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МАТЕРІАЛОЗНАВСТВО

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SOFTENING OF HARDENED MEDIUM-CARBON STEEL DURING HEATING

Purpose. The work is aimed to clarify the softening mechanism during the heating of martensite hardened carbon steel, which is of practical importance, especially in the development of the production technology of rolled products with different levels of hardening. **Methodology**. The samples after martensite hardening were tempered at the temperatures of 300-500˚C. The microstructure was investigated under the electron microscope. Thin foils were made using the Bolman and tweezer methods in chlorous-acetic solution and Morris reagent. Phase distortions of crystalline lattice were determined by the methods of X-ray structural analysis, using the diffractometer. The cold-worked layer of metal after grinding was removed by electrolytic dissolution. Tensile strength brake of the metal was determined using the tensile diagrams of samples using the Instron type machine. Microhardness was measured using the PMT-3 device with indentation load 0.49 N. **Findings**. When heating the hardened steel to a temperature of 300˚C, the softening effect is mainly related to the rate of reduction of the accumulated as a result of martensitic transformation, density of the crystalline structure defects. The total result is caused by the development of dislocations recombination and strengthening because of the emergence of additional number of cementite particles during the martensitic crystals decomposition. Starting from the heating temperatures of 400˚C and above, the development of polygonization processes in the ferrite is accompanied by the emergence of additional sub-boundaries, which enhance the effect of metal strengthening. With increase in the heating temperature of the hardened steel, the level of strength properties is determined by the progressive softening from the decrease in carbon atoms saturation degree of the solid solution, dislocations density and increase in the size of cementite particles over the effect of strengthening from hindering of mobile dislocations by carbon atoms and the emergence of additional sub-boundaries. **Originality**. For the tempering temperature of 300-400˚C, the absence of the phase distortion change indicates the emergence of additional factor in strengthening the metal from the formation of sub-boundaries and the dispersion strengthening from the carbide particles. **Practical value**. The given explanation of the mechanism of structural transformations in the process of tempering in the average temperature range of the hardened carbon steel can be used to optimize the technology of thermal strengthening of rolled metal.

Keywords: microstructure; martensite; cementite; sub-boundaries; hardening; softening

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Introduction

During the thermal hardening using the heat of rolling heating [2, 4, 10], in the elements of products of large sections a gradient of structures is formed, which correspond to certain values of the strength characteristics [1, 9, 11]. The observed structural inhomogeneity is caused by the cumulative effect of the development of phase transformation processes with different nature of influence on the properties. Thus, the achieved level of metal hardening is proportional to the speed of accelerated cooling, which decreases as the distance from the cooled rolling surface increases [5]. At the same time, the heating temperature of the metal will increase when approaching the central volumes [1, 4]. For the detailed analysis, the continuous nature of the structure change across the section of the rolled product, depending on the distance from the cooling surface, can be conditionally divided into separate volumes with corresponding structure and ability to work hardening [3]. Based on the above-mentioned, the changes in the nature of behaviour when loading the specified volumes of metal will be similar to the influence of the temperature of individual heating when tempering the hardened steel [1]. Takin into account the additive nature of influence of the dispersion and morphology of the structural components on the general level of carbon steel hardening [3], the formation of certain gradient of structures across the rolled product section should be considered as one of the directions of technology optimization to achieve the required structural state and the level of strength characteristics [1, 3, 5].

Purpose

The work is aimed to clarify the mechanism of softening during the heating of the hardened medium-carbon steel in the average temperature range.

