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Original article

The Role of Carbides in Forming the Steels Structure and Properties under Pulsed Laser Irradiation

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Abstract

Introduction. At present, in scientific publications, there is no unambiguous understanding and reasoned metal physical justification of the role of the carbide phase of irradiated materials in forming the required structure and achieving a given degree of hardening of surface layers of steels during pulsed laser treatment, especially in the zone of laser hardening from a solid (austenitic) state. The solution to this issue is of great importance, since it allows us to reasonably and purposefully design the required structure of surface layers of products of various functional purposes with high performance properties. The complexity and insufficiently detailed study of the process of structure formation in the surface layers of steels under extreme thermal effects of pulsed laser radiation required a series of metal physical experiments to study the fine structure of steels after high-speed high-temperature hardening. The aim of this article was to obtain, quantify and critically analyze the array of results of metal physical studies and to assess the degree of influence of the carbide phase on the formation of structure and properties of surface layers of steels in the process of pulsed laser hardening in different modes, that is, with and without melting the surface of the samples.

Materials and Methods. In the work, carbon and alloyed tool steels were subjected to surface laser irradiation at a Kvant 16 installation. The radiation power density was 70–200 MW/m^2. Optical, scanning probe and electron microscopy were used in conducting metal physical studies, as well as methods of diffractometric, spectral and durometric analysis of steels before and after laser treatment.

Results. It was shown that laser treatment of steels with a radiation power density of 130–200 MW/m^2 led to a local change in the chemical composition in the laser-fused areas of the spot, partial or complete dissolution of carbides present in the irradiated metal and an increase in the amount of residual austenite in the fused areas up to 40–60%. It was found that on P6M5 steel, the maximum possible hardness of the irradiated zones was achieved by dissolving 30% of carbides, on 9XC, HVG steels — 60–70%. It was shown that under pulsed laser irradiation with q=70–125 MW/m^2, that is, without melting the steel surface, "white zones" formed around carbide inclusions under the influence of thermo-deformation stresses at the boundaries of the "carbide – steel matrix" composition. They had irretrievability, dispersion of the structure and increased hardness (10–12 GPa). It was determined that the maximum hardness of laser-hardened metal in the zones of laser hardening from a solid state was achieved if the "white zones" occupied 40% of the irradiated area of steel. It was found that the dispersion of carbides in this case was 0.5–1.5 microns.

Discussion and Conclusion. The results of the conducted studies indicate that in order to obtain the best combination of hardness and viscosity of the irradiated zones during laser treatment with melting of the surface of steels of different chemical composition, it is necessary to dissolve different amounts of carbides. The dispersed structure of laser-fused steel zones, along with a sufficiently high content of residual austenite, predetermine the possibility of improving the operational characteristics of irradiated materials, especially under conditions of external shock loads.

The analysis of the conducted metal physical studies irradiated without melting the surface of steels allows us to conclude that in order to obtain a high degree of hardening, it is necessary and expedient to ensure the presence of a certain volume of dispersed carbides in the structure of the irradiated steel. The structural composition of "white zones" formed during laser treatment without melting the steel surface contributes to obtaining a unique level of operational properties.

The results of the performed studies contribute to the theory of steel structure formation under conditions of extreme heat exposure and allow for a rational choice of modes of surface laser processing of products and their operability.

Keywords: carbides in steel, laser irradiation, structure, properties

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Научная статья

Роль карбидов в формировании структуры и свойств сталей при импульсном лазерном облучении

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Аннотация
Введение. В современных научных публикациях не существует однозначного суждения и аргументированного металлофизического обоснования роли карбидной фазы облучаемых материалов в формировании требуемой структуры и достижении заданной степени упрочнения поверхностных слоев сталей при импульсной лазерной обработке, особенно, в зоне лазерной закалки из твердого (аустенитного) состояния. Решение этого вопроса имеет большое значение, так как позволяет обоснованно и целенаправленно конструировать требуемую структуру поверхностных слоев изделий разного функционального назначения с высокими эксплуатационными свойствами. Сложность и недостаточно подробная изученность процесса структурообразования в поверхностных слоях сталей при экстремальном тепловом воздействии импульсного лазерного излучения потребовали проведения серии металлофизических экспериментов по изучению тонкого строения сталей после скоростной высокотемпературной закалки. Целью данной статьи явилось получение, количественная оценка и критический анализ массива результатов металлофизических исследований и оценка степени влияния карбидной фазы на формирование структуры и свойств поверхностных слоев сталей в процессе импульсной лазерной закалки на разных режимах, то есть с оплавлением и без оплавления поверхности образцов.
Материалы и методы. В работе поверхностному лазерному облучению на установке «Квант 16» подвергались углеродистые и легированные инструментальные стали. Плотность мощности излучения составляла 70–200 МВт/м^2. При проведении металлофизических исследований использовались оптическая, сканирующая зондовая и электронная микроскопия; методы дифрактометрического, спектрального и дюрометрического анализа сталей до и после лазерной обработки.
Результаты исследования. Показано, что лазерная обработка сталей с плотностью мощности излучения 130–200 МВт/м^2 приводила к локальному изменению химического состава в лазерно-оплавленных зонах пятна, частичному или полному растворению присутствующих в облучаемом металле карбидов и к увеличению количества остаточного аустенита в оплавленных зонах до 40–60 %. Установлено, что на стали Р6М5 максимально возможная твердость облученных зон достигалась при растворении 30 % карбидов, на сталях 9ХС, ХВГ — 60–70 %. Показано, что при импульсном лазерном облучении с q=70–125 МВт/м^2, то есть без оплавления поверхности стали, вокруг включений карбидов под действием термо-деформационных напряжений на границах композиции «карбид – стальная матрица» формировались «белые зоны». Они обладали нетравимостью, дисперсностью строения и повышенной твердостью (10–12 ГПа). Определено, что максимальная твердость лазерно-закаленного металла в зонах лазерной закалки из твердого состояния достигалась в случае, если «белые зоны» занимали 40 % облученной области стали. Установлено, что дисперсность карбидов в этом случае составляла 0,5–1,5 мкм.
Обсуждение и заключение. Результаты проведенных исследований свидетельствуют о том, что для получения наилучшего сочетания твердости и вязкости облученных зон при лазерной обработке с оплавлением поверхности сталей разного химического состава необходимо растворить разное количество карбидов. Дисперсное строение лазерно-оплавленных зон стали, наряду с достаточно большим содержанием остаточного аустенита, предопределяют возможность повышения эксплуатационных характеристик облученных материалов, особенно в условиях действия внешних ударных нагрузок.

