

Extended Field Microscopy and Image Analysis of Porosity in PM Steels

Measurement of porosity in PM steels by conventional image analysis requires a large number of individual images of the microstructure to be analysed. These images should be representative of the true porosity distribution within the material and good metallographic preparation is essential. Poor preparation can lead to phase tear out or rounding of pores, each of these will lead to errors of measurement. Image analysis of features of porosity such as area, perimeter and shape factor are dependent upon good image resolution and calibration and techniques for the measurement process are well documented.

However, as part of a quality system, a large number of images are required both for measurement and archiving, which takes a considerable amount of resources. Additionally, post processing of data may require the original images to be re-assessed and in some cases additional images to be acquired and measured, which might not be practicable.

Extended field microscopy (EFM) has been developed to solve these problems. Instead of multiple images of the microstructure being obtained, a single image at the same resolution is obtained. A measuring microscope and CCD camera captures a continual field of view which software then compiles into a single image in real time. Subsequent image analysis can then be carried out in the same manner with the added feature that the co-ordinates of the specimen where measurements are made are incorporated into the results.

E G Bennett and A Gant

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Materials

Four PM steel samples were supplied by Hoeganaes for the determination of porosity by image analysis. These are identified by the supplier's codes, 508-2000, 614-2001, 645-2001 and 610-2001 and are referred to in this report as samples 1,2,3 and 4 respectively.

Preparation Route

PM steels are difficult to prepare due to pore rounding and entrapment of polishing debris. However, good preparation is the key to representative images and subsequent microstructural characterisation by image analysis. The following preparation technique was employed: -

- Embedding: Hot pressed in Struers Isofast mounting medium in Struers ProntoPress 20.
- GRINDING: 40 micron Struers Diadisc (metal bonded) for 3 minutes, 10µm Diadisc (resin bonded) for four minutes. Both were used wet at maximum speed of rotation.
- POLISHING: (a) 6 micron diamond on Buehler nylon cloth for 10 minutes at 250 rpm. (b) 1 micron diamond on Buehler nylon cloth for 10 minutes at 250 rpm. (c) Chemical polish using Buehler Mastermet 2 on Struers OP-CHEM cloth for 2 minutes at 150 rpm.

IsoFast was used as the mounting medium to give maximum edge retention. Grinding on Diadisc as it is a fixed diamond system; thereby minimising entrapment of diamond particles in the sample pores. SiC papers were not used as it had earlier been found that it was time consuming due to the fact that extremely fine papers (2400-4000 grit) had to be used prior to

polishing, as the scratches produced by earlier stages using coarser grits were unacceptably deep. A metal bonded disc was used in the first stage as it is far more efficient at material removal than resin bonded discs. Samples were thoroughly cleaned in alcohol in an ultrasonic bath prior to polishing and between polishing stages to minimise carry over of debris to the next stage. Nylon cloths were used for both efficient material removal and minimising pore rounding and relief effects. The final (chemical) polish was found to be necessary and was used in preference to a fine diamond spray (e.g. 0.25µm) as it removes residual scratches in 1-2 minutes whilst using the OP-S cloth does not produce relief effects. During this last stage the cloth was also kept wet with de-ionised water to prevent snagging and/or pullout.

Image Acquisition and Mapping of Porosity

Images were obtained using a Nikon Measuring microscope fitted with displacement transducers with a resolution of 1 micrometer. The specimen stage has a rotation plate centered on an x-y stage with a maximum movement of 200mm. A rotating plate inserted into the x-y stage allows easy alignment of the specimen such that the co-ordinates of the specimen are easily obtained and easily repeated providing confidence as to where images and subsequent measurements are made. A microscope objective with a numerical aperture of 0.46 was used to provide a nominal magnification of x200 of the surface of the specimen. The digital images for measurement of porosity distribution were obtained from a Magnafire CCD colour camera at a resolution of 1280 by

1024 pixels, each image representing an area of 0.145mm^2 . An image of the microstructure from one of the samples is shown in Figure 1.

Image mapping was obtained using the co-ordinates of the samples obtained from the measuring microscope. The samples were aligned such that one corner had the co-ordinates 0,0,0 for the x, y and z axes respectively. For each of the 4 samples the area investigated was nominally 6.0 mm by 12.7 mm. Images were obtained from a 1mm array across the samples, resulting in a 12 by 5 grid. Images obtained are archived in accordance with the quality system operating within the Materials Centre, National Physical Laboratory.

