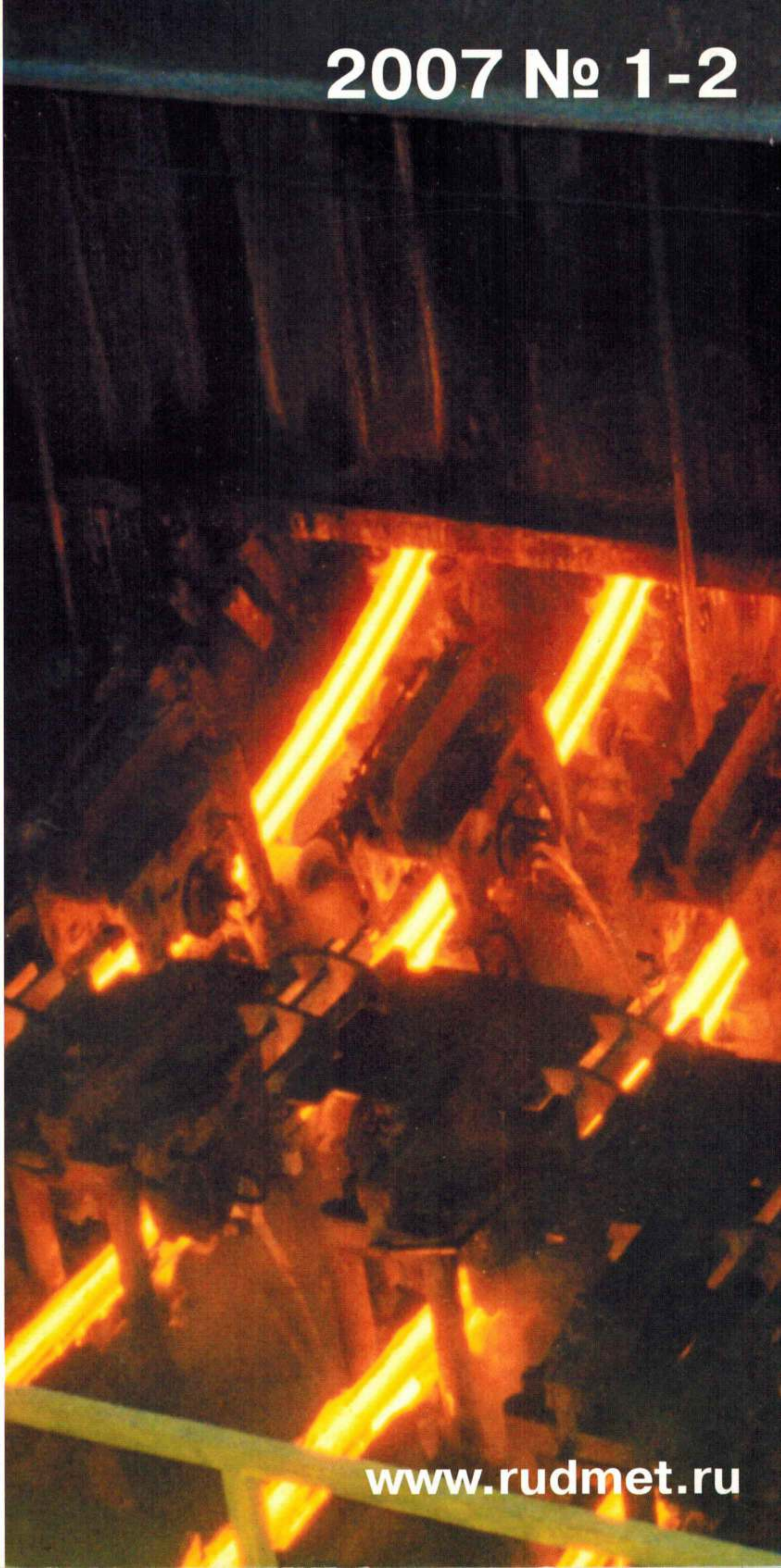
CIS Iron and Steel Review

2007 № 1-2



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- necessary level of safety during exploiting of pipeline systems;
- quality of pipes and tubes as well as oil and gas equipment;
- carrying out the program for protection of the internal market from unfair competition on the side of foreign tube- and pipemakers.