Анализ проведенных металлофизических исследований, облученных без оплавления поверхности сталей, позволил сделать вывод, что для получения высокой степени упрочнения необходимо и целесообразно обеспечить присутствие в структуре облучаемой стали определенного объема дисперсных карбидов. Формирующаяся при лазерной обработке без оплавления поверхности стали структурная композиция «белых зон» способствует получению уникального уровня эксплуатационных свойств.
Результаты выполненных исследований вносят вклад в теорию структурообразования сталей в условиях экстремального теплового воздействия, а также позволяют осуществлять рациональный выбор режимов поверхностной лазерной обработки изделий и гарантированно обеспечивать их работоспособность.

Ключевые слова: карбиды в стали, лазерное облучение, структура, свойства

Благодарности. Авторы выражают благодарность рецензентам, чья критическая оценка представленных материалов и высказанные предложения по их усовершенствованию способствовали значительному повышению качества настоящей статьи.

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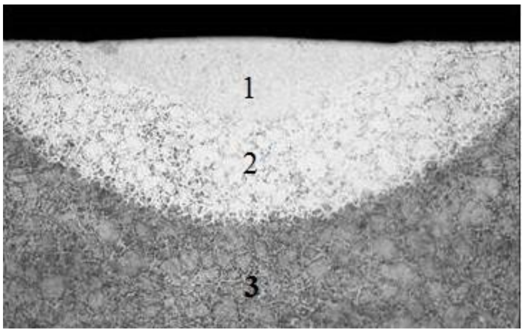
Introduction. During pulsed laser irradiation, the surface layers of the material are subjected to a powerful thermal "shock". Under these conditions, high temperature gradients, concentrations, as well as stress fields appear — thermal, phase, etc. The dissipation of energy acquired by the material can be as follows: partial dissipation of external energy by the dislocation mechanism by local plastic deformation; the dissipation of elastic energy by the mechanism of mass transfer due to the movement of carbon atoms and alloying elements from carbides into solid solutions in contact with them to defects in the crystal structure, etc. [1–4]. It should be noted that mass transfer, which leads to a local change in the chemical composition of laser-irradiated steel zones, plays a particularly important role in the process of structure formation of multiphase steels and alloys containing a significant volume of the carbide phase. The dissolution of carbides, even partial, affects the structure and properties of the surface layers of steels and products in general [5–8].

With high-speed laser processing, that is, in conditions of time scarcity, the effects of carbide dissolution and accelerated mass transfer can be observed only in laser-fused metal zones, at their borders with the initial steel, in thin areas around carbides [9–12]. Emerging microparts with changed chemical composition, structure and properties are of great practical importance, but have not been studied enough. This limits the possibilities for creating a material with a given structure and increased performance in the surface layers of the alloy. Thus, the aim of this study was to determine the influence of the carbide phase on the formation of structure and properties of the surface layers of steels during pulsed laser hardening in different modes, that is, with and without melting the surface of the samples.

Materials and Methods. The analysis of structure formation processes under conditions of high-speed laser heating was carried out on samples of steels U8 (GOST 1435 99), R6M5 (GOST 19265–73), R18 (GOST 19265–73) and others subjected to preliminary volumetric quenching for a martensitic structure and tempering.

