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Refinement of Techniques Metallographic Analysis of Highly Dispersed Structures

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Abstract. Flaws are regularly made while developing standards and technical specifications. They can come out as minor misprints, as an insufficient description of a technique. In spite the fact that the flaws are well known, it does not come to the stage of introducing changes to standards. In this paper shows that in the normative documents is necessary to clarify the requirements for metallurgical microscopes, which are used for analysis of finely-dispersed.

1. Introduction

The methods of metallography analysis based on comparing with standard scales still remain up to date, despite intensive development of digital image processing and a broad range of computer programs for metallographic analysis.

The major advantages of comparative method are its operational efficiency (several seconds are required for analysis) and cost efficiency. Besides, during analysis process it is necessary to estimate the form of structural components, their distribution and combinations that are difficult to describe quantitatively. Thus comparative methods have become widely spread in practical metallography among production laboratories in spite the fact that these methods have considerable disadvantages. The major method disadvantages are the following: low precision at quantitative estimations and an operator's subjective apprehension.

The total microscope magnification is considered to be one of the basic observation conditions in GOST but without additional requirements to its resolving power or lens aperture [1–9]. That is why it is magnification which is taken into account by a specialist while choosing the equipment. Sometimes it is difficult to make a case for additional equipment requirements due to the absence of these requirements in the corresponding GOST.

The classification of the most disperse steel structures (perlite, sorbite and martensite) observed in an optical microscope is defined according to GOST 8233-56 [1]. Perlite with dispersibility up to 0.6 μM is referred to grades 1, 2, 3, 4 (sorbitic pearlite, latent lamellar pearlite, thin-lamellar pearlite, fine lamellar pearlite). The most dispersed structure is characterized by the fact that specialists are unable to distinguish separate particles (flakes, needles, grains) with the 0.2 μM size by 1000^x magnification. It is necessary to get 0.2 μm magnification (eyes resolving ability is 0.2 mm with the distance of 200-250 mm) in order to achieve useful magnification. It is possible to achieve such kind of resolution in lightning with violet colour-filter by using lenses with aperture not less than 1.25. Useful 1000^x magnification in more comfortable colour range is achieved by increasing aperture up to 1.4–1.5 values.

At the same time one of the trends in the development of microscope equipment has become the increase of ergonomics and the efficiency increase of setting changes and magnifications. Therefore, the use of dry (not immersion) lenses allows to save time that is wasted on a sample and a lens cleaning after analysis and during the change to weaker lenses. However, the resolving power of such kind of lenses is less than resolving power of immersion lenses. The use of lenses with increased working distance allows to increase the efficiency of change resolution without the threat of damaging a monitored object and the frontal lens. However, the working distance increase is often achieved by the number aperture decrease.

On the other hand, high magnification ratio can be achieved by combination of a 40^x lens with 0.55-0.65 aperture and a 25^x ocular. The resolving power of such kind of systems does not exceed 0.45 μM.

Thus metallographic microscopes with maximum magnification of 1000x are being produced in increasing frequency. This magnification is achieved by the use of lenses with N.A.=0.55÷0.95 aperture that corresponds to resolving power of d=0.7÷0.35 μm relatively.

GOST 8233-56 (Union State Standard) [1] is one of the foundational documents that classifies the structure of steels. It practically has not been changed since its creation.

The aim of the work is to highlight the necessity for changes that have to be introduced into GOST 8233. In particularly, the necessity for detailed requirements to metallographic microscopes and to the replacement of photo-standards.

2. Experiment

The use of lenses with lower resolving power can be justified while observing relatively large structural constituents with the size of 0.5 μM and more: (the determination of non-metallic constituents, lamellar structure, the ratio of large structural constituents and etc.). However, the evaluation structure results highly depend on microscope resolving power while estimating finely-dispersed structures especially when 1000^x magnification is required. If using dry lenses so the structure of lamellar pearlite with dispersibility less than grades 3 and 4 can be correlated to grade 1 according to GOST 8233-56 (figure 1a).

a b

Figure 1. Photo-standards of lamellar pearlite GOST 8233: a – grade 1, b – grade 2.

