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Image analysis in quantitative metallography

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ABSTRACT

Quantitative metallography (QM) deals with the relationships between multi-dimensional features of a metallographic sample and its projection/intersection on a two dimensional plane.Subsequently QM can be related to other properties of materials. Number of parameters to be measured for QM are numerous and time consuming and this has promoted the development of automatic image analyser by which, even, the routine quantitative measurements can be done easily and quickly. So, for quality control of industrial products, it plays an important role. With the availability of reliable automatic image analyser equipment, the quantitative characterisation of a metallurgical samples becomes easier and it takes less amount of time. At the same time reliable data can be generated with suitable software and hardware attached with modern microscope. The analysis modules of a metallurgical sample are broad and they are - grain size analysis, inclusion rating, percentage, volume & area fraction, porosity, particle size, image features etc. In this review article the importance of image analyser for the quantification of a polished and etched surface has been discussed and some of the applications for quantitative metallography of metallurgical sample have also been highlighted.

INTRODUCTION

The aim of quantitative metallography (QM) is to get features of three dimensional effect from the measurement of two dimensional objects of a polished and etched metallic or alloy surface. The three-dimensional features are shown to be related with physical and mechanical properties of materials. In practice, these expected properties are never attained but may be approached with any desired degree of accuracy by simply increasing the number of measurements[1,2]. Care must be taken that the observed samples must be representative. Modern image analysis system has become an important tool for quantitative measurement of microstructural aspects.

The application limits of a material are becoming to narrow and more specific, so it is necessary to specify and control the microstructure quantitatively, though the improvement of metallurgical smelting processes have led to the production of very pure metals and alloys. The visual evaluation of a polished section of such materials has become increasingly more difficult and time consuming. As a result, efforts have been made both to improve the methods of specimen preparation, as well as to automate the process of evaluation. The QM with modern image analysis plays an active role in the industry for controlling the quality of their products. The statistical out-put from an image analysis system becomes essential for total quality control (TQC).

In the year 1964 the first instrument Quantimet B was available for QM. Though, it had some problems relating to light source, insufficient automation and poor linearity that prevented the instrument from being used for routine checking in the industry. In 1970 the third generation image analyser came into the market which was also mainly employed for research purposes. Even, these were not fast enough to carry out routine test work. For day to day quality control, the image analysis system was employed only after modern economical computer became available. Towards the end of 1977 a special version of Quantimet was available for the first time for the routine determination of inclusions in steels. In the year of 1983, a modern image analyser TAS-Plus with modern auto-focusing system came to the market.. In additions to measuring the purity and grain sizes of specimen, it also measures others parameters like percentage, volume and area fraction and distribution of phases, image features, porosity, particle size, distribution analysis etc[3-5].

BASIC MEASUREMENT FOR QUANTITATIVE METALLOGRAPHY

The basic measurement can be done by using areal ratio, linear ratio and point ratio of selected objects on a microstructure. The measurement of volume fraction can be obtained through some basic equations (mentioned in the section 3) from any of the above mentioned measurement ratio.

The point count method refers to the measurement of number of points that fall on a specific features of a microstructure. The ratio (P_p) of number of points that fall on a specific phase (P_a) by the number of total points (P_T) gives the quantitative measurement. An application of point-count measurement to a graphite nodules in ferrite matrix is shown in figure 1[6]. Again, the number of intersection per unit length of a line (drawn on a microstructure) also gives the quantitative measurement of phases. Here a test line or linear array is drawn randomly on a microstructure containing linear features. The points of intersection along the test line are counted.

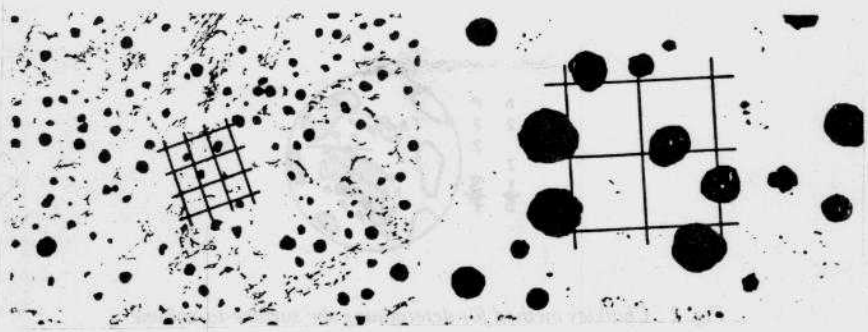

Fig. 1 : Application of point-count grids to graphite nodules in the ferrite matrix of grade 60-45-12 ductile iron. (a) 2% nital 100X (b) 2% nital, lightly etched 140 X