Optical, scanning probe and electron microscopy were used in conducting metal physical studies as well as methods of diffractometric, spectral and durometric analysis of steels before and after laser treatment. Pulsed laser irradiation was carried out at a Kvant-16 installation (Russia). Changes in the radiation energy, the degree of beam defocusing (3–6 mm), and the duration of the radiation pulse (1–6) 10^{-3} s allowed varying the radiation power density in a wide range (70–200 MW/m^2). Metallographic studies were carried out on transverse and longitudinal sections on microscopes MIM-7 (Russia) and Neophot-21 (Germany). Studies of fine structure of steels, as well as the determination of chemical composition of the studied zones of irradiated materials were carried out on scanning electron microscopes Hitachi TM-1000 (Japan) and Mira3 (Czech Republic). A diffractometer DRON-0.5 (Russia) was used for X-ray diffraction analysis. Microhardness measurements were carried out on a PMT-3 device (Russia) with a load of 0.49 N.

Results. Metal physical studies showed that the irradiated zones on steels had a heterogeneous structure in the depth of the hardened layer. As it can be seen in Figure 1 *a*, when processing with a radiation power density q=130–200 MW/m^2, a melted quenching zone from the liquid state (1 — LS zone) and a quenching zone from the solid state (2 — SS zone) were observed.



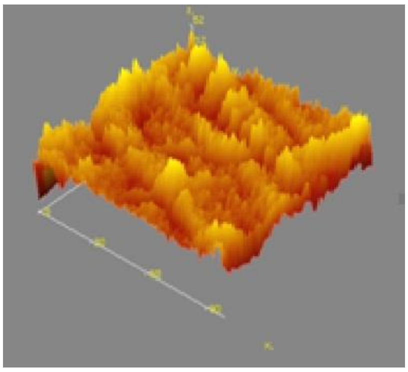
a)

b)

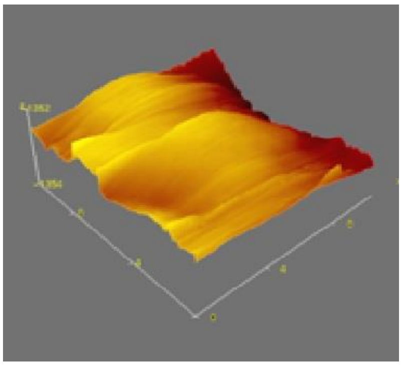
Fig. 1. Microstructure of irradiated steel R6M5: *a* — 1 — LS zone,
2 — SS zone; 3 — initial steel (×200);
b — convective process in the steel melting zone (interference microscopy) (×300)

Further, the features of formation of structure and properties in both zones of the irradiated spot were considered and described, taking into account the influence of the carbide phase in the steels on these processes.

The LS zone features were its incorrigibility in conventional reagents, the dispersion of the structure and high hardness (8–10 GPa), as well as a noticeable decrease in the volume of the initial carbide phase, even when using optical microscopy. Despite the short exposure time of the laser pulse (10^{-3} s), this was facilitated by the high heating temperature and convective mixing of a thin layer of liquid metal caused by the action of thermostrictive stresses (Fig. 1 *b*). The partial or complete dissolution of carbides was evidenced by the results of studies on a scanning probe microscope (SPM) (Fig. 2), which clearly demonstrated that the dissolution of carbides smoothed the surface relief near carbides due to the mass transfer of their components into the surrounding steel matrix.



a)



b)

Fig. 2. Structure of surface layers on R6M5 steel:
a — before laser reflow (SPM); *b* — after laser reflow (SPM)

Confirmation of the possibility of partial dissolution of carbides during high-speed laser quenching with melting of the surface of R6M5 steel was also the results of X-ray diffraction analysis shown in Figure 3.

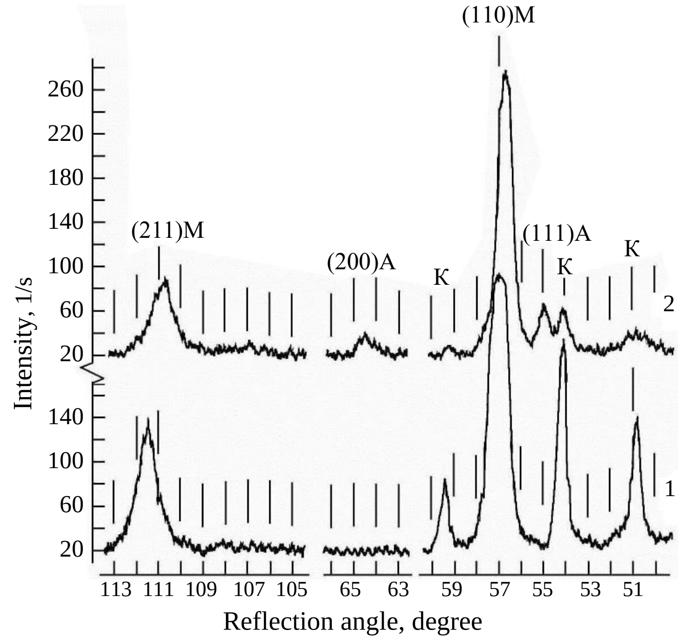
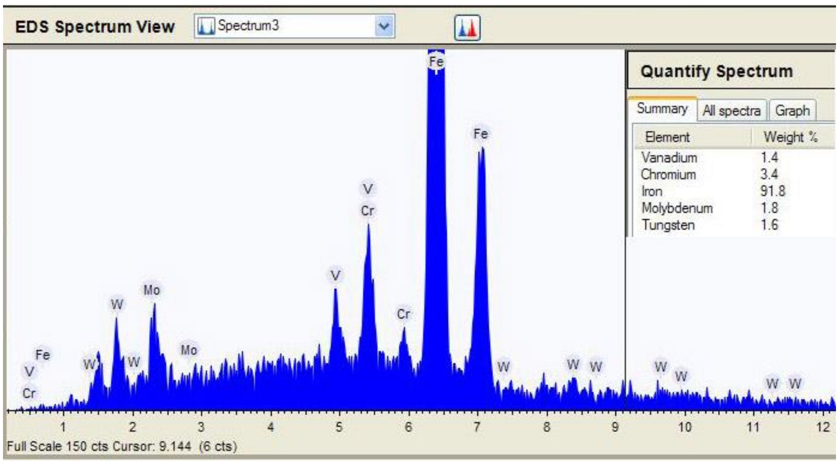