Methodology

The fragments of the railway wheel set axis made of carbon steel with the concentration of chemical elements: 0.41% C, 0.36% Si, 0.61% Mn, 0.0022% S, 0.015% P served as a material for the research. The plates 2 mm thick, 20 mm wide and 250 mm long were the test samples. The samples were austenitized at temperatures higher than Ac_3 followed by forced cooling in a water tank. After hardening, the samples were subjected to tempering at temperatures of 300-500°C during 1 h. The heating was carried out in the SNOL electric furnace – 1.6.2.5.1/11-IZ. The temperature was controlled by the chromel-alumel thermocouple and DC potentiometer. The microstructure of the samples was studied under electron microscope at accelerating voltage of 100 kV. Thin foils for the research under electron microscope were made using the Bolman and tweezer methods in chlorous-acetic electrolyte and Morris reagent. During the manufacture of the objects for X-ray studies, the cold-worked layer of metal, after making the polished sections were removed by dissolution in the chlorous-acetic electrolyte. The phase distortions of crystalline lattice ($\Delta a/a$), where a is the ferrite lattice parameter, were determined by the methods of X-ray structural analysis using the DRON-2.0 diffractometer at scintillation registration of reflexes, in monochromatic CuK_α radiation, at room temperature. The error in the determination $\Delta a/a$ was up to 5%. Tensile strength brake of the metal was evaluated under tension using the Instron type machine, at room temperature and deformation rate of $10^{-3}\,s^{-1}$. As a characteristic of the strength of metal microvolumes, the microhardness was used, which was measured using the PMT-3 device with indentation load 0.49 N.

Findings

The level of the values of tensile strength break of the studied steel after hardening and tempering at the temperatures of 300-500˚C corresponded to the known experimental data for the steels with similar chemical composition [1, 5, 7]. As the tempering temperature increases, well-defined structural changes, detected starting from the temperatures of 200-250˚C, correspond to the progressive metal softening [3, 9]. Indeed, as shown in the works [6, 8] for carbon steels with a carbon content of about 0.4%, the temperature of the beginning of martensitic transformation is about 300°C. This temperature should be enough

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to start the emission of cementite particles from the solid solution during the formation of the martensitic crystal itself. Thus, depending on the temperature of the end of forced cooling, a different combination in the development of the processes of hardening and softening in steel, determines the final level of strength properties.

The effect of hardening from the ordering of carbon atoms in the crystalline lattice of the martensitic crystal [6] and quenching aging [2, 10], when tempering at 300°C, is almost completely suppressed by the progressive metal softening (Fig. 1, *a*).

Indeed, for steels with medium carbon content, the degree of supersaturation of the solid solution achieved during the martensite hardening determines the resource of maintaining the increased metal strength after heating to certain temperatures. Based on this, it is safe to believe that for the studied steel after hardening, the effect from reducing the tetragonality degree of the ferrite crystalline lattice (martensitic crystal) during low-temperature tempering can be to some extent compensated by the increasing role of quenching aging processes [2, 10]. However, already after heating of hardened steel, starting from 300°C, the monotonous nature of the decrease in strength characteristics indicates a definite excess of softening effect over hardening (Fig. 1, *a*). A qualitatively different nature of the dependence on tempering temperature is observed for the value of the phase distortions (Fig. 1, *b*). A comparative analysis of the absolute values of these characteristics, depending on the structural state of the metal, showed that as a result of tempering at 400°C, the H_μ reduction reaches about 40-43%, and for $\Delta a/a$ up to 20% of relatively hardened state. After tempering at 500°C, the values of the corresponding characteristics are 58 and 30%, respectively. Thus, the observed differences in the nature of changes of H_μ and $\Delta a/a$ at tempering up to 400°C (Fig. 1) can be associated only with different sensitivity to sub-structural changes during tempering of the hardened steel. It is hoped that the analysis of the microstructure of hardened steel after tempering at 400°C made it possible to explain the nature of change of $\Delta a/a$.

