Int. J. Appl. Math. Comput. Sci., 2008, Vol. 18, No. 1, 33–39
DOI: 10.2478/v10006-008-0003-1

IMAGE BASED ANALYSIS OF COMPLEX MICROSTRUCTURES OF ENGINEERING MATERIALS

TOMASZ WEJRZANOWSKI, WOJCIECH LEON SPYCHALSKI, KRZYSZTOF RÓŻNIATOWSKI, KRZYSZTOF JAN KURZYDŁOWSKI

Warsaw University of Technology
Faculty of Materials Science and Engineering
ul. Wołoska 141, 02–507 Warsaw, Poland
e-mail: {twejrzanowski,wspych,krozn,kjk}@inmat.pw.edu.pl

The paper presents various methods for quantitative description of material structures. The main focus is on direct methods of description based on image analysis. In particular, techniques for the estimation of the size, shape and spatial distribution of structural elements observed by different microscopic techniques are described. The application of these methods for the characterization of the structures of engineering materials is demonstrated on a stainless steel used in petrochemical installations. It is shown that the methods applied are useful for the assessment of service degradation of materials.

Keywords: Image analysis, quantitative description, microstructure.

1. Introduction

Many engineering applications require materials which are able to resist many forms of static or dynamic forces and arduous service conditions such as variable dynamic and static mechanical loadings, high operating temperatures, corrosion, etc. However, it is well known that the properties of materials are related to their structure. One of the most famous relationship was proposed by Hall and Petch about sixty years ago (Hall, 1951; Petch, 1953). They found experimentally that the flow stress of metals at relatively low temperature is a function of the average grain size. Later, a similar relationship was deduced between the hardness and the average grain size. There is also evidence that the shape of structural elements may have influence on materials properties such as resistance to cracking (Spychalski *et al.*, 2002).

Since the properties of a material directly depend on its structure, it is reasonable to create and/or quantitatively characterize the structure possessing desirable parameters. To this end, it is necessary to use techniques which allow investigating structural elements (grains, particles) in terms of features such as the volume fraction, number, size, shape, orientation and spatial distribution.

2. Methods

Quantitative description of a material microstructure can be achieved with two different techniques. The first one can be called an "indirect technique", where the structural parameters, such as the grain size, are estimated from measurements of other parameters. The second type, a "direct technique", enables the structural parameters to be obtained directly by a measurement technique. The most popular technique of the first type is X-ray diffraction (Kril and Birringer, 1998). In this method the average grain size can be measured from periodic multiplication of the lattice parameter. However, a sufficiently correct estimate of the average grain size can be obtained only for polycrystals free from residual stresses and morphological texture.

Direct techniques, usually based on microscopic investigations, can be employed for extended studies of a material microstructure. The application of such methods is limited by the fact that they have a destructive character. However, some modification allows service inspections using mobile optical microscopes (see Fig. 1). Thus, structural investigations of materials in industrial installations are virtually non-destructive.

A typical analysis of a material structure requires the following stages (Kurzydłowski and Ralph, 1995):

- preparation of the surface of the material (cutting, grinding, polishing, etching),
- microscopic observation and image collection,
- image processing,

- quantitative image analysis,
- interpretation of results.

Fig. 1. Mobile system for microscopic observations of material structures.

The first two stages have to be adapted for particular situations but the others can be unified for many applications.

The quality of the images obtained during the inspection of an industrial plant is usually poor. Very often, it is not possible to recreate the real network of grain boundaries so the lines representing grain boundaries are drawn manually. Much better results can be obtained by processing images with features (i.e. inclusions) which are dispersed in space.

The final image after processing separates the objects of interest from the background. Very often it is a binary image. There are also some useful transformations made on binary images (Russ, 1999). Examples of such transformations are illustrated in Fig. 2.

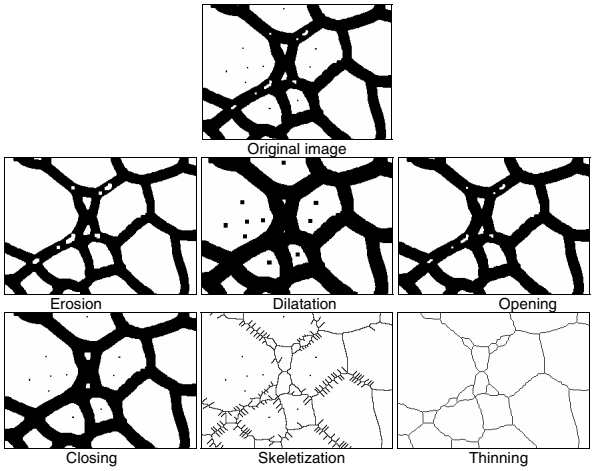


Fig. 2. Example of morphological transformations usually used in the processing of structural images.