To measure the surface to volume ratio of discrete particles (or phases) on a specimen, the equation can be used as following[7] :

$$\frac{S_a}{V_a} = \frac{4P}{hl} \qquad (1)$$

where, S_a and V_a are mean particle surface area and volume, P = point of intersection of the boundary, l = lenth of test line drawn randomly on the microstructure and h = the end point that hit within the 2nd phase. A schematic presentation is shown in figure 2 which explain how a surface area can be converted to volume fraction. Again, using superimposed square grid on a microstructure the following equation may be employed for surface to volume conversion[7].

$$\frac{S_a}{V_a} = \frac{2P_L}{P_P} \qquad (2)$$

where, P_P and P_L are point count and intersection count respectively. Example is shown in figure 3[6, 8].

For a system, with various particles in a matrix the equation will be in the form of

$$\frac{(S_V)_a}{(V_V)_a} = \frac{2P_L}{P_P} \qquad (3)$$

where, S_V = surface or interface area divided by total test volume (surface to volume ratio) and V_V = sum of volumes of structural features divided by total test volume.

Fig. 2 : Chalkley method for determining the surface-to-volume ratio of discrete particles.

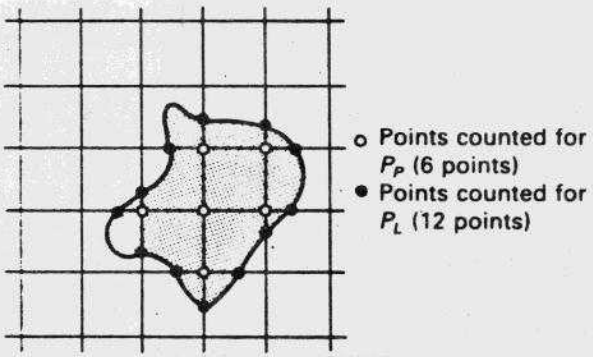

Fig. 3 : Superimposed square grid used in the Saltykov method for determining the surface-to-volume ratio of discrete particles. P_P represents a point count and P_L represents on intersection count.

BASIC EQUATIONS FOR QUANTITATIVE METALLOGRAPHY

The three basic equations which are mainly employed for the quantitative measurement of a metallic samples, are as follows[6,3,9] :

$$V_V = A_A = L_L = P_P \quad (4)$$

$$S_V = (4/\pi)\, L_A = 2\, P_L \quad (5)$$

$$L_V = 2\, P_A \quad (6)$$

where, A_A = sum of areas of intercepted features divided by total test area, L_L = sum of linear intercepted lengths divided by total test line length, L_A = linear traces on the plane of polish surface, L_V = length of features per test volume and P_A = number of point feature divided by total test area.

Equation (4) show the equality of volume fraction to areal, linear and point ratio of the selected phases on a matrix. When surface area per unit volume and linear traces on microstructure are of interest the equation (5) can be used. For example, inter-phase boundary area (S_v), length of grain boundary traces can be measured by this equation. In case of estimating linear elements on microstructure (like dislocation lines, grain edges where three adjacent grains contact, needle like precipitate particles and slag or oxide stringers) per unit volume is necessary to measure from points counted on the microstructure, then equation (6) can be employed. A typical example is shown in figure 4 where, to get dislocation density the number of dislocation etch pits is used.

