Features of silicon saturation of austenite pipe steel samples with different operating time

Nikita V. Lemeshko*, Aleksey V. Rubtsov, Jamshed N. Shermatov, Sergei V. Chertovskikh, and Ruslan M. Tazetdinov

Ufa State Petroleum Technological University, Department Technological machines and equipment, Ufa, 450064, Russian Federation

Abstract. In the oil refining and petrochemical industries, the problem of premature failure of furnace equipment operating at high operating temperatures due to diffusion saturation of metal with structural elements with carbon is quite acute. Sections of coils of reaction furnaces operating under severe temperature conditions are especially often rejected due to carburization. The carbon saturation of the surface layers of the metal of the pipes to different depths leads to a change in the chemical composition and mechanical properties, and as a result, to a significant deterioration in plasticity, which increases the tendency to crack. One of the most significant causes of carburization of the metal of furnace tubes is coco-deposition along the inner surface of the tubes and this leads to a more intensive rotation of the mechanism of diffusion of carbon into the metal from coke. The rate and degree of carburization can be reduced or in some cases even prevented from forming surface modified silicon-based layers since it is silicon that is a carbon antagonist. In this regard, the current topic is the study of the features of silicon saturation of the surface layer of samples of austenitic pipe steel with different operating time.

1 Introduction

Carburization of the internal surface of the tubular furnaces of the oil and gas industry, operating on the principle of reactors in conditions of simultaneous exposure to high temperatures, internal pressure and hydrocarbon raw materials, is one of the main reasons for the frequent rejection of coils. In the audit process, such areas are detected using modern methods of monitoring the technical condition, for example, hardness measurements. The metal in areas saturated with carbon has a limitation on plastic properties, and therefore the hardness of the metal becomes higher. To a large extent, diffusion saturation of the metal with carbon is facilitated by coke deposition along the inner side of the coil tubes. Uneven coke deposition on the inner surface of the pipes leads to overheating of individual areas and contributes to the intensification of the carburization process. Silicon can be an effective protection against the introduction of carbon into the metal. Moreover, the features and mechanism of forming a silicified layer in the metal of pipes with different conditions and service life can have fundamental differences. Therefore, research on the development of

* Corresponding author: lemeskonikita4@gmail.com
optimal techniques for the formation of modified surface layers based on silicon is currently relevant and may allow us to come close to solving the problem of carburization. So, for example, in [1-2], work was carried out on the development and study of a diffusion silicidal coating to increase the resistance of the metal of furnace pipes to coke deposition. It has been found that the silicification allows the adhesion strength of petroleum carbon to the metal surface to be approximately halved, but to some extent reduces the fatigue strength of the steel. In work [3], the authors established an optimal time period for the siliconization of samples from 10Kh23N18 steel with different terms and conditions of operation to form the largest thickness of the siliconized layer. Studies have shown that the most optimal holding time in the silencing mixture is a time interval of 8 hours. It was also found in [3] that the thickness of the siliconized layer on the concave surface of the siliconized samples is higher than on the convex one. In work [4], the authors conducted studies to study the formation of a silicified layer of the greatest thickness on samples cut from fragments of pipes with dimensions of 160×6 mm, as well as fragments of small diameter pipes made of 08Kh18N10T steel. The results show that the formation of the siliconized layer of the greatest thickness in the samples occurs within 8 hours, however, for pipe fragments, the formation of the siliconized layer of the greatest thickness occurs within 6 hours. From the results obtained, the influence of the scale factor can be clearly traced. Also, research in the field of metal silicification is devoted to some works [5-13].

2 Research methodology

Figures Samples with a size of 10×10 mm, cut from the annular section of the fragments of the coil of the reaction furnace from heat-resistant austenitic steel 10Kh23N18 dimensions of 114×7 mm with different terms and conditions of operation, were subjected to filtration:

- Fragment No. 7 was operated in the radiation chamber for 1200 hours at a temperature of about 840 °C;
- Fragment No. 8 was operated in the radiation chamber for 8000 hours at a temperature of about 840 °C;
- Fragment No. 9 was operated in the convection chamber for 36346 hours at a temperature of about 650 °C;
- Fragment No. 11 was operated in the convection chamber for 64466 hours at a temperature of about 650 °C.

The internal overpressure in the furnace coil varies to 0.55 MPa. The samples were siliconized in a silicon carbide-based silicate powder mixture. Information on the siliconization technology is given in [1-3]. In work [3], it was found that when samples with different periods and operating conditions are siliconized during 2, 4, 6, 8, a gradual increase in the thickness of the siliconized layer occurs and reaches the maximum value at 8-hour exposure, however, when samples are held for 9 hours, the thickness of the siliconized layer decreases compared to the thickness obtained during exposure for 8 hours. Therefore, the task was set to experiment the above samples for 8 hours and re-siliconize the same already filtered samples for another 8 hours. After carrying out the siliconization and performing certain preparatory work using an optical microscope, images were obtained on a computer and the thickness of the modified silicon-based surface layer was measured in samples on the inner (concave) surface. The inner (concave) surface was chosen because the coking occurs precisely on the inside of the coil tubes, and the inner surface needs more protection from carburization. After conducting the siliconization and performing certain preparatory work using an optical microscope, images were obtained on the computer and the thickness of the modified silicon-based surface layer was measured in samples on the inner (concave) surface. The inner (concave) surface was chosen because the coking occurs precisely on the inside of the coil tubes, and the inner surface needs more protection from carburization.
3 Research results and discussion

Figure 1 shows the images of the siliconized layer and the results of measurements of its thickness on the inner surface of samples with different operating times and under different operating conditions, which were filtered once for 8 hours.