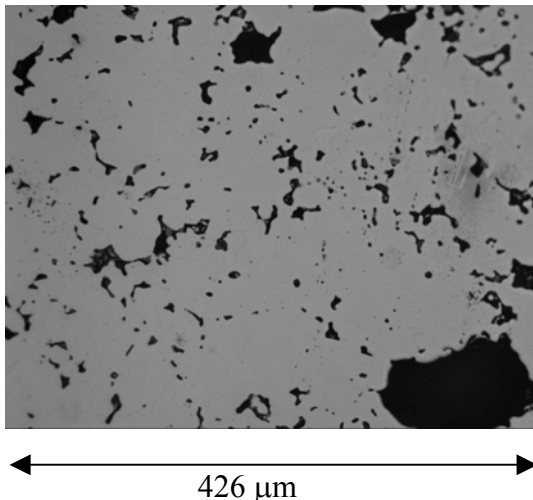


Figure 1 *Image of microstructure obtained from sample 2. The image shows a large area of porosity which is not totally within the field of view and which can cause problems with subsequent measurement by image analysis.*

The choice of magnification is a trade off between the smallest region that needs to be measured (resolution of system) and largest feature within the microstructure. Most microscopes have

limited sets of magnification, which limit the size of feature that can be measured.

Image calibration

Images were calibrated using certified stage graticule and checked using a certified image analysis graticule traceable to National Standards Repeated measurements from the graticule and microscope are used to determine errors of measurement to provide confidence in the results obtained from subsequent image analysis.

Image analysis

The first stage of the image analysis process, having obtained digital images of the microstructure, is to obtain a binary image of the regions of interest. Grey level discrimination is used to select the pixels in the image corresponding to the regions of porosity. In a few cases, manual image editing was necessary to remove unwanted features from the binary image. An example is that of a stain on the surface caused by polishing media being trapped in a pore and subsequently leaching onto the surface of the material during the cleaning stage. The second stage is to calibrate the image analysis system using artifacts of known size such as those from an image analysis graticule.

The image analysis system can now identify and measure the regions of porosity. However, certain conditions have to be applied to the image measurement process. The first is the deletion of regions which are below the resolution of the system [1]. The second is the introduction of a guard frame as shown in Figure 2 to prevent systematic errors [2]. Figure 2 shows that the guard frame reduces the area, which is

analysed. Additionally, the frame must be larger than the largest region of porosity to be measured, or a lower microscope magnification used. However, this has the effect of removing more small regions of porosity, as they would then be unresolved.

As a result, more fields of view of the microstructure need to be measured to obtain statistically significant results. However, this assumes that the material is homogeneous, and this is not proven until the measurements have been made and the data analysed.

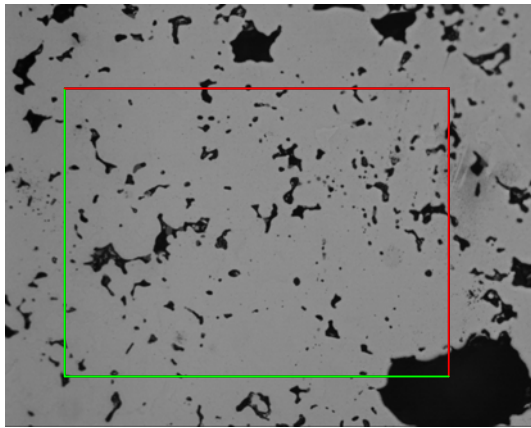


Figure 2 *Guard frame introduced into image. Regions of porosity completely within the frames are measured. Those regions intersecting the green lines only are also measured. Regions that intersect red or red and green lines are excluded.*

After taking these conditions into consideration, the area fraction of the 4 samples were measured using image analysis of the images previously obtained. Figure 3 shows the results for one of these samples. As can be seen from the 3D plot, there is a large variation in area fraction of porosity from position to position across the

sample. Similar variations were found for the other samples and the overall results for the four samples are shown in Table 1.

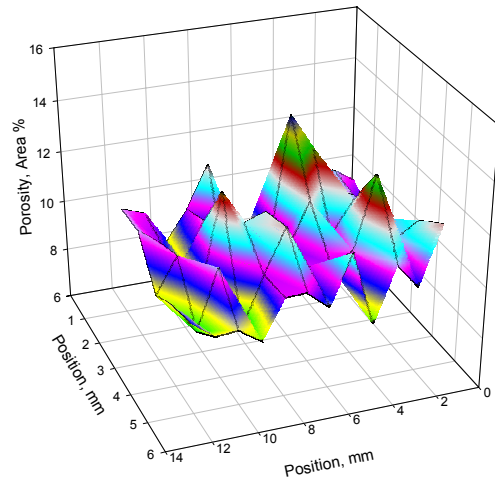


Figure 3, *3D plot of porosity area% as measured at discrete positions on the polished surface of sample 1.*

As can be seen from the plot in figure 3, there does not appear to be any pattern to the spatial distribution of porosity.