Fig. 3. Phase composition of R6M5 steel before (c. 1) and after (c. 2) laser treatment with surface melting

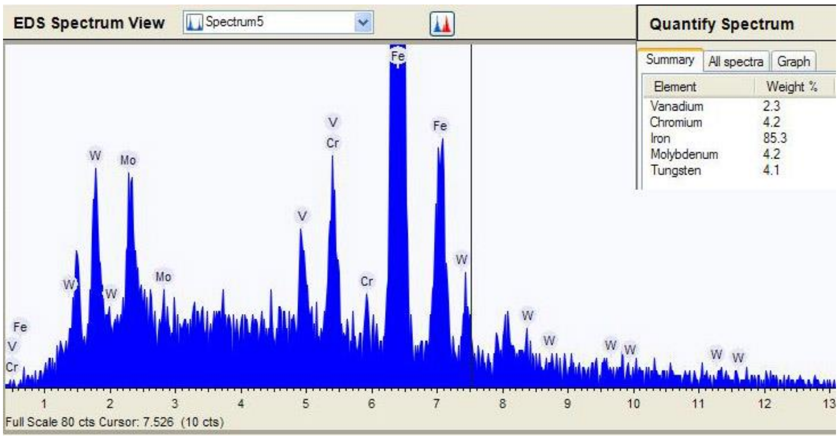
As it can be seen, laser reflow of the steel surface led to a decrease in the height of the carbide phase reflexes (K). Their intensity varied from I=143.24 s^{-1} in the initial steel (Fig. 3, c. 1) to I=65.38 s^{-1} in the LS zone (Fig. 3, c. 2). It can be concluded that the volume fraction of carbides in the LS zone decreased. The angle of the carbides on the radiograph also changed. In the fused zone, carbide reflexes were fixed at angles 2Q=54.1839 compared to 2Q=54.1219 before laser treatment, that is, they shifted to large reflection angles. These results, as well as an increase in the physical expansion of the carbide phase reflexes from 0.6392 mrad for the starting metal to 0.9000 mrad for the LS zone, indicated a change in the stoichiometric composition of carbides, their partial dissolution and an increase in the density of defects of the crystalline structure.

As it can be seen in Figure 3, curve 2, austenite reflexes were also observed on the diffractogram, and martensite reflexes were shifted to smaller reflection angles. This was a consequence of the appearance of areas with high saturation of carbon atoms and alloying elements, and also indicated a high dispersion of the fine structure of the phases of the irradiated metal.

Figure 4 shows the results of determining the local chemical composition of the laser treatment zone of R6M5 steel with surface melting.



a)



b)

Fig. 4. Results of spectral chemical analysis of R6M5 steel samples:
a — in the initial state; *b* — after laser quenching

As a result of changes in the chemical composition, the points of martensitic transformation in the LS zones decreased and a significant amount (40–60%) [13] of residual austenite, characterized by a dispersed structure, remained in them [14–16].

This had a positive effect on the operational properties of irradiated products, especially when exposed to external shock loads.

Figure 5 provides the results of a quantitative assessment of the effect of the volume of dissolved carbides on the degree of hardening of the surface layers of metal obtained during durometric studies of steels irradiated in different modes.

