DEVELOPMENT OF A NEW METHODOLOGY FOR ASSESSING THE QUALITY OF ROLLED METAL FOR ENAMELING

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Received 02 November 2022
Accepted 20 December 2022

ABSTRACT

One of the most important indicators of the quality of rolled metal for enameling is the hydrogen embrittlement index. The amount of hydrogenation determines the propensity of rolled metal products to the formation of a "fish scale" defect in the finished enamelled product. The article considers a new indicator - the volume fraction of structurally free cementite in the microstructure of rolled metal, which correlates to a very high degree with the value of hydrogen embrittlement index. The developed method for determining a new indicator is described, which allows, on its basis, to predict the probability of the occurrence of the "fish scale" defect on enameled products. It has been established that the test of rolled metal products complies with the normative documents in terms of the hydrogen embrittlement index value, if the volume fraction of structurally free cementite is more than 2.7 %. A technology has been developed for hot rolling of low-carbon steel, which makes it possible to provide high values of the volume fraction of cementite in the microstructure.

Keywords: cold-rolled steel for enameling, "fish scale" defect, hydrogen embrittlement index, the volume fraction of structurally free cementite.

INTRODUCTION

As it is known, the preparedness of rolled metal for enameling depends on many factors, such as the chemical composition of steel, the condition of the surface, the technology of production of rolled products [1 - 5]. According to foreign and national researchers [6 - 11], hydrogen is the main cause of the occurrence of defects in enamel coatings of steel products. The presence of inclusions, violation of the etching mode, incorrect temperature-speed roasting mode for this kind of enamel may cause the occurrence of the "fish scales" defect [12 - 15]. The most common steel grade of rolled metal for enameling in Russia is steel 08ЮР (Russian Standard). To assess the tendency of metal to hydrogenation, index of hydrogen embrittlement (HEI) is used in national practice. The method of HEI determination and threshold values are stipulated by GOST 24244-2018 (Russian National Standard) and Technical Specifications 14-101-321-2008 (Russian Standard). At the same time, in accordance with GOST 24244-2018, the value of hydrogen embrittlement index less than 40 % is considered unsatisfactory. According to Technical Specifications 14-101-321-2008 for rolled steel 08ЮР, the tendency of steel to hydrogen embrittlement is indicated by HEI which value should be at least 30 - 50 % for various steel groups. That is, at low HEI values, the probability of the formation of a "fish scale" defect in the finished enameled product increases significantly.

The method of determining hydrogen embrittlement index consists of the following steps. The sample under study is subjected to 90° bending until its destruction. Another similar sample from the same steel grade is immersed in sulfuric acid, kept in it for a certain time, after which it is also subjected to a bending test until destruction. The ratio of the number of bends until destruction in the initial sample to the number of bends of the sample soaked in acid determines the HEI value in percentage terms. This method of assessing the propensity of rolled metal to form a "fish scale" defect seems to

be uninformative and not completely unambiguous. We believe the described method of determining HEI has a number of disadvantages. In particular, it is required to maintain high purity and equal acid concentration in the process of testing, which, obviously, is not always possible to ensure because of a human factor when tested by a laboratory assistant, and not by a machine (speed of bends, angle of inclination, etc.). In addition, the equality of the values of the HEI may be incorrect when tested in different laboratories. Appropriate cleaning of the rolled metal surface is also required, therefore, different values of hydrogen brittleness are possible when exposed to different solvents, including mechanical cleaning.

Numerous studies conducted by scientists of Nosov Magnitogorsk State Technical University together with specialists of the Magnitogorsk Iron and Steel Works (MMK), allowed us to develop a new criterion - the volume fraction of structurally free cementite that correlates with the value of the hydrogen embrittlement index and allows us to assess the propensity of rolled metal to the appearance of a "fish scale" defect [16]. As it is known, the following factors significantly affect the entrapment and retention of hydrogen in the steel surface: extended grain boundaries (for example, the ferrite grain score 8 - 10), various structural components and phases in addition to the ferritic matrix (in particular, cementite mesh, nitrides since boron is a strong nitride-forming agent along with aluminum), pores due to the possible presence of acid-soluble elements (for example, aluminum) [2]. Therefore, the presence of a significant number of cementite globules, preferably in the form of a grid, allows us to strengthen the entrapment and retention of surface hydrogen appearing in the enameling processes. Thus, the microstructure of rolled metal from low-carbon steels with a low content of cementite for enameling significantly affects the mechanism of formation of the "fish scales" defect and is closely related to the HEI value.

Taking such approach to the problem, the formation of cementite is carried out at the stage of hot rolling. Technological modes of rolling steel 08ЮР were developed, providing high values of the volume fraction of cementite in the microstructure. At the same time, the temperature of final stage hot rolling, depending on the thickness of the finished rolled steel, was in the range of 860 - 890°C, the winding temperature was not lower than 740°C. However, in addition to the temperature ranges, the speed parameters of rolling and winding, as well as the cooling strategy on the discharge roller of the hot rolling mill, were significant factors.