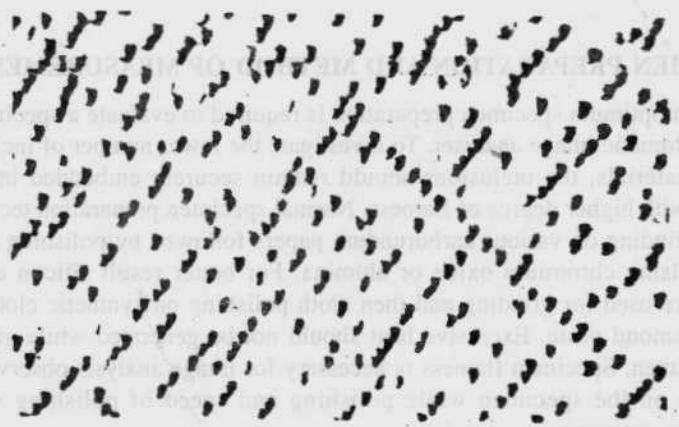

Fig. 4 : Etch pits due to dislocation lines in copper

WHAT IS AN IMAGE ANALYSER AND HOW IT WORKS?

Image analyser is primarily concerned with the extraction of useful information and data from an image, which is the ultimate product of numerical output. In an image analyser, the images are digitised which provides description of complex scene in simpler way from a very large and often ill-defined objects. Image manipulation can be done by which the representation of images are more appealing and more understandable. It consists of a microcomputer to process images. It is user friendly and can be operated manually or automatically by using suitable software. Its performance also depends on operator's knowledge and how the operator relates the objects to one another and to the back-ground[6,10].

The basic operations are as follows[11]:

- Image input or captured,
- Digitisation of images,
- Selection of different grey levels,
- Segmentation of images into binary regions of interest,
- Reprocessing of binary images,
- Defining and extraction of specific features.
- Mode of extraction,
- Measurement of selected parameters and
- Data output/stored.

SPECIMEN PREPARATION AND METHOD OF MEASUREMENT

The most optimum specimen preparation is required to evaluate a specimen by using automatic image analyser. To investigate the lower number of inclusions in the materials, the inclusions should remain securely embedded in metal surface with higher degree of flatness. Normal specimen preparation technique is that grinding on various carborundum papers followed by polishing on felt clothes using chromium oxide or alumina. For better result silicon carbide papers are used for grinding and then cloth polishing on synthetic clothes by using diamond paste. Excessive heat should not be generated while grinding the specimen. Specimen flatness is necessary for image analysis observations. Pressure on the specimen while polishing and speed of polishing wheels should be optimum.

The actual configuration and arrangement of an image analyser is not described here. Some points which are of practical importance for the analysis have been described. Time requirement for the entire operation on an image analyser is high. So, the stress is also given to reduce the time obligation for the analysis in addition to improvement of the techniques and quality. Entire time requirement for a measurement can be divided into three stages.

- Operation of specimen table,
- Focusing of the images and
- Determination of the parameters to be measured and data analysis.

Specimen table must operate quickly and smoothly and remain free of all kinds of vibration. The movements may be programmable. As numerous parameters to be measured and a large amount of data to be generated, proper auto-focusing helps to minimise the time requirement of over all operation.

Higher memory capacity of a powerful microprocessor can be introduced to an image analyser, by which the time needed for data analysis can be reduced considerably. At present grey scale and shape are main parameters for quantification. In future the colour differentiation may be an additional parameter to distinguish various microstructural constituents.

APPLICATIONS

Grain Size Measurement

Measurement of grain size is one of the most oldest and important techniques for QM as it influences many properties of metals and alloys. There are several methods which have been for the measurement of grain sizes or diameters though the definitions of diameter is usually arbitrary. These methods are intercept or plainmatric or by chart comparison.

In the intercept method, developed by E.Heyn in 1903, is based upon the number of counts by a line (the line may be either straight or curved or circular) of either the grain or grain boundaries. The plainmatric method, developed in the year of 1916, is based upon on the determination of the number grains per unit area (n) which can be directly converted to average ASTM grain size by[12-15]

$$N = \frac{\log n}{\log 2} + 1.0000 \qquad (7)$$

where, n = number of grains per square inch area at 100X magnification. Normally, to obtain a reasonable ASTM grain sizes, 50 grains in each of a 3 areas must be counted at 100X magnifications. In the chart comparison method, the experimental microscopic images are compared to a set of standard ASTM chart [ASTM E 1382-91 / ASTM E112). A standard ASTM grain size chart is shown in figure 5[16].

For many random planes, of course, the average intersection values are true representative of a 3-diamensional parameters. The mean intercept length is defined as :

$$L = \frac{1}{N_L \text{ (or } P_L)} = \frac{L_T}{PM} \qquad (8)$$

where, N_L = number of intercept per unit length, L_T = total test length, P = number of grains or number of grain boundary intersection and M = magnification.