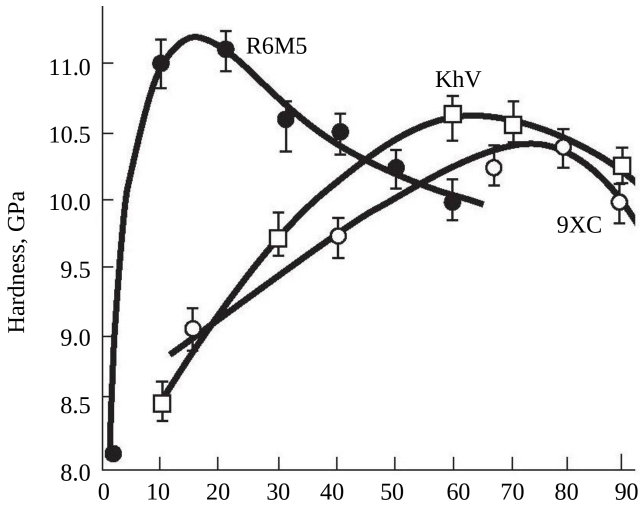
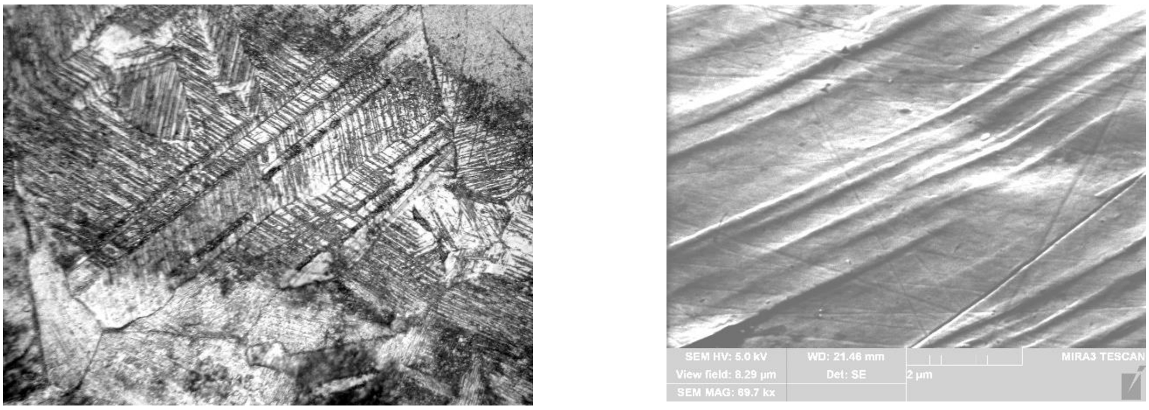


Fig. 5. The effect of the volume of dissolved carbides on the microhardness of irradiated steels

Considering the influence of the carbide phase on the structure of the SS zone, it was necessary to take into account some features of the process of pulsed laser irradiation of materials. They were caused by the appearance of thermostrictive stresses in the irradiated steel zones, the relaxation of which led to local plastic deformation, an increase in density of defects in crystal structure, dynamic return, polygonization and early stages of recrystallization [17, 18]. There was also dispersion of the structure, acceleration of mass transfer of the atoms of the elements and hardening of the metal in the SS zones. Figure 6 provides visual consequences of the influence of local plastic deformation on the structure of polished 12X18H9 steel samples after laser treatment. The traces of deformation in the form of a line or slip bands were clearly visible.



a) *b)*

Fig. 6. Deformation twins after laser treatment of 12X18N9 steel:
a — optical microscopy (×500); *b* — scanning electron microscopy (×10,000)

Figure 7 shows a reconstruction diagram of such characteristic structural features of polygonization and recrystallization processes in laser treatment zones as the formation of a developed substructure, grain refining, grain formation around inclusions, etc.

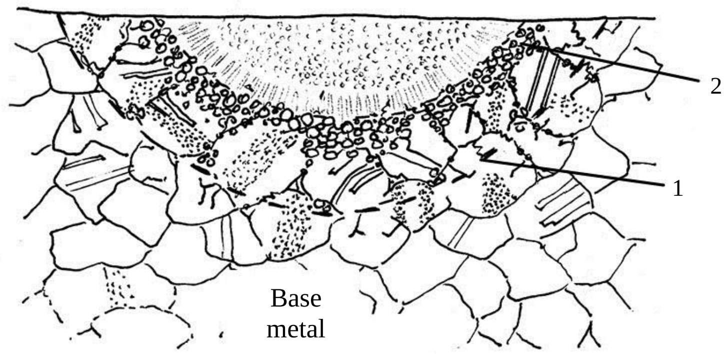
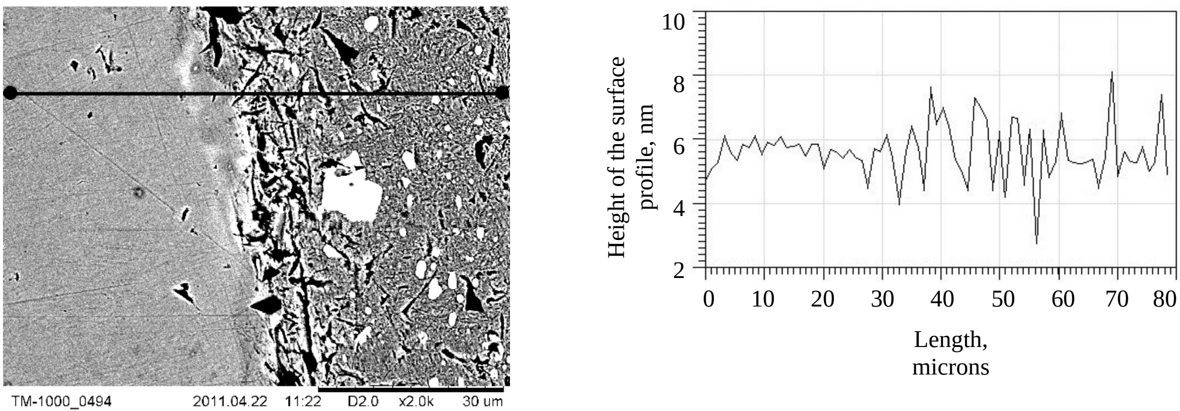
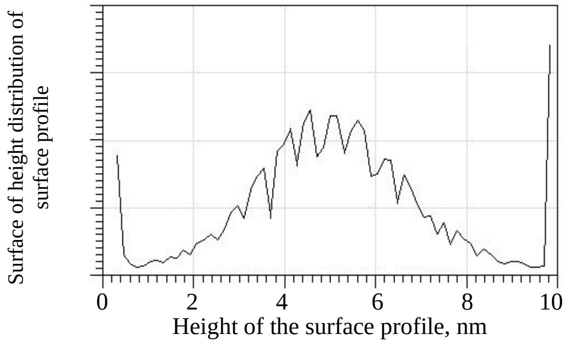


