Measurement of Porosity by EPMA-EDS Image Processing*

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ABSTRACT

Porosity is one important characteristic feature and structural index of sprayed coatings. A method of measurement of porosity, EPMA-EDS image processing, is developed in the paper. The characteristics of pores can be determined by processing of the image obtained from an electron microscope *via* VISTA. Not only the porosity can be presented, but also the statistical result of pore size distribution. Finally, it can be drawn from this paper that EPMA-EDS is a quite effective method to completely characterize the pores in plasma sprayed coatings.

1. INTRODUCTION

The boundary structure between splats or between the substrate and splats greatly affects the physical characteristics of thermal sprayed coatings, especially the porosity[1]. But, measuring porosity is inherently challenging. Traditional porosity measurement methods are water-absorption, optical microscopy (OM) and mercury intrusion porosimetry. However, water-absorption and OM are time consuming methods and highly dependent on the operator, moreover, for the latter, pores smaller than 5μm will not be detected[2]. And mercury intrusion porosimetry is really a poisonous method and usually used for large porosity measuring (e.g. more than 10%). Clearly, a new characterization tool for porosity is needed.

Electron probe microanalyzer (EPMA) is mainly for structure analyzing. Combined with energy dispersive spectrometer (EDS), EPMA can be used not only to observe the morphology, size and content of a certain phase, but also to quantitatively analyze geometric parameters such as diameter, perimeter and area *et al.* of the particles. This paper describes such an image processing method that provides a good means to characterize coating porosity. Here, an advanced image acquisition and processing program, VISTA, is employed. Pores are treated as one of phases in the study. The measured porosity is the whole porosity rather than open porosity by water-absorption. While the data obtained is two dimensional, it is virtually the volume percent porosity according to stereology principles.

2. PRINCIPLES

Image. Image formed in EPMA are the reflection of different intensities signals of electron-specimen interactions such as the secondary electron or backscattered electron. Before image processing, these analogical images[3] must be transformed into digital images. There are only two

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types of digital images that can be acquired in EPMA-EDS. One is video images acquired from an electron microscope detector such as the secondary electron interaction or backscattered electron interaction using the microscan digital beam controller. The other is X-ray maps acquired from standard EDS point dwell.

Pixel. Images displayed on the monitor actually consist of hundreds of tiny dots called pixels (picture elements). A pixel is the smallest portion of an image that can be addressed (changed in some way). Each image pixel is assigned an intensity or gray-level value (0~255) based on the data from which the image is constructed. For video image, the intensity value for a given pixel is based on the intensity of the secondary electron or backscattered electron signal for that portion of the sample. For X-ray maps, the intensities are based on the X-ray count for that portion of the sample, the higher the number of counts, the greater the assigned intensity.

Gray-level segmentation. The digital images acquired consist of pixels contained within a particular gray-level intensity range. Each phase of the image has a gray-level range. If the pixels that stand for the analyzed phase are off or on and all the others are on or off, a special image so called binary image is created. Therefore, when some parameters such as magnification, number of measurement for pore diameter, minimum and maximum size of the phase are set up, the size (length, width, diameter, etc.) of the interested phase can be determined.

Image processing procedure. The description below is just for pore analysis. First of all, a backscattered electron image must be acquired by EPMA. The corresponding video image is then created by VISTA in accordance with the intensities of electron-specimen interactions in the electron microscope. The digital image therefore consists of hundreds of pixels each which has been assigned an intensity or gray-level value (0~255). Gray-level 0 stands for the lowest intensity, gray-level 255 stands for the highest intensity. The digital image is further processed to create a binary image in which the pixels for pores are all off by selecting a certain gray-level range. Finally, when appropriate parameters are set up, some features of pores such as porosity, pore area distribution, etc. will be presented. It should be pointed out that the image resolution should be selected higher than the display resolution when processing.

3. EXPERIMENTAL

The sample used is a Cr_2O_3 coating prepared by air plasma spraying. The coating is then polished and sputtered with a layer of Au conduct film of thickness 20~30nm. The employed image processing analyzer is EPMA-8705QH electron probe mecroanalyzer (SHIMADZU) and TN-5502N energy dispersive spectrometer (NORAN). As a comparison test, the open porosity measurement of the coating by water-absorption is also carried out.