The most important stage in terms of structural characterization is quantitative image analysis. This image analysis makes it possible to compute the parameters which describe the structure globally or describe the features of individual elements.

Among the global parameters, the following ones are frequently used:

- V_V, volume fraction (applicable for multi-phased materials),
- S_V, surface per unit volume,
- $\bar{V}$, mean value of the grain volume,
- N_V, number of elements (grains, particles) per unit volume.

The volume fraction for an isotropic and homogenous material can be replaced by the aerial fraction, A_A, which is determined by counting the pixels which have registered the particular phase in the image.

The geometry of grain boundaries can be quantitatively described using principles of stereology and image analysis. From the stereological point of view, grain boundaries form an inter-connected set of curved 2-D objects. The total surface area of such objects in unit volume, S_V, can be estimated using a relatively simple technique illustrated in Fig. 3. As can be seen, this technique is extremely simple in the case of grain boundaries which are oriented randomly in space. In the case of boundaries exhibiting a measurable degree of anisotropy (morphological texture), a slightly more complicated method, which is based on the principle of vertical sectioning introduced in (Baddeley *et al.*, 1986), is required.

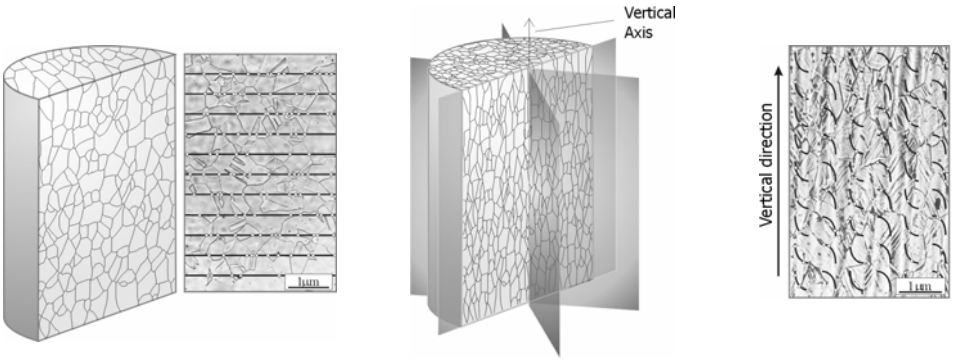
Stereological arguments show that the surface of the grain boundaries in a unit volume can also be used to estimate the mean intercept length, $\bar{l}$, which is used in a pile-up model of the grain boundary effect on the flow stress of polycrystals (Armstrong, 1983). Geometrical probability theory shows that the mean intercept length of grains is proportional to $(S_V)^{-1}$:

$$\bar{l} = \frac{2}{S_V}. \tag{1}$$

In investigations focused on grains, rather than on grain boundaries, the most natural size parameter is the grain volume, V. The most popular methods used for the determination of the mean value of a grain volume are called:

(a) di-sector,
(b) point sampled intercepts,
(c) Saltykov reconstruction.

The di-sector technique is illustrated in Fig. 4. In this case, given a usually small volume of the specimen, we count the grains which are either entirely within this volume or are cut only by half of the external surfaces. The density of



(a) (b) (c)

Fig. 3. Measurements required for an unbiased estimate of the surface area of grain boundaries in a unit volume of a material: (a) isotropic geometry of grain boundaries, (b) vertical sectioning, (c) cycloids used in the anisotropic case.