Fig. 7. Diagram of structural features of polygonization (1)
and recrystallization (2) processes in laser irradiation zones

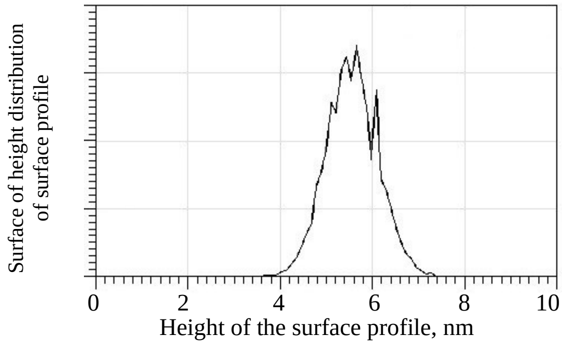
As shown by metal physical studies, the solid state laser quenching zone (SS zone) had a dispersed, poorly etched structure.



a) *b)*



c)



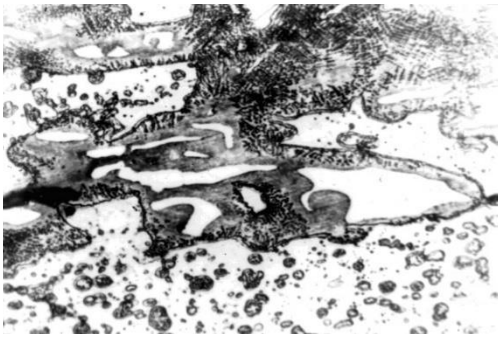
d)

Fig. 8. Microstructure on irradiated R6M5 steel:
a — profilogram of the transition zone from the source metal to the SS zone; *b* — profilogram obtained in the Gwyddion program; *c* — histogram of the distribution of heights of the surface profile in the base metal; *d* — histogram of the distribution of heights of the surface profile in the SS zone

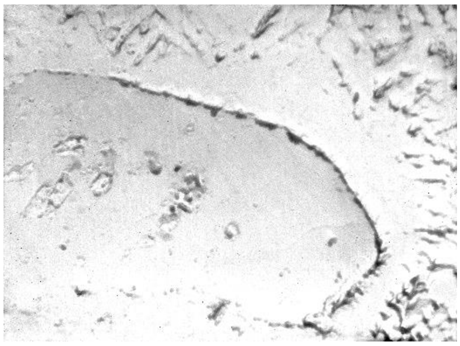
Figure 8 shows the results of the studies of microstructure of irradiated R6M5 steel on a scanning electron microscope.

As it can be seen, carbides were not etched in the SS zone, the surface profilogram was more even than in the base metal (Fig. 8 *b*) and there were no sharp fluctuations in properties at the boundaries in the compositions "carbide – steel matrix".

To confirm the formation of light non-etching shells with an ultradisperse structure ("white zones") around the inclusions of carbides, metallographic studies of the surface of the irradiated without melting zones of R6M5 steel were carried out (Fig. 9).