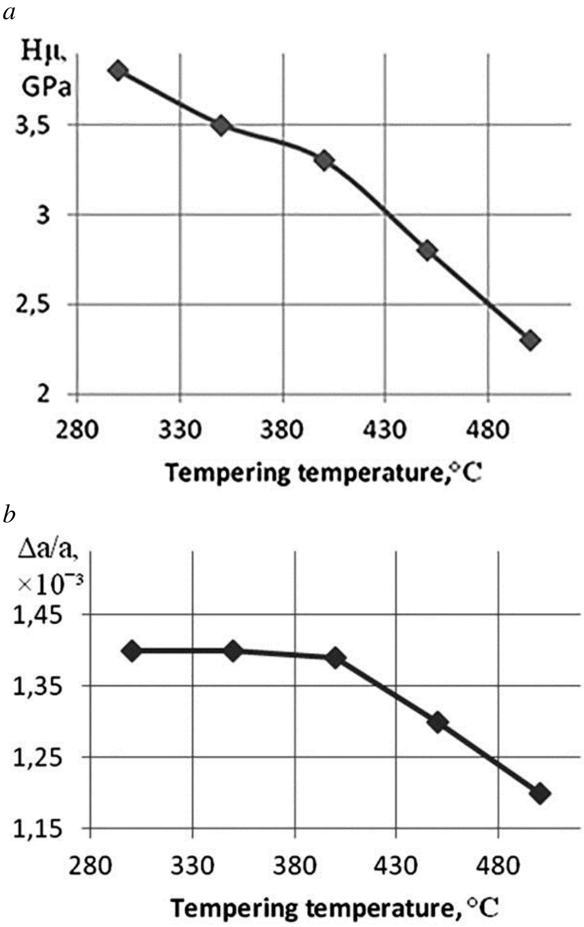

Fig. 1. Influence of tempering temperature on micro hardness (*a*) and micro distortions (*b*) of the hardened steel

The microstructure presented in Fig. 2, *a* corresponds to the hardened state, and in Fig. 2, *b* – to the state after tempering at a temperature of 400°C. The detected sections of the lath martensite with a high dislocation density (Fig. 2, *a*) indicate the development of austenite transformation according to a shear mechanism. The laths width is generally up to 1 micron.

At sufficiently careful study, in separate martensitic crystals, simultaneously with thin twins (designation 1), stroked randomly oriented cementite emissions of high dispersity (designation 2) were found, Fig. 2, *a*. Analysis of the results [2, 8] indicates that the emergence of the observed emissions of the carbide phase is in fact caused by the development of the decomposition processes of martensitic crystals, which are formed at a relatively high transformation temperature.

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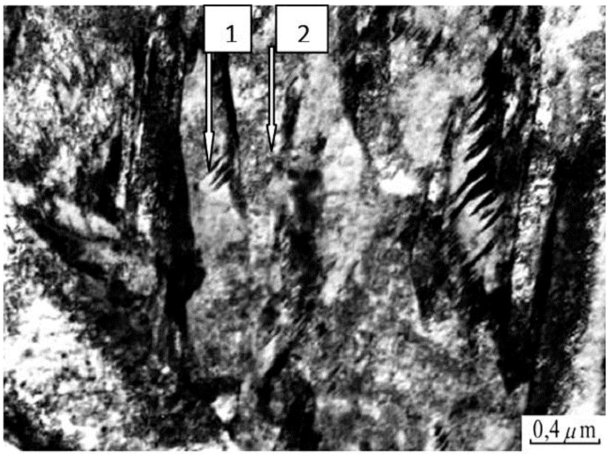
a



b

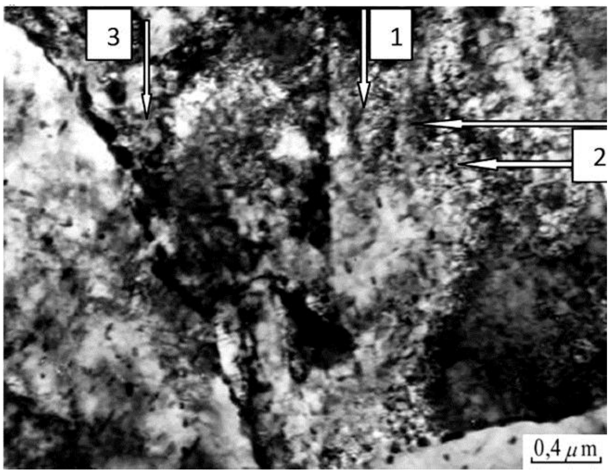


Fig. 2. Microstructure of steel after hardening (*a*) and tempering at 400˚C (*b*)