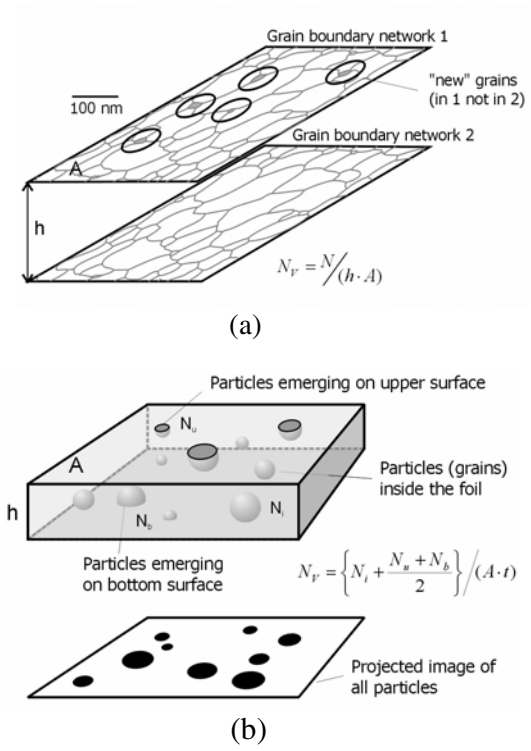


Fig. 4. Explanation of the di-sector measurements in the case of observations on sections (a) and projections (b).

grains can be subsequently obtained by dividing the number of the counted grains, N, by the studied volume of the specimen, V_o:

$$N_V = \frac{N}{V_o}. \tag{2}$$

Once the value N_V is known, $\bar{V}$ is calculated as

$$\bar{V} = \frac{1}{N_V}. \tag{3}$$

The concept of point-sampled intercepts was introduced in (Jensen and Gundersen, 1982). Its implementation is explained in Fig. 5. It should be noted, however, that this method systematically overestimates the value of $\bar{V}$. The difference between a true estimate of this parameter and the one with point-sampled intercept is higher for structures exhibiting a large degree of non-homogeneity of the grain size.

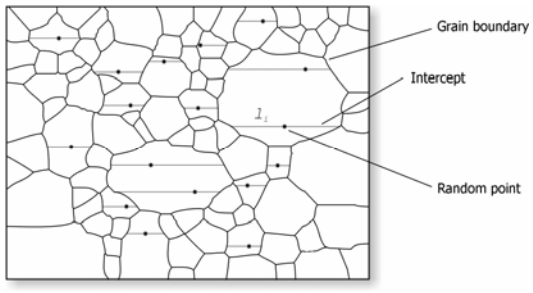


Fig. 5. Implementation of point-sampled intercepts.

Saltykov's reconstruction can be used to obtain a full distribution function of the grain volume, $f(V)$. However, this method requires the shape of grains to be specified. If this shape is fairly regular, one may assume that the geometry of grains can be approximated by a Kelvin tetrahedron. This, in turn, leads to a system of algebraic equations which links the relative frequencies of grain sections/projections and the grain volume, see Fig. 6 (more can be found in (Kurzydłowski and Ralph, 1995)).

The commonly used parameters describing the structure are as follows:

- d_2, equivalent diameter,
- A, area,
- $d_{\max}, d_{\min}$, maximum and minimum chords,
- $\theta_{\max}$, orientation of the maximum chord.

The parameters A and d_2 describe the size of a particular element, since $d_{\max}$ and $d_{\min}$ are useful in shape determination. $\theta_{\max}$ expresses the orientation of an element. The interpretation of these parameters is given in Fig. 7.

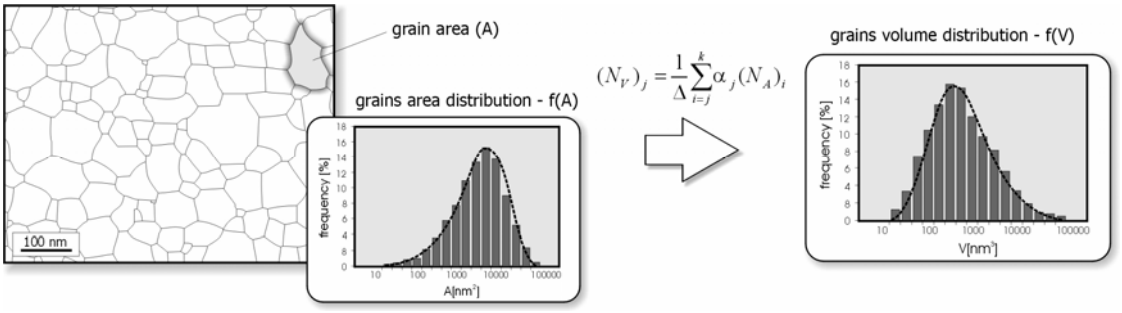


Fig. 6. Principles of Saltykov's reconstruction.