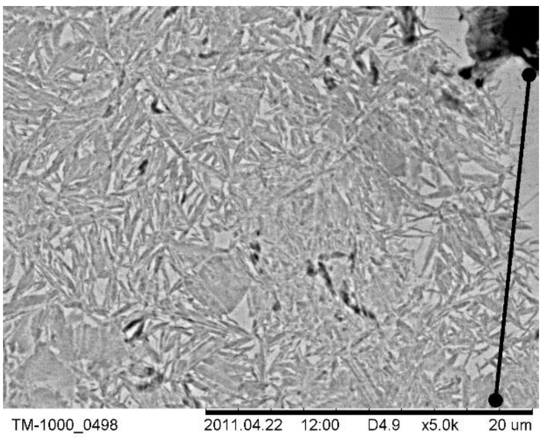
a)



b)

Fig. 9. Boundary dissolution of carbides in irradiated areas of R6M5 steel without melting:
a — metallographic microscope (×800); *b* — electron microscope (×10000)

Studies of the structure of the "white zone" using a scanning probe microscope (SPM) and atomic force microscope (AFM) (Fig. 10 *a*, *c*) showed that martensitic crystals had the form of thin slats 4–7 nm thick and ~150 nm long [19] (Fig. 10 *b*)



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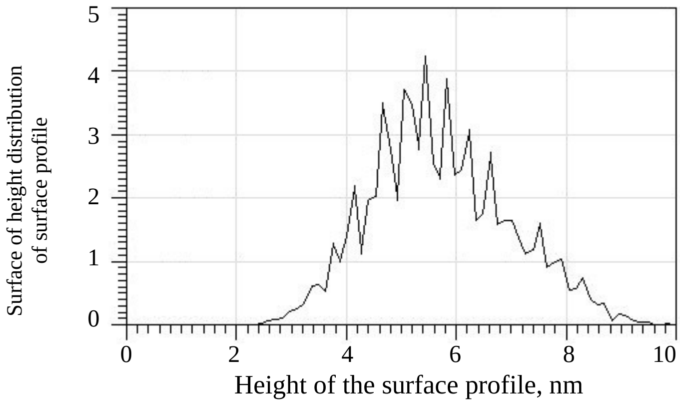

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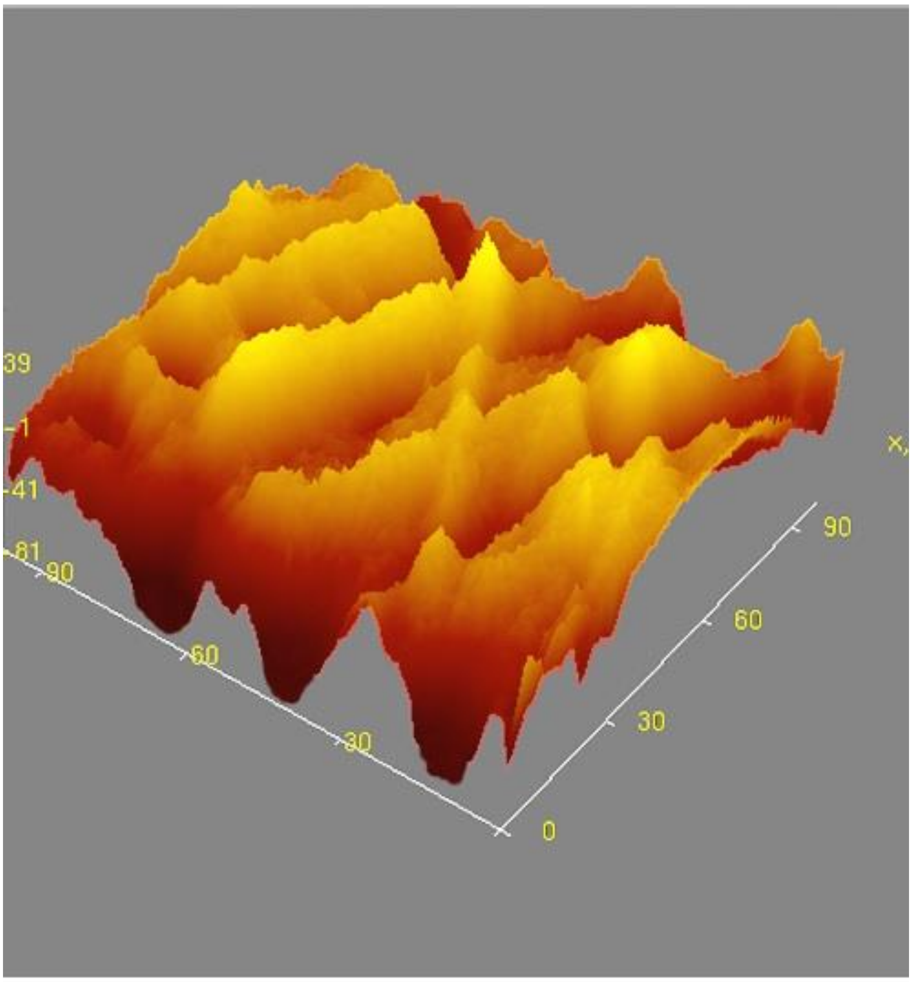

c)

Fig. 10. Microstructure of martensite of laser-quenched R6M5 steel:
a — Hitachi TM-1000 SPM; *b* — distribution of heights of the profile of martensite needles;
c — AFM — image of the structure of the "white zone"

In order to expand knowledge about the fine structure and properties of the SS zone, scratch tests were conducted on a Nanotest installation. A friction probe equipped with a load cell was used, which made it possible to determine the friction force between the indenter and the sample under the influence of gravity of the calibration weights.