As compared to H_μ, the absence of the decrease $\Delta a/a$ indicates the complex change in the ratio in the development of the softening and hardening processes of the metal. In the first approximation, the hardening effect is provided by the simultaneous influence of the formation of additional interfaces in the ferrite (sub-boundaries) and dispersion hardening from the emitted carbide particles (Fig. 2, *b*). Considering the influence of cementite particles on the hardening effect separately, it should be noted that in addition to the carbide particles formed during steel hardening, the influence from the emitted particles even when tempering of the hardened steel is added. As follows from the works [6, 8], simultaneously with the emission of the dispersed particles during tempering, the processes of spheroidization and coalescence for the particles that are formed during martensitic transformation receive a certain acceleration. Thus, the expected total hardening effect from the dispersed particles of cementite, which are formed as a result of tempering, will in fact be somewhat reduced. However, in fact, based on a comparative analysis of the microstructure (Fig. 2), no evidence of the existence of carbide particles with markedly increased sizes was found. At a sufficiently careful study of the structure, the particles of cementite located at the sub-boundaries (formed during tempering) and in the internal volumes of the former martensitic crystals (hardening precipitation) (Fig. 2, *b*, designation 1) have almost identical diameters. On the basis of the known results of the study, the permanence of particle sizes is explained by the cyclical nature of the change in the stages of particles growth, when they are located at the grain boundaries due to the dissolving particles located inside of the grain. Thus, despite the fact that at heating temperatures up to 400 ˚C, the process of carbon atoms emission from the tetragonal insertion positions is almost completed, as evidenced by the decrease in the blurring of reflections in microdiffraction photographs and the broadening of x-ray interferences [2, 6]. The insufficient acceleration of cementite coalescence is one of the reasons for a certain decrease in the rate of steel softening. The presence of unessential anomaly on the monotonous course of the curve H_μ (Fig. 1, *a*) confirms these positions.

With a further increase in tempering temperature, the sub-structural changes observed at 400°C receive additional acceleration. As follows from the microstructure analysis (Fig. 3), the heating to 500°C is already sufficient for the beginning of the formation of the sections with almost equiaxial subgrains in the hardened steel. (Fig. 3, *a*, designation 1). At the same time, there are volumes in the metal in which the formation of sub-boundaries is just beginning (Fig. 3, *a*, designation 2).

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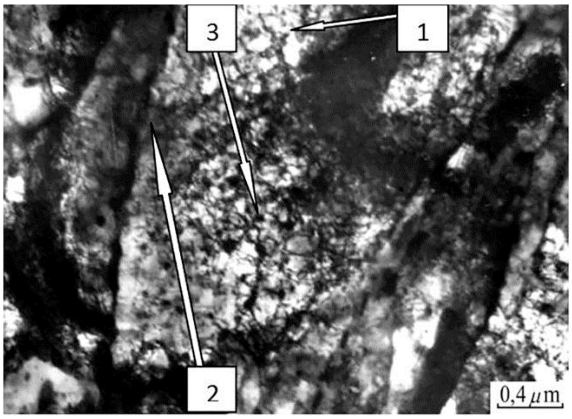
a