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Metallurgical Quality Characterization of Nickel-based Superalloys

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Abstract

A new image analyzer, Thixomet®, has been developed with the ability to create "panoramic" images of material microstructure for investigations. Capabilities of the Thixomet Image Analyzer for material microstructural characterization are demonstrated with examples of nickel-based superalloys. Practical use of the Thixomet for controlling material properties by controlling their microstructure is demonstrated.

Introduction

One of the most common uses of Image Analysis Systems (such as the Quantimet, Omnimet, Clemex, Leco and others) is a quality estimation of a material's structure. However, it is not uncommon that the capabilities of these image analyzers are insufficient for solving certain specific measurement tasks, especially those involving new and some specific materials as the superalloys. This paper describes the Thixomet Image Analyzer, which has the ability to conduct metallurgical quality control of superalloys structure. The Thixomet system also provides the most commonly used metallographic evaluation methods for nonmetallic inclusions and grains size in accordance with DIN, ASTM and GOST.

The classic publications of the late Ervin E. Underwood, such as his book, Quantitative Stereology [1], were stimulated by his (and several colleagues) efforts to translate the 1958, second edition, of the landmark text of Sarkis A. Saltykov [2] who developed a scientific foundation for stereological methods in materials science in Russia. Saltykov's work has become the foundation for the majority of the quantitative materials science research studies throughout the world. The recent progress in computer hardware design and production, appearance of less expensive powerful devices, like high-resolution CCD cameras and frame grabbers, digital cameras and the advanced development systems have reached the level sufficient for comprehensive digital image processing. Thus, the theoretical foundations of quantitative metallography and image processing [1–4], and the technical progress in computer technologies and digital television have made possible the creation of the Thixomet Image Analysis System.

Thixomet software is a family of products, which includes image analysis software for the quality estimation of the structure of various materials [5–7] and provides solutions for many specific technological problems.

Thixomet Image Analyzer capabilities will be demonstrated using examples of nickel-based superalloys.

Special features of the technology. It is well known that the technology of production of finished parts from superalloys (for example, turbine blades) includes several stages. First, the billets are produced from the original charge materials in large vacuum induction furnaces (VIM). Then the slugs are machined from the billets and used in small induction furnaces for the production of finished parts by investment casting. As a result, a large quantity of scrap results from this method of casting and as a consequence the recycling problem appears. However, the scrap fraction in the VIM charge for billet production should not be above about 30 %, as a rule. It is possible to increase the fraction of the scrap in the charge if it has been previously remelted using active refining melt technology. Therefore, not only the chemistry of the billet, obtained by the refining of the scrap, must be checked, but also the billet's metallurgical quality must be examined before it is used to produce turbine blades.

Principle of estimation method. The known visual estimation by operator method of Willan Metals Company has been taken as a basis. According to this procedure, a specimen is examined metallographically at 200 to evaluate the impurity of the metal by the determination of the impurity rate. The specimen is machined from the bottom part of the cylindrical billet. The surface area to be examined is equivalent to the area of a circle with the diameter of 20 millimeters. The total area of the cross section is observed sequentially, field by field.

The types of superalloy impurities are sorted by their size at 200:

Oxide film (OF) is characterized by its size:

- a "large" film has a size equal to the diameter of the field of view (DFV);
- a "middle" film has a size equal to one-half of the diameter of the field of view; and,
- a "small" film has a size equal to one-fourth of the diameter of the field of view.

Slag globules (SG) and nitride clusters (NC) are characterized by size:

- a "large" one occupies one-tenth of the field of view area (SFV);
- a "middle" one occupies one-fiftieth of the field of view area; and,
- a "small" one occupies one-hundredth of the field of view area.

The total rate is calculated from the following equation with regard to the contribution of each impurity type revealed on n-observed fields of view:

$$R_{\Sigma} = \sum_{i=1}^{n}\sum_{j=1}^{3}\sum_{k=1}^{3}\left(R_{j,k} \cdot N_{j,k}^{i}\right), \qquad (1)$$

where $R_{j,k}$ is the rate coefficient for defect j of k-dimensional group and $N_{j,k}^{i}$ is the number of defects j of k-dimensional group on the field of view i.