The values of parameters obtained for each individual element involve the distributions and statistical moments, such as the mean, $\mathrm{E}(x)$, and the standard deviation, $\mathrm{SD}(x)$, where x represents a given factor to be calculated. Very often the coefficient of variation, $\mathrm{CV}(x)$=$\mathrm{SD}(x)/\mathrm{E}(x)$, is used to describe the size and shape dispersion.

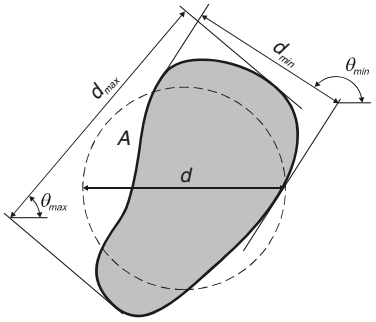


Fig. 7. Parameters describing structural elements.

The shape of grains can be relatively simply quantified by measurements carried out on sections and projections of grained structures. The ratio of the grain's longest chord, $d_{\max}$, and its perimeter, p, to the equivalent diameter, d_2, can be computed using image analyzers. The ratios $d_{\max}/d_2$ and p/d_2, called shape factors, are sensitive to grain elongation and convexity, respectively, cf. Fig. 8.

The orientation of an element is represented by the angle $\theta_{\max}$. The frequency distribution of the angles creates the so-called "rose of directions", see Fig. 9. This makes it possible to determine whether a structure is isotropic or there is some directional anisotropy in terms of element morphology.

The arrangement of specific grains within the volume of the material can be investigated using the concept of the covariance function, $c(r)$ (Susagna *et al.*, 2000). This function defines the probability of the event that if a given randomly placed point hits such a grain, a point placed at a distance of r also hits a grain of that kind (including the grain hit by the first point itself). Some properties of the covariance function are demonstrated in Fig. 10. The

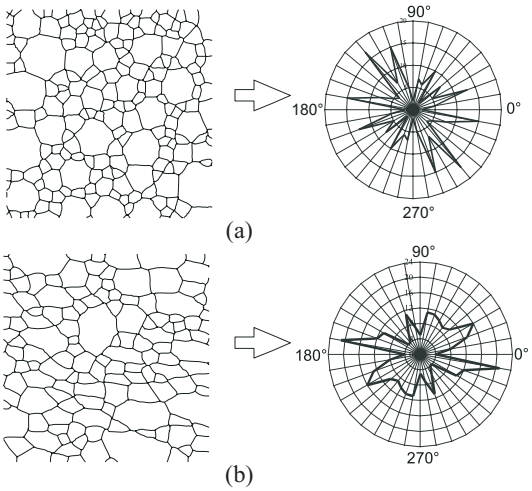


Fig. 9. Rose of directions for a material without and with a morphological texture, (a) and (b), respectively.

existence of a maximum value of $c(r)$ for $r > 0$ indicates that the analyzed grains tend to be dispersed with some characteristic distance with respect to others.

3. Exemplary application

Some methods presented in this paper have been applied to quantitative description of structural degradation of a material from a petrochemical catalytic cracking installation. Catalytic cracking is the process where complex organic molecules such as heavy hydrocarbons are broken down into simpler molecules (e.g. light hydrocarbons) by the breaking of carbon-carbon bonds in the precursors. The processes take place in the reactor at the required working temperature of $550°$C at a pressure of 0.344 MPa. The scheme of the cracking reactor, showing the regions where the measurements were made, is presented in Fig. 11.

The investigations were made in 2004 and 2007. The microscopic observations were performed using a mobile NIKON microscope (see Fig. 1) on the surface of the reactor elements. The surface preparation included grinding,

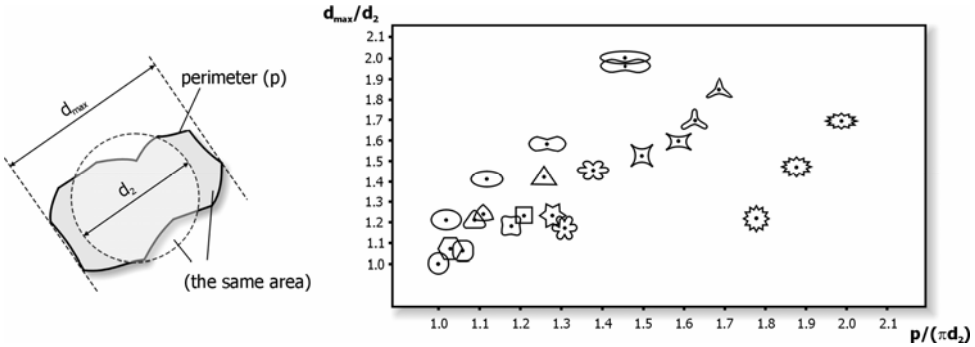


Fig. 8. Measurements of grain shape factors and their interpretation.