b

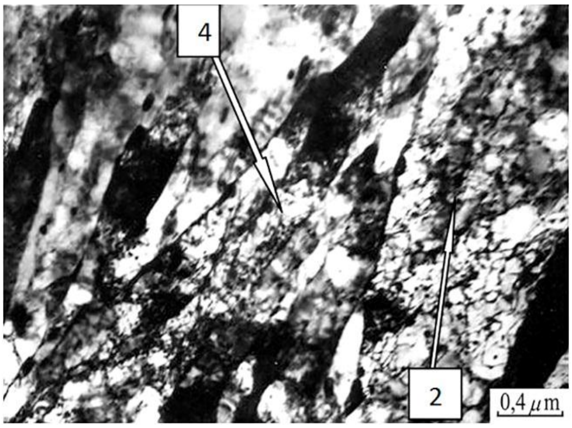


Fig. 3. Microstructure of steel after hardening and tempering at 500˚C

Based on this, the differences in the conditions for the beginning of spheroidization and coalescence processes of cementite particles in the metal microvolumes are one of the reasons for the formation of a certain sub-structural heterogeneity. In microvolumes in which a significant amount of finely dispersed carbide particles have been preserved, the polygonization processes are significantly inhibited, as evidenced by the increased dislocation density (Fig. 3, *a*, designation 3). In contrast, in the sections with more intensive polygonization development, the dislocations redistribution leads to the improvement of sub-boundaries, the purification of the internal volumes of fragmented martensitic crystals from unbound dislocations (Fig. 3, *b*, designation 4). Considering that the additional formation of sub-boundaries and the emission of cementite particles by the nature of their influence are related to hardening, these phenomena should slow down the development of softening processes with increasing the tempering temperature. From the analysis of the results, it follows that the cumulative effect of softening during tempering of hardened steel from the solid solution decomposition, reducing the dislocation density and the coalescence of carbide particles exceeds the hardening from the interaction of dislocations with carbon atoms, the formation of additional sub-boundaries and the hardening from cementite particles of various dispersity.

Originality and practical value

During the research it was for the first time established that for the tempering temperatures of 300–400°C, the absence of phase distortions change indicates the emergence of additional factor in the metal hardening from the formation of sub-boundaries and the dispersion hardening from carbide particles.

The given explanation of the mechanism of structural transformations in the process of tempering in the average temperature range of the hardened carbon steel can be used to optimize the technology of thermal hardening of rolled products.

Conclusions

1. During the tempering of hardened steel in the temperature range of 300-500˚C, the effect of softening from the solid solution decomposition, the reduction of dislocation density and the coalescence of carbide particles exceeds hardening from the dislocations interaction with carbon atoms, the formation of additional sub-boundaries and dispersion hardening from cementite particles.

2. The dependence of the value of the phase distortion indicates the development of complex structural changes during the tempering of 300–400°C in hardened steel with the opposite nature of influence on the strength properties.

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ЗНЕМІЦНЕННЯ ПІД ЧАС НАГРІВАННЯ ЗАГАРТОВАНОЇ СЕРЕДНЬОВУГЛЕЦЕВОЇ СТАЛІ

Мета. У роботі необхідно провести уточнення механізму пом’якшення при нагріві загартованої на мартенсит вуглецевої сталі, що має важливе практичне значення, особливо при розробці технології виробництва прокату з різним рівнем зміцнення. **Методика.** Зразки після гартування на мартенсит

