×10^5 h of operation in the zone of the stretched bend of the main steam pipeline of a thermal power plant, metallographic and fractographic studies, hardness measurements, and mechanical tensile and impact tests were used. The main results of these studies were as follows:

1. To restore the structure and mechanical properties of long-term operated 12Kh1MF steel, a two-stage heat treatment was proposed. Before using the traditional normalization mode from 960 °C with tempering at 740 °C, provided for by the regulatory document for this steel in its initial state, it was pre-normalized from a temperature of 1100 °C.
2. In the structure of the restored steel, the proportion of large grains (more than 30 μm size) decreased to 10% and the proportion of small grains (less than 10 μm in size) increased to 55%, while in the exploited steel, their proportions were 48 and 8%, respectively.
3. After applying the proposed restorative heat treatment mode, the mechanical properties of the long-term exploited 12Kh1MF steel from the stretched bend zone of the TPP steam pipeline increased throughout the entire cross-section of the pipe. In particular, its hardness HB increased by almost 40%, ultimate strength by 19%, and RA by 60%. At the same time, the impact toughness of the restored steel increased more than twofold. In addition, all the obtained characteristics exceeded their regulated values, which can be considered as convincing arguments to justify the legitimacy of using the proposed restorative heat treatment regime to extend the service life of 12Kh1MF steel in critical elements of thermal power plant steam pipelines.
4. Fractographic studies of tensile samples from both types of steel (in service and restored) showed the dominance of the ductile dimple mechanism of their fracture.

A feature of the operated steel was nanosized particles (up to 300 nm) found at the bottom of large dimples. They were considered to be fragments of large carbides that retained a partial connection with the matrix even after the long-term operation of the steel, breaking only during tensile testing of the sample. At the same time, small carbides were observed at the bottom of most of the dimples on the fractures of the restored steel, indicating that their cohesion with the matrix was maintained during the destruction of the sample. The absence of large carbides and pores along the grain boundaries with the simultaneous presence of large dimples with deep traces of carbides (not dissolved and moved inside the newly formed grains during steel restoration) were also considered as signs of the transformation of the steel structure during restoration.

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Nomenclature

RHT	Restorative heat treatment
TPP	Thermal power plants
SEM	Scanning electron microscope
SBZ	Stretched bend zone
KCU	Impact strength
D	Grain size
HB	Hardness
RA	Reduction in area
σ_{UTS}	Ultimate strength
σ_{YS}	Yield strength
N_D	Number of grain sizes
t	Pipe wall thickness

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