Sample	Area % Porosity	Standard Deviation	Maximum	Minimum
1	9.9	1.3	13.9	7.1
2	8.9	1.3	12.6	6.9
3	15.3	1.7	18.9	11.1.
4	10.1	1.7	14.5	7.0

Table 1, *results from all of the porosity measurements on each of the four samples.*

As can be seen from the maximum and minimum values in table 1, the result of a large pore can cause a large change in the apparent porosity fraction. For

example, the pore which is visible in the bottom right of figure 2 would be discounted from the measurement process. However, if it were included the result would be a far higher area fraction measurement.

It is also evident from the 3D plot shown in figure 3 that insufficient measurements were made to determine if there was a trend in porosity throughout the sample. In order to determine a trend, far more measurements would be necessary to remove the statistical bias caused by large regions of porosity. Alternatively, lower magnification images with a larger field of view could be used. However, this would place many regions of porosity below the resolution of the system and introduce a bias in the measurements.

Extended Field Microscopy

Extended field microscopy has been developed to address many of the problems identified above. The technique has only recently become feasible with advances made in computer operating systems, speed, RAM memory and image acquisition methods.

Extended field microscopy is a technique whereby digital images larger than the normal field of view are obtained from an optical microscope. Montage software supplied by Synoptics “stitches” together in real time the field of view of the image of the microstructure as the sample is moved on the microscope stage producing a montage of adjacent fields of view.

A typical image of a scan across a section of sample 1 is shown in figure 4.

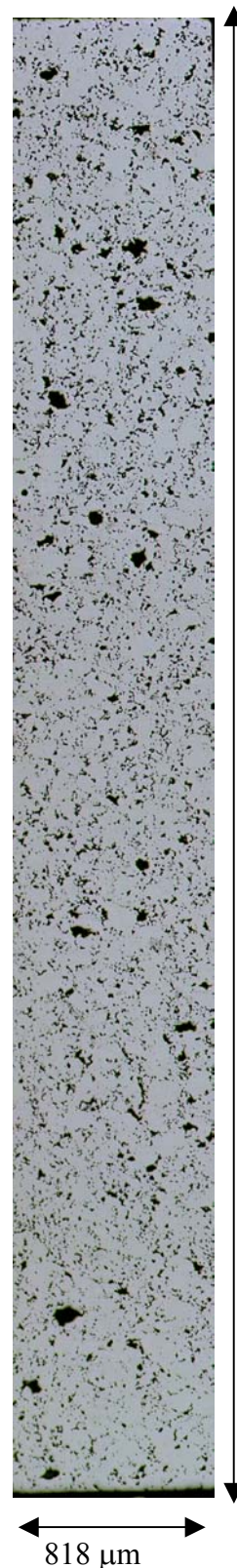


Figure 4 *Extended field image across the width of sample 1, taken at a position of 6mm along the length of the sample. The image is of an area of 6,000 by 818 μm , or 4.91 mm². The image is stored as a single TIFF colour image file of 4,812 by 655 pixels and is 9.01Mb in size*

6,000 μm

818 μm

The porosity in Figure 4 was measured using a KS400 image analysis system. The porosity area fraction was measured as 8.0%, no standard deviation is calculated from a single measurement. As well as measuring the area fraction, a total of 4,672 individual pores were measured as well as their centers of gravity within the image. Since the image starts at the specimen edge, the measured centre of gravity in the Y direction is also the position of the pore. Additionally, the position of the scan line from the measuring microscope is added to the values of the centre of gravity in the X direction. Thus, the exact position of each individual pore within the material can be measured as well as its size or other microstructural feature such as shape factor or perimeter.

The data obtained from these measurements can then be analysed separately from the measurement process. For example, the variation in porosity fraction across the sample can be calculated in say, steps of $100\mu\text{m}$ as shown in figure 5

As can be seen from the plot, there is a large variation in area% of measured porosity across, but no obvious trend. A plot of the running average does not settle down to a constant value, but does suggest a trend. The reason for the data having such a large scatter is due to large areas of porosity being included within one measuring frame, but not adjacent frames. A plot of the smoothed data, i.e., averaging across adjacent fields also shows a trend in the area% of porosity from one side of the sample to the other.