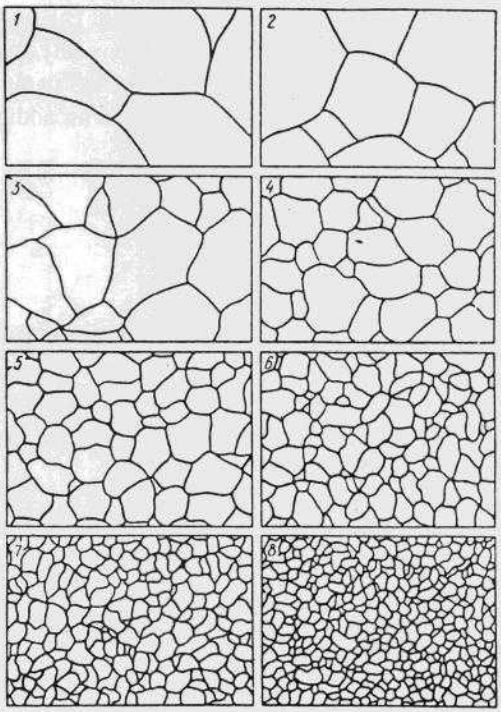

Fig. 5 : Standard classified grain sizes for steel (grain size numbers are indicated). X 100 (reduced to 3/5 size in printing)

Now-a-days, in most of the advanced laboratories, image analysis systems are in use for quantitative metallography. First, it transfers the incoming images in to a form which can be easily analysed and this process is known as "transformations" or "convolutions" or convolvers" and present grey level operations are employed for the quantification of multiphases in a polycrystalline materials. For a greater degree of accuracy a better input images are required. A modern image analysis system equipped with better hardware and software, an image like figure 6 can be transformed to a image like figure 7(a,b)[18] through convolution. Using various analytical techniques, each grain size groups can be sorted out by using a modern IA system. Furthermore, once grains have been sorted in grey range or colour groupings by their grain size values, they can be further classified as to the number and area percentage of each grain size range in the field of view. These detailed analysis provide a better characterisation of microstructures than was ever possible before.

Fig. 6 : Tantalum sample polished and etched. Note lack of highly defined grain boundaries 600X

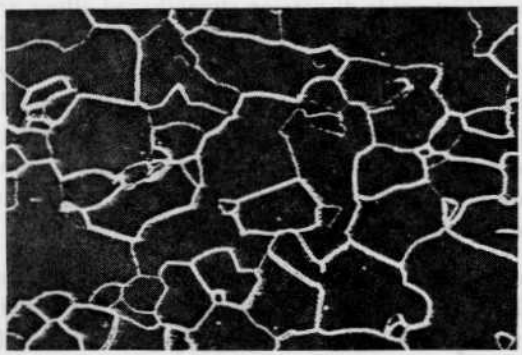

Fig. 7(a) : Same field of view as shown in Fig. 6 after application of Kirsch filter which highlights discontinuities, in this case, the grain boundaries

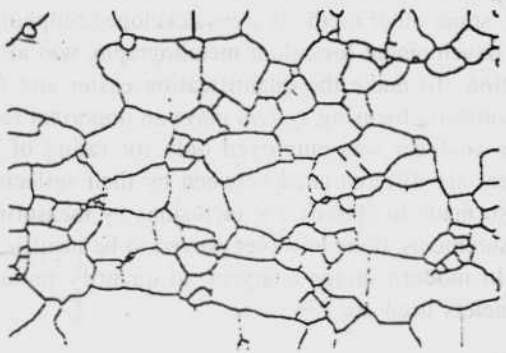

Fig. 7(b) : Same field of view as Figures 6 and 7a after application of a series of image processing steps to reconstruct grain boundaries, compare with Figure 6.

Shape and Size Distribution of Grains

In some circumstances of industrial practice, it can be observed that the two specimens of a given material can behave in different ways though their equivalent mean grain sizes are same. That difference may frequently be quantitatively related to grain size distribution around the mean value. The importance of size distribution is felt where there are existing heterogeneous distribution of grains resulting in the inconsistency of mechanical properties. Criteria are available for admissible degree of heterogeneity to avoid the deleterious influence of the presence of coarse grains in a finer microstructure. In case of hot rolling there may be, in some cases, partial recrystallization which results the formation of heterogeneous grains[18].