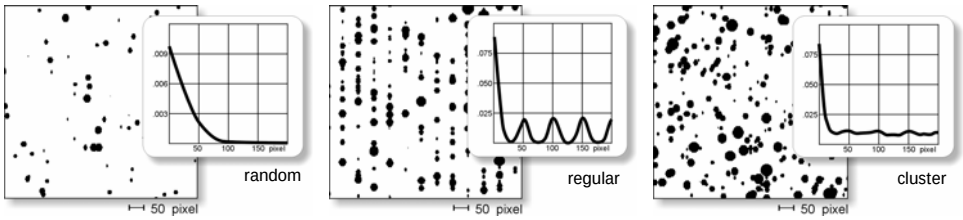


Fig. 10. Explanation of the properties of the covariance function.

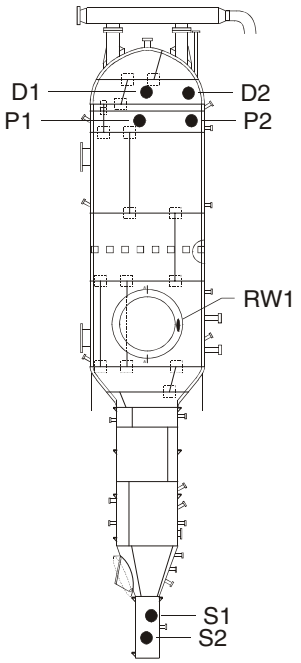


Fig. 11. Location of measured regions.

polishing and etching with a 4% solution of Nital (Polish standard PN61/H-04502).

The microstructure of materials was examined at the magnifications of 100 and 500. The images were acquired by a digital camera mounted on the microscope.

The microscopic observations showed that each section of the reactor was made of different materials. The upper part of the reactor exhibited a ferrite structure with carbide inclusions (regions $D1$, $D2$, $P1$, $P2$, RW). The bottom region ($S1$, $S2$) had a ferrite and pearlite structured material.

Typical images of the structure of materials examined in 2004 and 2007 are presented in Figs. 12–15.

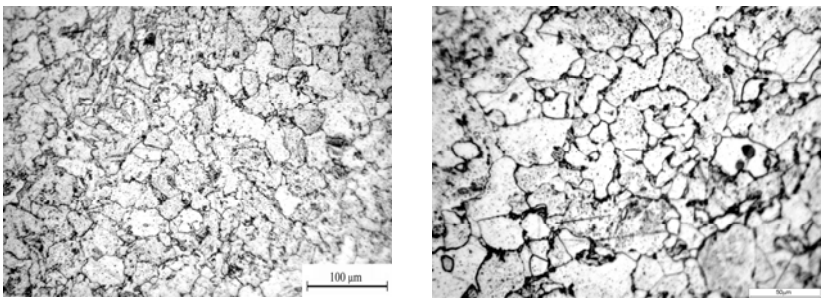
Quantitative microstructure description was achieved using the MicroMeter software (Wejrzanowski, 2000). Special attention was devoted to the analysis of the size and shape of grains. The size was characterized by an equivalent diameter coefficient, d_2, and the shape was described by the elongation factor, $d_{\max}/d_2$. The average values of these parameters, obtained for the materials from different regions of the reactor, determined in 2004 and 2007 are presented in Figs. 16 and 17, respectively.

The results show that there are no significant changes in the grain size in the material of upper regions of the reactor. The average size was about 30 μm. However, a noticeable difference in the grain size was found in the bottom region. Since the hardness measurements did not reveale significant changes, a sub-inspection was proposed for this area after a further year of service.

It was found that the elongation factor remained at the same level ($d_{\max}/d_2 \approx 1.4$) in all regions. This value is typical for non-elongated grains.