The following rates are defined by the expert estimation approach for each impurity type:

- for oxide films and slag globules $R_{j,k}$ is equal 1, 0.5 and 0.25 for large, middle and small defects, respectively;
- for nitride clusters $R_{j,k}$ is equal 10, 5 and 2 for large, middle and small defects, respectively.

It should be noted that the rate for nitride clusters is 10 times higher than for slag globules in the same dimensional group. It depends upon the fact that nitrides and their clusters are more difficult to remove from superalloy melts than slag globules, which are easily assimilated by the refractory lining of the crucible during remelting or by ceramic surface of the filter during pouring.

The Thixomet software has been developed for automation of the measurements required for this approach. Unique methods of quantitative metallography have been successfully applied in Thixomet that result in increased accuracy in determining a defect's dimensions. The defect's size measurements provided in Thixomet are more precise than visual estimation by operator. Therefore, it was possible to use an improved method of impurity estimation by a monotonic function of the defect rate versus its dimension instead of the three-stepped function of the Willan Metals' procedure (Fig. 1)

The strategy for the Thixomet procedure can be summarized as follows. The images with metallurgical impurities (oxide films, slag globules and nitride clusters) are transmitted through the CCD camera and frame grabber to the image analyzer monitor. The sharpness adjustment has to be carried out so that the carbides and nitride clusters have uniformly colored surfaces. Then, the image is captured.

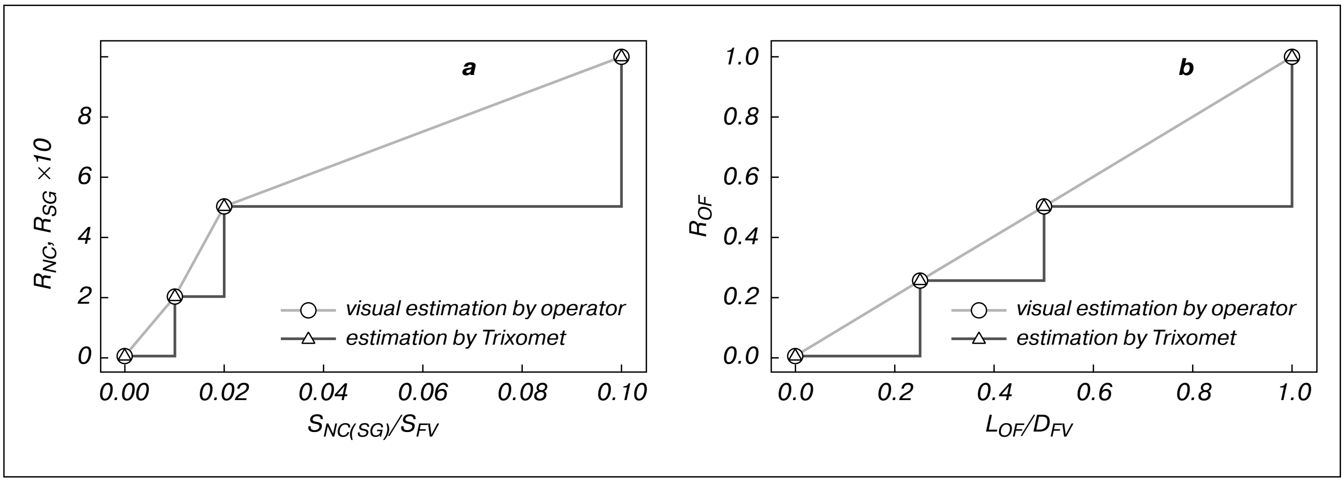

Fig. 1. Defect rate versus its dimension for nitride clusters, slag globule (*a*) and for oxide film (*b*)