Let us give an example. Figure 1 shows photo-standards of lamellar pearlite with grades 1, 2 according to GOST 8233-56. Further figure 2 represents photographs of one and the same part of bearing steel as that was annealed on lamellar pearlite. These photographs were taken with microscope

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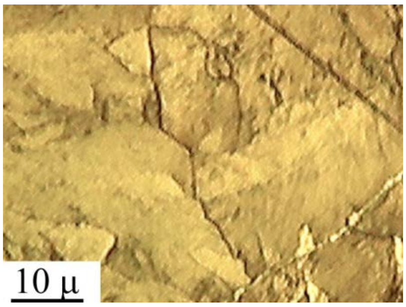
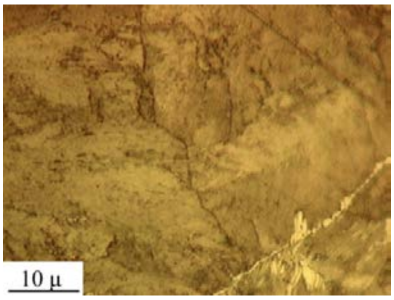
Olympus BHMJ using the following lenses: Plan 40 LWD with 0.55 aperture, Plan 100 with 0.9 and 1.25 aperture. Photography was carried out with Lab 200C US camera, the lightning was organized with the use of JS-18 (yellow) colour-filter.

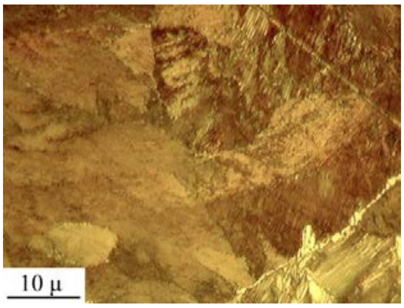
The metallographic templates preparation was carried out according to a standard technique that includes grinding, polishing and further etching with a reagent (2% solution of nitric acid) in spirit. Grinding was conducted on an abrasive wheel with 450 μm grit and on paper with diamond paste. At the same time, the paper paste with grit of 60/40, 14/10 and 7/5 was being changed sequentially. Polishing was conducted on artificial chamois with diamond paste of 0.5/0 grit.



a



b



c

Figure 2. Microphotographs of bearing steel, taken with the use of lenses a – 40^x (N.A.0.55), b – 100^x (N.A.0.9), c – 100^x (N.A.1.25).

The grains (pearlite colony) which can be graded as 1 according to GOST 8233-56 are observed in the first two cases (figures 2a and 2b) as pearlite lamellar structure can not be distinguished. The grains take the form of distinct lamellar structure. It means that the distance between cementite plates in pearlite is no more than 0.2 μm. A such kind of structure can be graded as 2 according to GOST 8233-56.

Spoilage metal products can be let pass at the controlling stage because of this mistake. According to technical specifications under number 14-15-254-91 steel rope wire structure must have not less than 30 % of lamellar pearlite and graded as 1 according to GOST 8233-56.

In order to prevent mistakes while choosing equipment it is necessary to introduce to GOST 8233 the following points: the section about equipment and the correspondence table of magnification ration, the aperture of a lens and resolving ability.

Table 1.the correspondence between lenses aperture and resolving power in magnification ratio

Microscope magnification ratio	Numerical aperture value of a lens not less than	Resolving power* with lightning through blue colour-filter (450 nm)
100^x	0.15	2.0 µm
200^x	0.30	1.0 µm
500^x	0.65	0.4 µm
1000^x	1.25	0.2 µm

* The resolving power is a minimal distance in which two points can be distinguished separately

Besides the obvious flaw in GOST 8233-56 is a relatively low quality of photographs in working samples and the absence of size scales in these samples. As can be seen in figure 1 image sharpness in photo-standards leaves much to be desired especially in digital versions. According to the article authors‘ experience in the usage of this GOST on different factories it can be added that photographs in different paper copies of an original edition have different quality and that can be related with out of date photoprinting technology. It is sometimes hard to distinguish principle difference between the first three grades of lamellar pearlite.

The refinement of photo-standards quality can be achieved by means of digital scanning of original photographs (negatives) or by means of microphotograph changes into new ones.

3. Conclusion

It was proved the necessity of evaluation technique refinement of highly dispersed structures. In particularly, it is necessary to introduce a suggestion to Federal Agency on Technical Regulating and Metrology about the change in GOST 8233-56. The change should include requirements to microscope resolving power and to lens aperture with 1000x magnification or it should include a correspondence table of minimal aperture values with different magnification ratios.

It was shown the necessity of photo-standards revision and the necessity of microstructures scales replacement that can be done partly or completely.

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