4. RESULTS AND DISCUSSION

Fig.1 shows the morphology of the cross-section of Cr_2O_3 coating. Fig.2 is Fig.1's binary image. From Fig.2, the analyzer gives the porosity 4.59%. The result of a similar analysis but with another magnification, 2000×, is shown in Fig.3 and Fig.4, and the measured porosity is 8.48%. If we assume that human eye's resolution is about 0.1mm, pores whose diameter is less than

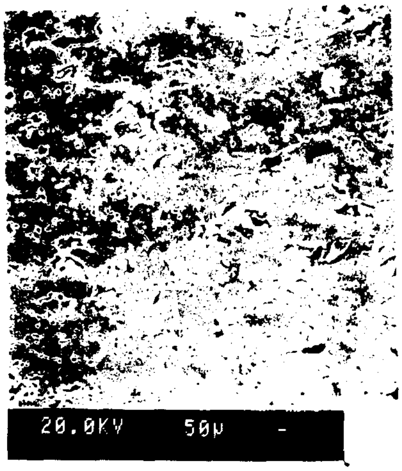


Fig.1 The image from backscattered electron of cross-section of Cr_2O_3 coating, 200×

Fig.2 The binary image of Fig.1

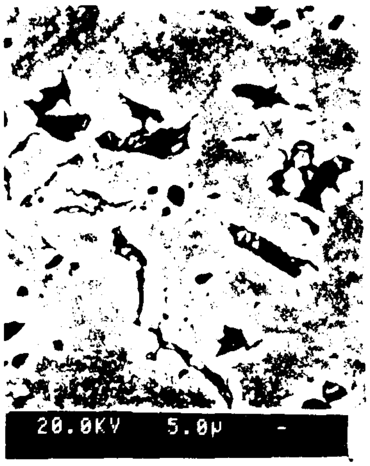
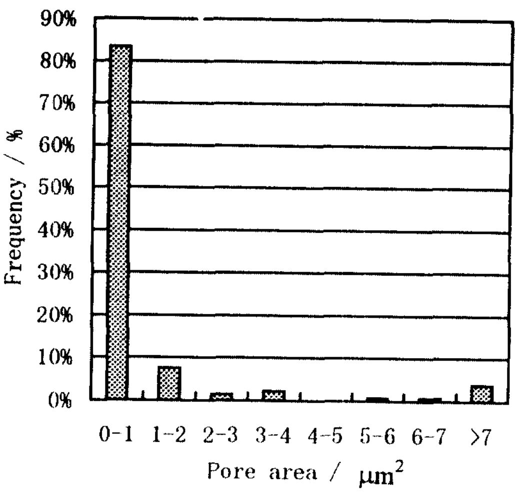


Fig.3 The image from backscattered electron of cross-section of Cr_2O_3 coating, 2000×

Fig.4 The binary image of Fig.3

$\frac{0.1mm}{200}$=0.0005mm=0.5μm will not be detected with 200 magnification selection. But with 2000 magnification selection, pores up to $\frac{0.1mm}{2000}$=0.00005mm=0.05μm will be included. In most plasma sprayed coatings, esp. those deposited by a high velocity gun, pores are mostly distributed less than 1μm[4]. Hence, the result obtained with 2000 magnification selection is higher and more reasonable than that with 200 magnification selection. In fact, the open porosity of the Cr_2O_3 coating measured by water-absorption is 6.8%. This further confirms the discussion above.

It is usually not enough to characterize pores in the coating just with a porosity value. Pores area distribution is necessary in some applications. Fortunately, this can be easily obtained in EPMA-EDS. The histogram in Fig.5 gives the frequency distribution of pores, and that in Fig.6 gives the area percentage of pores. From Fig.5 and Fig.6, we can also conclude that while the number of the small pores (less than 1μm^2) are relatively very large, the several big pores occupies a unnegligible area percentage.



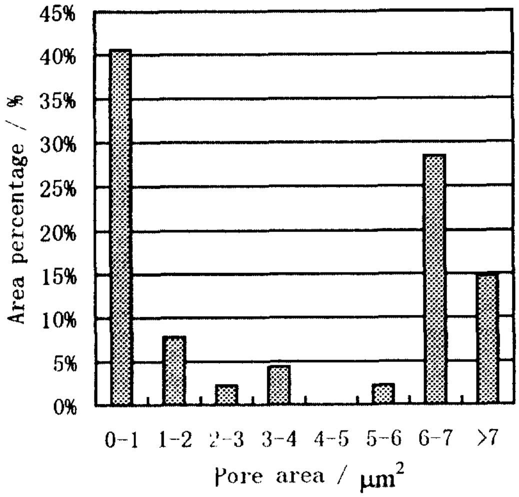


Fig.5.The histogram of pore frequency distribution. Fig.6.The histogram of pore area distribution

EPMA-EDS pores analysis method has a number of advantages as well as some limitations. Firstly, up to tens of thousands magnification can be used, which provides a possibility to analysis the very tiny pores if necessary. This is impossible with OM. Secondly, it can completely characterize the pores, while with OM or water-absorption only porosity can be obtained. Thirdly, unlike OM and water-absorption, it is a time saving method. Thus, EPMA-EDS is really an attractive technique for pores characterizing in plasma sprayed coatings owing to its special pore characteristics and coating thickness less than 1mm generally. One limitation of EPMA- EDS method in pores analysis is the relatively strict request for the uniform pore distribution, esp. when with high magnification selection. In addition, as mentioned in principles section, an appropriate gray-level range selection is very important when gray-level segmentation is carried out.

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