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відпускали при температурах 300–500˚С. Мікроструктуру досліджували під електронним мікроскопом. Фольги виготовляли методами Больмана і пінцету, в хлорно-оцтовому розчині й реактиві Морріса. Викривлення другого роду кристалічної решітки визначали за методиками рентгенівського структурного аналізу з використанням дифрактометра. Наклепаний шар металу після шліфування видаляли електролітичним розчиненням. Тимчасовий опір руйнуванню визначали з діаграм розтягання зразків на машині типу «Інстрон». Мікротвердість вимірювали з використанням приладу ПМТ-3, з навантаженням на індентор 0,49 Н. **Результати**. При нагріванні до температури 300 ˚С загартованої сталі ефект пом’якшення пов’язаний з темпом зниження накопиченої в результаті мартенситного перетворення щільності дефектів кристалічної будови. Сумарний результат обумовлений розвитком рекомбінації дислокацій та пом’якшенням від появи додаткових частинок цементиту при розпаді мартенситних кристалів. Починаючи від температур нагріву 400 ˚С і вище, розвиток процесів полігонізації у фериті супроводжується виникненням додаткових субмеж, які підсилюють ефект зміцнення металу. З підвищенням температури нагріву загартованої сталі рівень міцності властивостей визначається прогресуючим пом’якшенням від зниження ступеня пересичення атомами вуглецю твердого розчину, густини дислокацій і збільшення розміру частинок цементиту над ефектом зміцнення від гальмування рухомих дислокацій атомами вуглецю та виникнення додаткових субмеж. **Наукова новизна**. Для температур відпуску 300–400 ˚С відсутність зміни спотворень другого роду свідчить про появу додаткового фактора в зміцненні металу від формування субмеж і дисперсійного зміцнення від карбідних частинок. **Практична значимість**. Наведене пояснення механізму структурних перетворень в процесі відпуску в середньому інтервалі температур загартованої вуглецевої сталі може бути використано для оптимізації технології термічного зміцнення прокату.

Ключові слова: мікроструктура; мартенсит; цементит; субмежі; гартування; пом’якшення

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РАЗУПРОЧНЕНИЕ ПРИ НАГРЕВЕ ЗАКАЛЕННОЙ СРЕДНЕУГЛЕРОДИСТОЙ СТАЛИ

Цель. В работе необходимо провести уточнение механизма разупрочнения при нагреве закаленной на мартенсит углеродистой стали, что имеет важное практическое значение, особенно при разработке технологии производства проката с разным уровнем упрочнения. **Методика.** Образцы после закалки на мартенсит отпускали при температурах 300–500˚С. Микроструктуру исследовали под электронным микроскопом. Фольги изготавливали методами Больмана и пинцета, в хлорно-уксусном растворе и реактиве Морриса. Искажения второго рода кристаллической решетки определяли по методикам рентгеновского структурного анализа, с использованием дифрактометра. Наклепанный слой металла после шлифования удаляли электролитическим растворением. Временное сопротивление разрушению определяли из диаграмм растяжения образцов при испытаниях на машине типа «Инстрон». Микротвердость измеряли с использованием прибора ПМТ-3, с нагрузкой на индентор 0,49 Н. **Результаты**. При нагреве до 300˚С закаленной стали эффект разупрочнения связан с темпом снижения накопленной в результате мартенситного превращения плотности дефектов кристаллического строения. Суммарный результат обусловлен развитием рекомбинации дислокаций и упрочнением от появления дополнительных частиц цементита при распаде мартенситных кристаллов. Начиная от температур 400˚С и выше, развитие процессов полигонизации в феррите сопровождается возникновением дополнительных субграниц, которые усиливают

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эффект упрочнения металла. С ростом температуры нагрева закаленной стали уровень прочностных свойств определяется прогрессирующим разупрочнением от снижения степени пресыщения атомами углерода твердого раствора, плотности дислокаций и увеличения размера частиц цементита над эффектом упрочнения от торможения подвижных дислокаций атомами углерода и возникновения дополнительных субграниц. **Научная новизна.** Для температур отпуска 300–400 ˚С отсутствие изменения величины искажений второго рода свидетельствует о появлении дополнительного фактора в упрочнении металла от формирования субграниц и дисперсионного упрочнения от карбидных частиц. **Практическая значимость.** Приведенное объяснение механизма структурных превращений в процессе отпуска в среднем интервале температур закаленной углеродистой стали может быть использовано для оптимизации технологии термического упрочнения проката.

Ключевые слова: микроструктура; мартенсит; цементит; субграницы; закалка; разупрочнение

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