EXPERIMENTAL

The methodology of estimating the volume fraction of cementite is as follows. Samples are taken out from the finished cold-rolled steel 08ЮР. Metallographic analysis is carried out on this sample using a scanning electron microscope and Thixomet Pro software package:

- preparation of microslices of cold-rolled products according to the standard procedure (GOST 5640 - 68 (Russian National Standard));
- the etching of samples in a four-percent solution of nitric acid in ethyl alcohol by immersion;
- electron microscopic examination of samples using a scanning microscope at 500 - 1000× magnifications to estimate the grain size of ferrite and the volume fraction of structurally free cementite. Furthermore, to estimate the average particle size of structurally free cementite and its volume fraction, an additional study is carried out at 2000 - 3000× magnifications. Shooting is carried out by 3 - 6 fields of view (3 - 6 photos) at each of these magnifications:
- input of raster electron microscopic (SEM) images into Thixomet PRO software with subsequent image calibration;
- quantitative analysis of the volume fraction of structurally free cementite in SEM images, carried out in automatic or manual mode using specialized programs by highlighting the area of the existence of structurally free cementite;
- statistical processing of the results obtained (calculation of the arithmetic mean of the volume fraction of structurally free cementite in all images), carried out in the automatic mode.

Based on the results obtained, the volume fraction of structurally free cementite is analyzed and evaluated. Evaluation and analysis of the results: the test is considered satisfactory (the sample has a high resistance to hydrogen embrittlement and a low tendency to form a "fish scale" defect) if the volume fraction of structurally free cementite is 2.7 % or more. In addition, microstructure contains a clear line arrangement of structurally free cementite with a particle size of 2 - 3 microns, representing one- or two-layer chains. The method to determine

the volume fraction of structurally free cementite in the microstructure of steel 08ЮР (Russian Standard) in Thixomet PRO software product in automatic mode (Fig. 1) and in manual mode (Fig. 2) is presented.

RESULTS AND DISCUSSION

Comprehensive studies have been carried out, including sampling both in length and width of rolls rolled according to various schemes. The analysis of the research results is presented in the Table 1.

As follows from the analysis of the research results, when the volume fraction of cementite in the rolled product structure is higher than 2.7 %, the HEI corresponds to regulatory documents.

A SEM study of rolled products with a high value of HEI was carried out (Fig. 3) shows the microstructure of sample No. 9 (volume fraction of cementite 3.33 %), Fig. 4 shows the microstructure of sample No 6 (volume fraction of cementite 3.22 %) (see Table 1).

Previously, the samples were subjected to simulated extraction on a molding die, after which double-sided powder enameling was carried out. In all the samples studied, the "fish scales" defect was not detected.

Fig. 5 shows the external appearance of the trial batches of rolled metal produced by PJSC Magnitogorsk Iron and Steel Works (Russian Federation) from steel 08ЮР

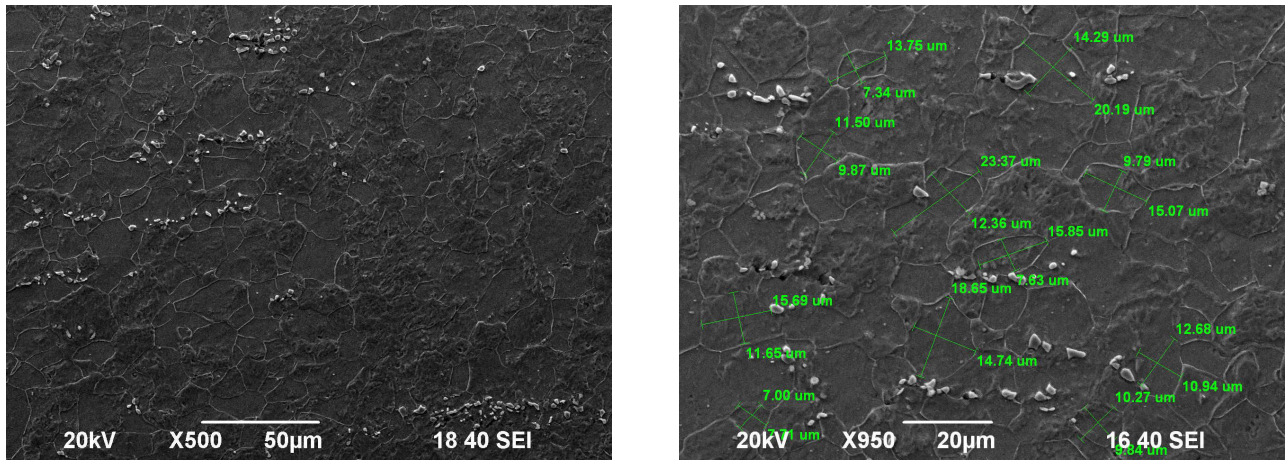

Fig. 1. Example of determination of volume fraction of structurally free cementite in 08ЮР steel (Russian Standard) microstructure in Thixomet PRO software: a - initial SEM-image; b - SEM-image after processing in the software product Thixomet PRO in automatic mode by selecting the area of structurally free cementite.