**Fig. 1.** Results of silicate layer measurement over the inner surface of samples aged in the silencing mixture for 8 hours: a) a siliconized layer of the internal surface of the sample from fragment No. 7; b) a siliconized layer of the inner surface of the sample from fragment No. 8; c) a siliconized layer of the inner surface of the sample from fragment No. 9; d) a siliconized layer of the inner surface of the sample from fragment No. 11.

**Fig. 2.** Results of measurements of the thickness of the siliconized layer on the inner surface of samples aged in the siliconizing mixture for 8 hours, and then again for 8 hours: a) a siliconized layer of the internal surface of the sample from fragment No. 7; b) a siliconized layer of the inner surface of the sample from fragment No. 8; c) a siliconized layer of the inner surface of the sample from fragment No. 9; d) a siliconized layer of the inner surface of the sample from fragment No. 11.
Figure 2 shows the images of the siliconized layer and the results of measurements of its thickness in samples filtered for 8 hours on the inner surface, aged in the siliconizing mixture for 8 hours again. Three measurements were taken at the points of the most uniform distribution of the thickness of the silicified layer, and the maximum thickness value was taken as the result.

Analyzing Figures 1 and 2, it can be concluded that the formation of a silicified layer of one or another quality is observed in all test fragments. The quality of the layer can be influenced by various parameters, such as, for example, surface quality, the ratio of the areas of saturated surfaces to the volume of the mixture, the location of the prototype relative to the container in which the filtration is carried out, etc. An uneven porous diffusion layer is observed, and after re-siliconization for 8 hours, the porosity of the diffusion layer becomes more pronounced. After re-siliconization, some samples show surface layer cleavage.

Based on the results shown in Figures 1 and 2, histograms were constructed showing the maximum values of the siliconized layer of the samples depending on the fragment number, as well as the holding time of 8 hours once and repeated exposure for 8 hours. Histograms are shown in Figures 3-4.

![Figure 3](https://example.com/fig3.png)

**Fig. 3.** Histograms of the thickness of the siliconized layer at exposure in the siliconizing mixture for 8 hours once for samples from pipe fragments with different operating life and operating conditions.

![Figure 4](https://example.com/fig4.png)

**Fig. 4.** Histograms of the values of the thickness of the siliconized layer at exposure in the siliconizing mixture for 8 hours again for samples already filtered for 8 hours from pipe fragments with different operating life and operating condition.
From the constructed histograms it is visible that when siliconizing samples within 8 hours once the greatest thickness is received for the samples which are cut out from fragment No. 9. It is a fragment it was operated at the softest, in terms of operating temperatures, conditions. Also, this fragment has the smallest term of an operating time in the conditions of the convection camera of the reactionary furnace. The smallest thickness turned out for three other fragments that is perhaps connected with the most severe conditions of operation in the conditions of the radiant camera of the furnace of fragments No. 7 and No. 8 and also with rather long term of operation in the conditions of the convection camera of fragment No. 11.

At repeated the 8th hour siliconizing of samples increase in value of thickness of the siliconized layer for all fragments is observed. And the greatest increase in thickness of the siliconized layer approximately by 1.7 times happened for the sample which is cut out from fragment No. 7 which was operated in the most rigid temperature condition the smallest of all test fragments time. At the samples which are cut out from fragments No. 8 and No. 9 increase in thickness of the siliconized layer happened approximately in 1.3÷1.4 of time. For the sample which is cut out from fragment No. 11 of increase in the maximum value of thickness of the siliconized layer practically it is not observed.

4 Conclusion

Based on the results of the studies, the following conclusions can be drawn:

- it is shown that the siliconized layer is formed to various degrees in samples cut from fragments of reaction furnace tubes operated in both the radiation chamber and the convection chamber;
- it was found that in case of one-time silencing for 8 hours of samples cut from fragments of tubes of the reaction furnace coil with different terms and conditions of operation, the greatest thicknesses are formed in fragments of tubes of the convection chamber coil with lower operating time, and the smallest thicknesses - in fragments of tubes of the radiant chamber, as well as tubes of the convection chamber with sufficiently long operating time;
- It has been shown that when repeated siliconisation occurs during 8 of the hourly samples, the thickness of the siliconised layer increases compared to a single 8-hour siliconisation, with the greatest increase in the thickness of the siliconised layer being observed in fragments of the radiant chamber tubes as well as a fragment of the convection chamber tube with short-term operation. For the pipe fragment of the convection chamber, which has been in operation for a fairly short time, an increase in thickness practically does not occur.

References