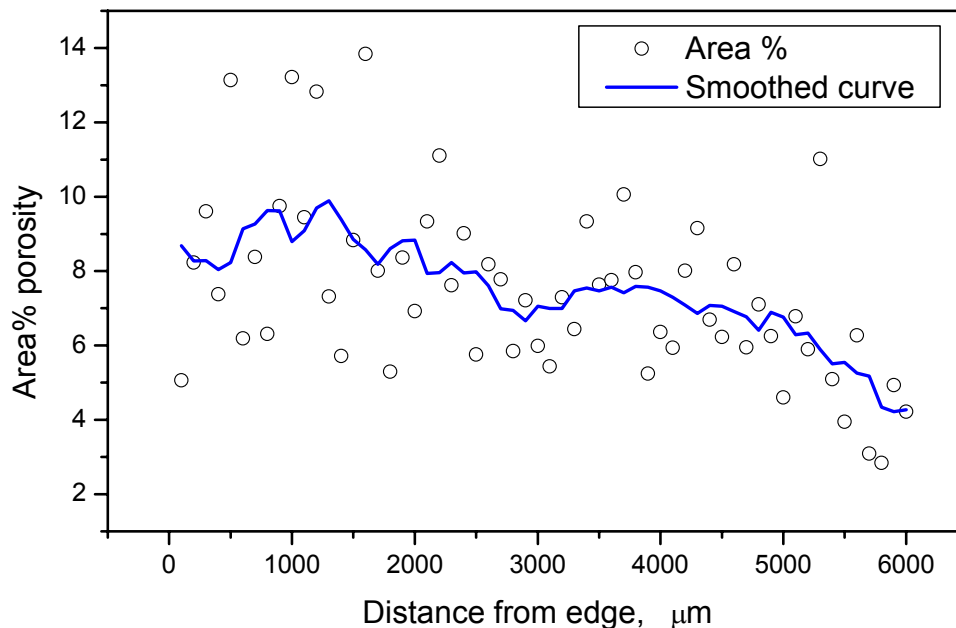


Figure 5, *variation in area % porosity with distance from edge, $100\mu\text{m}$ steps*

However, if the data is grouped over a greater distance, the effect of large pores is not as great on the area% of porosity. The effect is shown in figure 6 where the data has now been averaged over distances of 500 μm , and a trend in the level of porosity is now clearly evident.

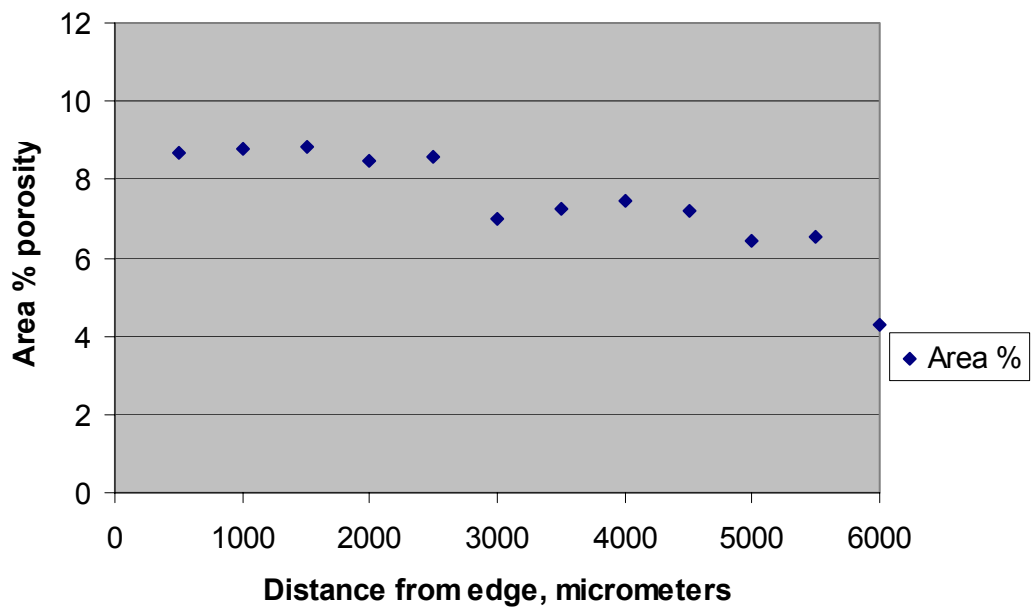


Figure 6, *variation in area % porosity with distance from edge, 500 μm steps*

SUMMARY

Extended field microscopy (EFM) overcomes many of the limitations encountered using image analysis when measurements have to be made over relatively large regions of microstructure. The measuring microscope maps out and references the regions within the microstructure to a resolution of 1 micrometre. The imaging software captures the whole of the surface of the specimen whose microstructure is to be characterised, and stores a single image of the microstructure as part of a quality system. A case study of a porous PM material indicates that EFM resolved a gradient in porosity across a sample that was not identified using conventional frame by frame analysis.

REFERENCES

[1] BS 3406: Part 4: 1993. Methods for determination of particle size distribution, Part4. Guide to microscope and image analysis methods.

[2] Practical Guide to Image Analysis. Analysis and Interpretation. ASM International. ISBN 0-87170-688-1, December 2000, 171-183.

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FOR FURTHER INFORMATION CONTACT

E G Bennett
Materials Centre
National Physical Laboratory
Queens Road
Teddington
Middlesex TW11 0LW

Tel: +44(0)20 8943 6633
Fax: +44(0)20 8943 2989
e-mail: eric.Bennett@npl.co.uk

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