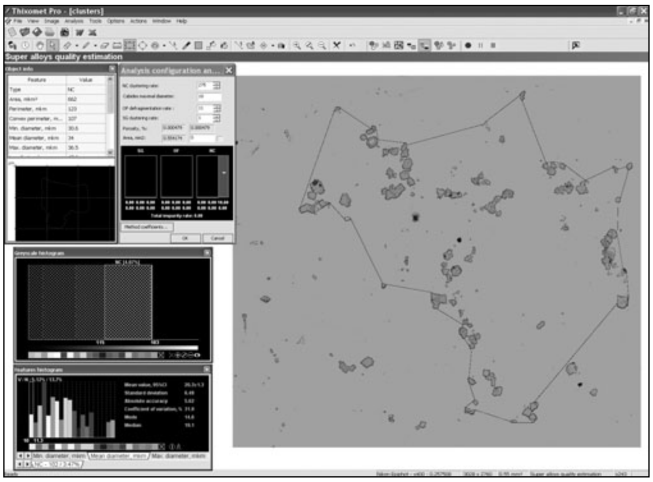

Fig.2. Thixomet screen. Estimation of impurity rate

As a rule the metallurgical defect does not fit one field of view, captured by camera. Decreasing the microscope magnification is impossible because it may lead to loss of resolution necessary for correct identification of the defects and precise measurement of their sizes. To overcome this problem, a special procedure was developed in Thixomet for the reconstruction of the full defect image by sequential capture and perfect matching of its segments "in flight". That is, while the microscope stage is moving to the next contiguous field of view, the present field of view is matched with high precision, "pixel to pixel", to the previous one. A microstructural montage of segments into a full panoramic image of the defect is carried out automatically according to a specially developed algorithm based on pattern recognition ability of Matrox Imaging Library. Creation of the full panoramic image of the defect can be observed at the Thixomet main window (Fig. 2).

Defect identification is performed according to the complex criteria that includes information on the grayscale level of the objects, their morphology and the nature of mutual arrangement. The grayscale level for each defect is pre-adjusted. For this purpose, there are threshold levels on the Thixomet control panel. Carbide and nitride inclusions have close grayscale levels, so they can not be separated using this criterion. To separate them, the character of their mutual arrangement, size and morphology are estimated. The carbides, which are rather fine and uniformly distributed across the cross section, are automatically removed from the analysis.

The image processing results are presented as a bar chart, which depicts the quantity for each type of defects. These bar charts are displayed in one of the operating windows of the Thixomet. The total impurity rate of the investigated sample, as well as specific rates for each type, is shown here as an accumulated sum (Fig. 2).

Film elongation is calculated as for a continuous linear feature; in this case the breaks in films are connected. The area occupied by slag globules and nitride clusters is estimated by the area of the figure, which is formed by the external boundary of the defect conglomeration. When evaluating the area, occupied by nitride clusters, the user is able to modify the clustering rate either by joining the inclusions to the cluster or by integration/separation of neighbor clusters (Fig. 3, *b*, *c*). The adjustment of size and quantities of slag globules conglomerates is carried out in the same way.

Another useful procedure of Thixomet corrects some of the defects in images. This procedure removes the scratches and corrects the nonuniform illumination present in optical systems. It is automatically performed in real-time mode before each measurement (Fig. 3, *b*, *c*).

The decision to use the charge billet in the production of turbine blades is based on metallurgical quality estimation results. Billets with an impurity rate more than a critical value have to be remelted using the refining technology. The critical value of the impurity rate for the charge billet is established by additional technical documentation.

So, the main capabilities of Thixomet are the following:

1. precise matching of the images with each other, obtained by the successive import of adjacent fields of view, permits:

- creation of a panoramic image;
- high resolution capacity while investigating the large area of the object;
- new possibility for quantitative analysis of the material structure without excluding marginal objects;