Discussion and Conclusion. The listed features of structural state of the melted zone can be associated with the course of dynamic high-temperature plastic deformation during high-speed laser processing. This contributes to the fragmentation of dendritic structure of the irradiated steel zones, accelerates the processes of mass transfer. Confirmation of the above is the result of determining the local chemical composition of the laser treatment zone of R6M5 steel with surface melting. As it can be seen in Figure 4 *b*, due to the dissolution of carbides, the general background of the intensity of reflexes of alloying elements increased in comparison with the original (Fig. 4 *a*) metal.

Based on the results of durometric studies of steels irradiated in different modes shown in Figure 5, it can be concluded that in order to obtain the maximum possible hardness during R6M5 steel laser treatment, it is sufficient to dissolve 30%, and for 9XC, KhVG steels — 60–70% of the initial carbides. The decrease in the hardness of the irradiated zones with an increase in the volume of dissolved carbides was probably due to an increase in the amount of residual austenite under these conditions.

It should be noted that during the operation of irradiated products under the influence of thermo-deformation loads, the decrease in hardness observed in Figure 5 compensated for the large amount of solid dispersed inclusions of hardening carbides released from austenite [20].

The analysis of the results of scanning microscopy of the metal surface showed that in the SS zone the profilogram was more even than in the base metal (Fig. 8 *b*) and there were no sharp fluctuations in properties at the boundaries in the compositions "carbide – steel matrix". This indicates the formation of transitional micro-regions with a changed chemical composition at the borders. As it can be seen in Figure 8 *c*, *d*, the metal in the SS zone was located higher above the plane of the metallographic section. The height of the surface profile on the initial steel was 5.0 nm, and for the laser-hardened zone — 5.8 nm.

The results obtained indicate that the metal regions in the SS zone were more solid and homogeneous, as well as possible partial dissolution of carbide inclusions, which began at the interface between them and the steel matrix. This was confirmed by the formation of light non-etching shells with an ultradisperse structure ("white zones") recorded during metallographic studies around the inclusions of carbides (Fig. 9 *a*), which was especially noticeable at high magnification (Fig. 9 *b*). The formation of such "white zones" during laser processing of steels with a radiation power density of 70–125 MV t/m^2 was facilitated by stresses of various kinds appearing at the boundaries of the "carbide – steel matrix" composition: thermostrictive, stresses due to different thermophysical coefficients in the composition, etc.

Microhardness of these sites was 10-12 GPa. The obtained hardness values corresponded to the hardness of the martensite of alloy steels. It can be concluded that the non-etching edge near the carbide particles was a laser-quenched martensite.

The complex structural picture that was formed around inclusions as a result of stress relaxation was proposed to be described as follows. First of all, due to contact melting, a thin shell of liquid metal was formed in the immediate vicinity of the boundaries of carbides, through which carbon atoms and alloying elements from carbides moved to nearby solid solutions of the irradiated spot. After crystallization, a superhard amorphous-like structure may appear around the carbides. In the rest of the part of "white zones", $\alpha \rightarrow \gamma$ transformation during heating under extreme temperature and force conditions was carried out by shear mechanism. This led to plastic deformation and dynamic polygonization of austenite with the formation of a fragmented substructure. In the process of high-speed hardening in the austenitic edge, $\gamma \rightarrow \alpha$ transformation occurred with the inheritance of the fragmented structure of austenite by martensite.

The results of metallographic and durometric studies showed that the maximum hardness of laser-hardened metal in the SS zones was achieved if the "white zones" occupied 40% of the irradiated area of steel. The dispersion of carbides should be 0.5–1.5 microns.

It was established that when the indenter scratched the base metal of R6M5 steel, friction force fluctuations were observed caused by the movement of the indenter through an inhomogeneous structure consisting of phases with different hardness. There were no significant friction force fluctuations in the SS zone of the laser-irradiated metal. It can be concluded that the SS zone was relatively homogeneous in structure and hardness, and its hardness was much higher than the hardness of the base metal. This was evidenced by the values of the friction force of about 17 MN in comparison with 11 MN for the base metal.

The analysis of the conducted metal physical studies indicates that laser treatment with a radiation power density of 130–200 MW/m^2, that is, with the melting of the surface of steels of different chemical composition, obtained the best combination of hardness and viscosity of the irradiated zones when dissolving different amounts of carbides. Due to the fixed dispersed structure of laser-fused steel zones, along with a sufficiently high content of residual austenite in them, it became possible to increase the operational characteristics of irradiated materials, especially under conditions of external shock loads.

Metal physical studies of irradiated steels, with a radiation power density of 70–125 MW/m^2, that is, without melting the surface, allowed us to conclude that in order to obtain a high degree of hardening in this case, it is necessary and advisable to ensure the presence of a certain volume of dispersed carbides in the structure of the irradiated steel. The structural composition of the "white zones" formed during laser treatment without melting the steel surface contributed to obtaining a unique level of operational properties.

The results of the research made a contribution to the theory of steel structure formation under conditions of extreme heat exposure, and also made it possible to make a rational choice of modes of surface laser processing of products of various functional purposes to ensure their operability.

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