Inclusions

Except few cases (like oxide dispersion strengthened alloys and free machining steels), it is well established that inclusions play a detrimental role in the performance of a material depending on their type, shape, size and distribution. It is defined as particles of foreign materials in a metallic matrix. The particles are usually compounds such as oxides, sulphides or silicates and insoluble in the matrix. Inclusions are relatively small, their volume fraction are low, they are not randomly distributed, and they deform at different rates than the matrix during working..

The oldest procedure for rating of inclusions are based on standard charts e.g, ASTM E45, JK (Jernkontoret, Swedish, E 1122). A standard JK chart for comparative inclusions studies of various type is shown in figure 8[19]. For general purposes this method is adequate but has a number of deficiencies. The main problem is related to the reproducibility, time consuming, difficult for easy handling and availability of standard charts are limited in numbers. Because of this, some other methods were developed.Sulphur prints was also developed. The development for colour metallography was also thought of for their identification. To make the quantification easier and faster, an image analyser with automatic focusing system plays an important role. In the beginning, the image analyser was employed only for rating of inclusions. The inclusions present are differentiated between by their reflecting grey values. Attempt was also made to classify the inclusions by measuring their lengths. But length measurements alone however proved to be insufficiently characteristic criterion. In modern image analyser, to quantify inclusions, the most important parameters used are[20-22]:

- Inclusion type,
- The volume fraction,

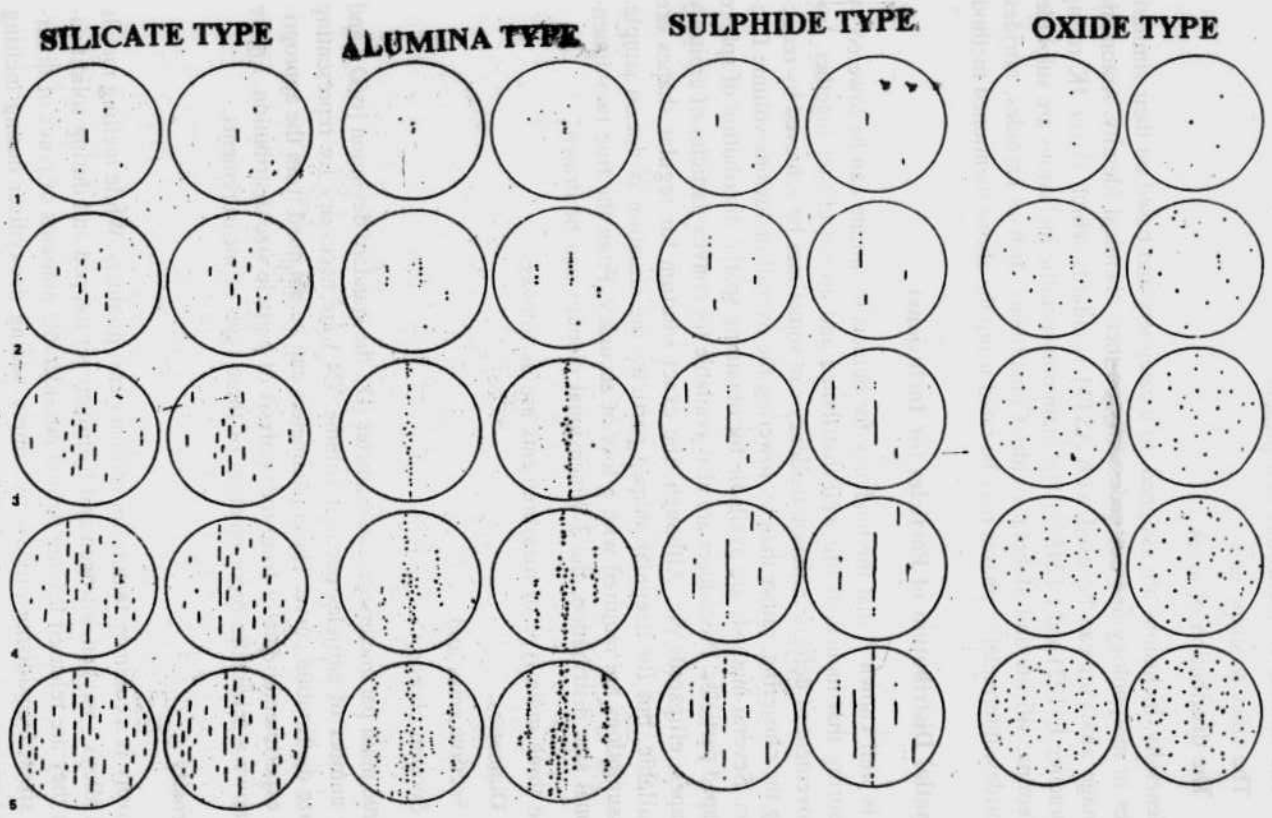

Fig. 8 : Jernkontorets comparative inclusions chart

- The number of inclusions that present
- The sizes & shape and
- The distribution of sizes.

Generally, inclusions are assigned to a categoery and based on their similarities in morphology and not necessarily on their chemical identity. Automatic image analyser can be used with ASTM standards which cover JK rating (practice E1122) and E1245[23]. The main non-metallic inclusions are sulphide, alumina, silicate and globular oxide. Constituents such as carbides, nitrides, borides, intermetallic phases may be rated using the above mentioned method.

Spatial Distribution of Particles (or Inclusions)

It is well known[24] that the tendency for defects to initiate can be lowered by insuring that inclusions are well distributed and do not cluster together. Improvement of defects initiation tendency can sometimes be achieved by reducing the clustering, rather than by lowering the overall inclusions volume fraction. Several methods are available for obtaining spatial distribution of sphere shaped particles. Procedures are also available for convex particles of arbitrary shapes, ellipsoids etc. Although, the exact solution for regular shapes are available, but for irregular shaped particles the situation is not so simple. Assumptions are required with a loss of accuracy. From the true two-dimensional size distribution, the 3-dimensional pictures can be drawn.

The three main type of measurements are as follows :

- Diameters,
- Section areas and
- Section chords.

Three main parameters : mean diameter (D), the standard deviation [σ(D)] and the number of particles per unit volume (N_V) are necessary for representing a size distribution curve. These parameters can be obtained from the appropriate experimental data or from the analysis of particle size distribution. Figure 9 shows a schematic representation of three type of measurements.

Porosity

Porosity is a common feature for ordinary cast products. While melting metals and alloys - oxygen, nitrogen and hydrogen get trapped and during solidification they are released leaving behind considerable amounts of pores of different sizes. Deoxidising process by simply adding deoxydiser during melting these gases can be removed. However, in practice for many cases some remain

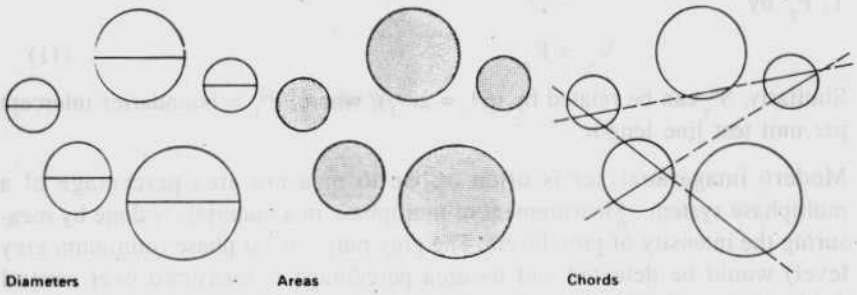

Fig. 9 : Schematic presentation of three main types of measurements made on planar section

trapped in the ingot. So, quantification of such porosity in terms of size distribution are essential for assessing the health of materials. A modern image analyser can be employed for exactly quantify the pores present in the materials. Care must be taken to differentiate inclusions and pores.

Multiple Phase Area percentage

In view of enormous development of processing technology the application of materials are more and more specific, so it has become important to quantify microstructure consisting of several phases. The mean free path or mean phase intercept of individually dispersed phase or particles in a matrix is given by[25]:

$$\lambda = \frac{4V_v}{S_v} \tag{9}$$

Where, V_v = the volume fraction of the obtained phase and S_v = the interface area. The above equation can also be extended for multiphase phases.