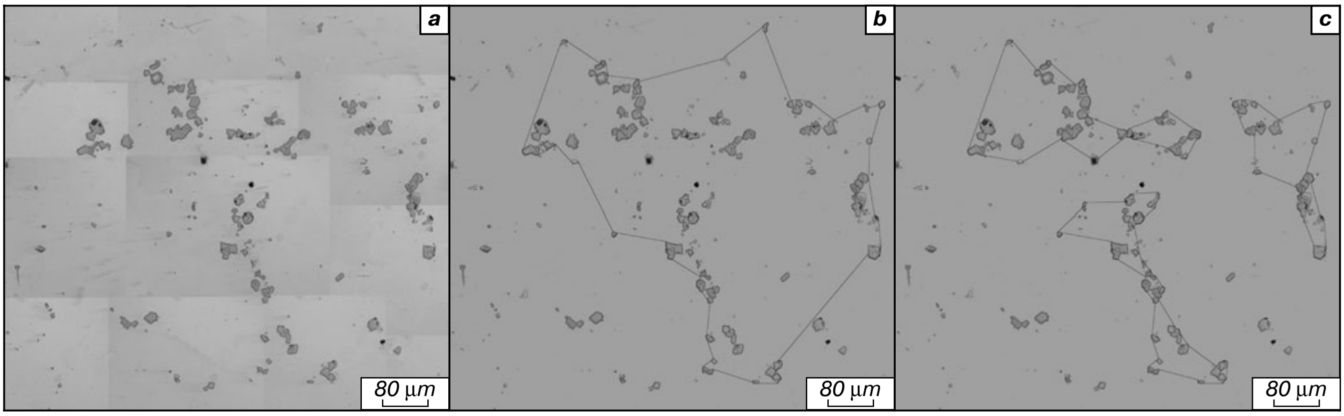

Fig.3. Nitride cluster identification

a **— original image automatically matched from 25 field of view (Previous image processed by leveling procedure);** ***b*** **— identification of nitrides as one big cluster; c — separation of neighbor clusters**

2. leveling of nonuniform illumination of the reflection light microscope image and removal of scratches from the surface of cross-section;

3. identification of structure objects using one or several criteria, e.g., grayscale level, morphology factors, and the pattern of the mutual arrangement of the objects; and,

4. estimation of total and specific impurity rates.

The additional capabilities of Thixomet include the estimation of porosity content in blade test sections and manual measurements operations of the features.

Improvement of the technology by controlling microstructure. The Thixomet Image Analyzer have been used for metallurgical quality estimation of all billets used in the production of turbine blades at St.-Petersburg Turbine Blades' Plant (Russia) since 1998, Joint-Stock Company "Kazan Motor Building Production Association" (Republic of Tatarstan) since 2001 and Federal State Unitary Enterprise "MMPP "SALUT" (Russia) since 2006. Those many years of experience of the Thixomet application under the plant conditions has demonstrated it's high performance and usability. Rejects of turbine blades have decreased because of elimination of charge billets with insufficient metallurgical quality from production. Moreover, the analysis of structure quality estimation results has made it possible to obtain new technological information. This information could be used for the analysis of production state as well as for the development of technology improvement methods. For example, the availability of specific impurity rates allows the correct refining technology for a given batch of billets to be found. So, high-temperature treatment of the melt should be recommended for billets with a high rate of nitride cluster impurities to dissolve the nitrides and to denitrate the melt. Filtration refining of the melt should be recommended for the billets with high rate of slag globules and oxide films.

Furthermore, while speaking about the nitrogen refining of the melt, it should be mentioned that currently the consumers of superalloys impose limitations on nitrogen contents in charge billet to 0.002 wt. %. But there are carbides with extremely unfavorable shape (i.e., highly elongated or as "Chinese hieroglyphs") within billets with such low nitrogen contents. Carbides of this type are inherent in massive sections of the blades and induce polishing cracks during further mechanical processing. As shown in our earlier studies [5], precise modification of the melt by nitrogen makes it possible to transform carbides with unfavorable morphology into equiaxial, well-dispersed carbonitrides, thus modifying the cast structure. As this takes place, the lifetime of finished turbine blade increases by 1.5 or 2 times under normal service conditions. Thus, the charge billet has to be controlled both in the metallurgical quality and the morphology of the carbides. As for the billets with unfavorable morphology of the carbides, precise nitrogen modification of the melt before blade production is recommended.

Summary and Conclusion

The Thixomet family of products for characterization of the materials structure have been developed. Capabilities of Thixomet have been demonstrated with examples of metallurgical quality estimation of nickel- based superalloys. Thixomet is used at the plants produced the products from superalloys for their quality management.

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