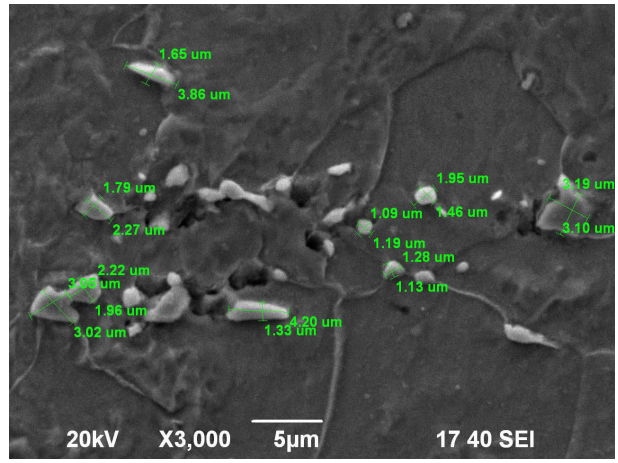

Fig. 2. Example of determination of volume fraction of structurally free cementite in 08ЮР steel (Russian Standard) microstructure (in manual mode).

Table 1. Results of samples tests of cold rolled 08ЮР steel (Russian Standard).

Sample number	Propensity to embrittlement, %	The volume fraction of cementite, %	The ferrite grain score
1	12.3	1.38	10
2	14.0	1.76	10
3	22.7	2.17	9, 10
4	36.9	2.20	9, 10
5	40.2	2.75	10
6	48.2	3.22	9
7	57.8	3.26	9, 10
8	60.0	3.29	10
9	63.7	3.33	10

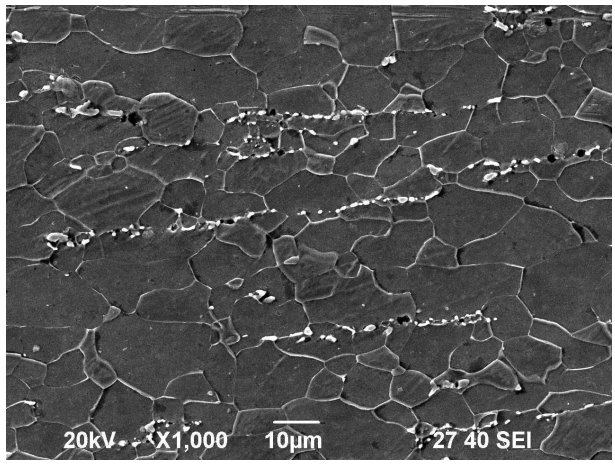

Fig. 3. Microstructure of sample No 9 of a pilot batch of cold-rolled steel with a volume fraction of structurally free cementite of 3.33 %.

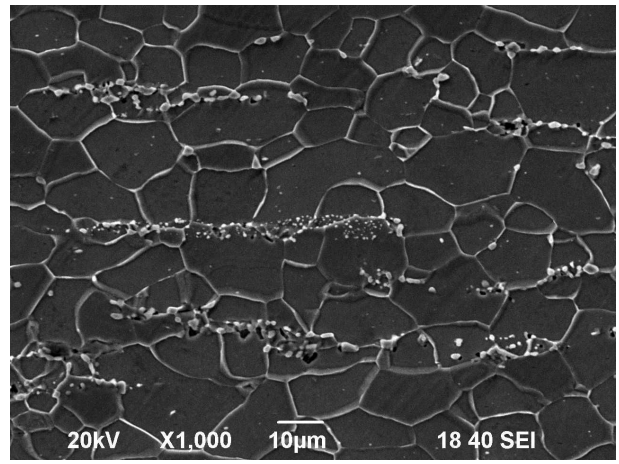

Fig. 4. Microstructure of sample No 6 of a pilot batch of cold-rolled steel with a volume fraction of structurally free cementite of 3.22 %.

Fig. 5. Appearance of powder enamelled trial batches of PJSC Magnitogorsk Iron and Steel Works (Russian Federation).

(Russian Standard), enameled by powder in LLC "NEXT" (Uzbekistan, Tashkent).

CONCLUSIONS

A new criterion is proposed - the volume fraction of structurally free cementite, which correlates to a high degree with the value of hydrogen embrittlement index and allows us to assess the propensity of metal rolling to the appearance of a "fish scale" defect. A research methodology has been developed for determining the volume fraction of structurally free cementite in rolled metal, which allows us to assess the tendency of cold-rolled products to form a "fish scale" defect on enameled products. The test is considered satisfactory

if the volume fraction of structurally free cementite is 2.7 % or more. With the values of the volume fraction of cementite in the rolled product structure above 2.7 %, the hydrogen embrittlement index corresponds to regulatory documents.

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