An additional parameter, similar to the mean phase intercept, is the mean random spacing and is defined as[25] :

$$\sigma = \frac{1}{N_L} \tag{10}$$

Where, N_L = particle (or clusters) intercept counts. σ may be interpreted as a mean centre to centre distance between dispersed particles or clusters of a particular phase.

The fundamental parameters used for mean free path relations are V^i_v and S^{ij}_v and, the volume fraction of phase "i" and the surface area per unit volume of

boundary shared by phase 'i' and 'j'. V^{i}_{v} can be found by point count procedure which can be related to the fraction of points on a grid occupying the phase 'i', P_{p}^{i} by

$$V^{i}_{v} = P^{i}_{p} \qquad (11)$$

Similarly, S^{ij}_{v} can be related by ($S^{ij}_{v} = 2P^{ij}_{L}$), where, P^{ij}_{L} = boundaries intercept per unit test line length.

Modern image analyser is often utilise to measure area percentage of a multiphase system. Measurement of multiphase in a materials is done by measuring the intensity of grey levels. The grey range of 1st phase (minimum grey level) would be detected and its area percentage is measured over several fields of view. By this way the detection of 2nd and 3rd phases can be done and areas are measured. This technique is requires a certain amount of dexterity and adjustment of thresholds for detection of individual phases. A video level histogram can show distinct peaks relating to the grey shades ranges of different phases. A typical video level histogram of four phases materials is shown in figure 10[17]. Again, after detection the various phases of the images can be coloured correspondingly to the colour assigned to the grey ranges.

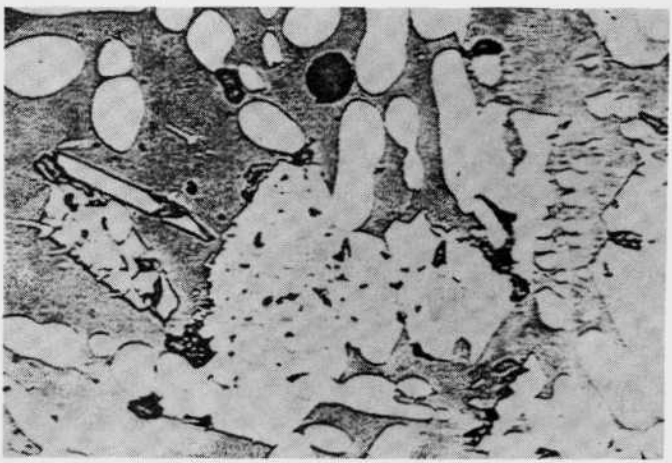

Fig. 10(a) : Typical AZS refractory material showing four distinct phases : Porosity, Silica, Alumina and Zirconia 400X.

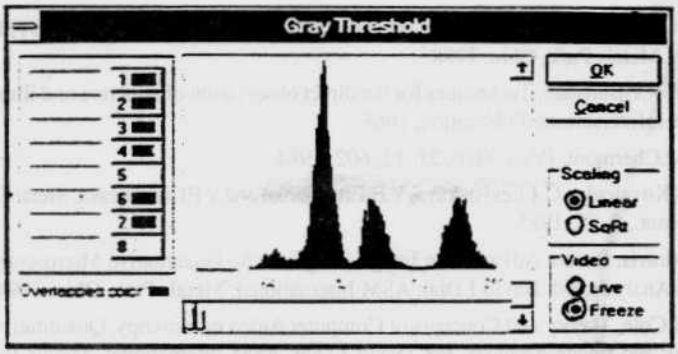

Fig. 10(b) : Typical video level histogram of four-phase AZS material seen in Fig. 10(a). Each peak corresponds to a separate phase

CONCLUSIONS

Quantitative data, extracted from polished and etched samples (if required), can be used for various purposes like quality control, process control, failure analysis or research purposes. A systematic and positive approach should be taken for precise measurement of various parameters. From sample preparation to data analysis - all steps must be properly and carefully performed to get best results. Sampling or identification of test samples from a large piece of materials or from a big component is most important. Specimen must be prepared properly, both during polishing and etching (if required). The best possible image should be fed into image analyser for proper quantification of various parameters of an image. Manual method can also be employed for image analysis but it is less sensitive and time consuming. Accuracy can also be improved simply by increasing the number of test measurements of fields or features. When all the steps will be fulfilled the final results will be precise and reproducible.

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