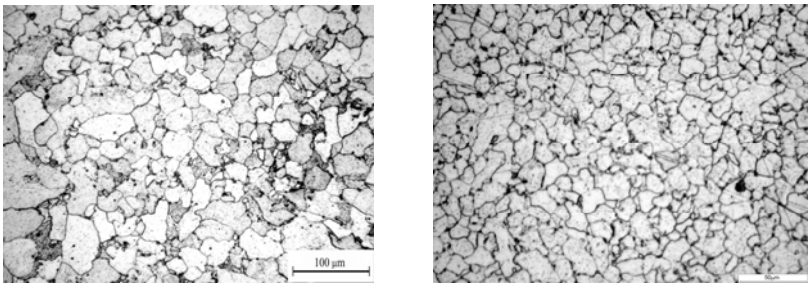
4. Summary

The paper gives an overview of methods useful for quantitative description of the structure of metallic engineering materials. It has been shown that several microscopic



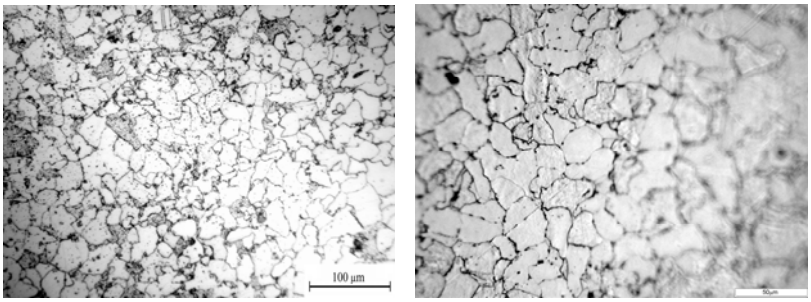
(a) (b)

Fig. 12. Typical microstructure observed in the regions $D1$ and $D2$: (a) year 2004 and (b) year 2007.



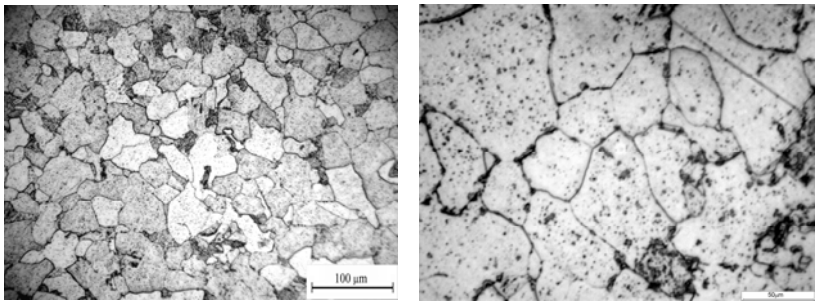
(a) (b)

Fig. 13. Typical microstructure observed in the regions $P1$ and $P2$: (a) year 2004 and (b) year 2007.



(a) (b)

Fig. 14. Typical microstructure observed in the regions RW: (a) year 2004 and (b) year 2007.



(a) (b)

Fig. 15. Typical microstructure observed in the regions $S1$ and $S2$: (a) year 2004 and (b) year 2007.

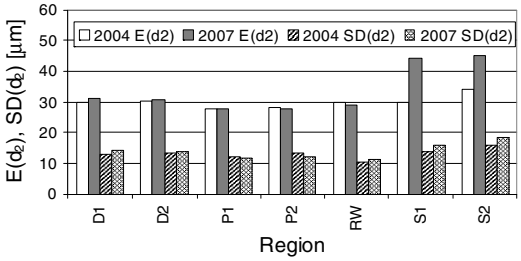


Fig. 16. Size dispersion of grains in the structure of materials from the reactor inspected in 2004 and 2007.

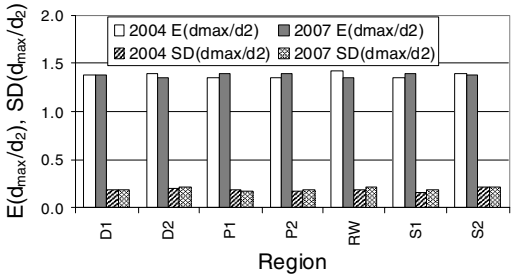


Fig. 17. Elongation of grains in the structure of materials from the reactor inspected in 2004 and 2007.

techniques supported by image analysis based methods provide advanced tools for the calculation of the grain size, shape and other parameters, which may have impact on material properties.

A variety of techniques available allow measurements in adverse industrial conditions, as well as in a laboratory environment, to be performed. The results of the investigations carried out on the petrochemical installation established the ability of the methods to identify structural degradation induced by the service conditions encountered over an extensive period of operation.

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