CHARACTERIZATION BY SYNCHROTRON X-RAY MICROTOMOGRAPHY OF INTERNAL MICROSTRUCTURAL FEATURES AND THEIR DETRIMENTAL EFFECTS WITH RESPECT TO THE FATIGUE PROPERTIES IN AN ALUMINIUM CAST ALLOY

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ABSTRACT

The X-ray microtomography technique was validated for the investigation of porosity in Al cast alloys. Two AlSiMg0,3 alloys with different porosity contents were studied and characterized by two parameters, equivalent size and sphericity. It was shown that the most porous alloy contains pores which are bigger and rounder than the other one. These features are discussed with regard to fatigue properties, which are worse for the most porous alloy.

Keywords: microtomography, fatigue, crack initiation

INTRODUCTION

In aluminium cast alloys, fatigue crack initiation occurs very often on pores which result from the material processing and which are located at or just beneath the surface of the sample. However, very little is known about the mechanisms which lead to the initiation of a crack from one of those surface or sub-surface pores. It has been suggested, for instance that the size of the pore or the square root of its projection on a plane perpendicular to the load axis could be used as a critical parameter to assess the probability of a pore to initiate a crack [1]. Those two parameters can be obtained by post mortem observation of fracture surfaces. However it is impossible to compare with pores which have not initiated cracks.

Any attempt to understand the crack initiation mechanisms on pores requires a detailed characterization of the pore size and shape distribution. This is very difficult to achieve by classical 2D metallographic observations which are very tedious and not free from artefacts. Therefore, such a characterization has been undertaken by high resolution X-Ray tomography. This non destructive technique provides three-dimensional images of the pores in the material.

Preliminary results obtained on two alloys showing different porosity contents are given in this paper. Examples of three-dimensional parameters calculated from three-dimensional images of pores are given and discussed as a function of fatigue properties.

EXPERIMENTAL METHODS

Materials - Samples

Two AlSiMg0,3 alloys (A and B) were chill mould cast. Each contains a different degree of porosity. This was achieved by introducing two gas mixings with different hydrogen/argon ratios in the melt through a propeller. The solubility of hydrogen being an increasing function of temperature, a part of the gas content is rejected and forms artificial pores during the beginning of casting. The hydrogen to argon ratios were respectively 2% and 5% for the alloys A and B.

The material (the composition of which is given in Table 1) was solution treated 10h at 540°C, quenched in cold water, and aged 6h at 160°C.

Si	Fe	Mg	Ti	Sb	Cr	others
6.6-7.0	0.09-0.14	0.29-0.34	0.11-0.14	0.12-0.14	<0.01	<0.01

Table 1 Chemical composition (percentage by weight) of the studied alloys

Giving the attenuation coefficient of aluminium and silicon, the optimal thickness of the samples for microtomography was calculated to be about 3 mm. Two cylindrical samples (from alloy A and B), 2 cm long and with a diameter of 2.85 mm then were machined for X-ray microtomography experiments.

The X-ray microtomography imaging technique

The X-ray microtomography experiments were performed on the ID 19 beamline at the European Synchrotron Radiation Facility (ESRF), in Grenoble, France. The chosen energy was 23 keV. The quasi coherent, parallel monochromated beam goes through the investigated sample. At about 80 cm behind the sample, a 1024x1024 CCD camera collects the intensity of the attenuated beam, forming one projected image of the sample. For a complete tomography scan, 600 projections were recorded, corresponding to 600 different angles of rotation of the sample from 0° to 180°. From this set of projections, three-dimensional images (blocks) were reconstructed. In addition to attenuation contrast, which is sensitive to ρZ (ρ is the density and Z the average atomic number), the special experimental conditions used at the ESRF give a so called phase contrast [2], wich underlines all interfaces in the material. The experimental resolution is 6.65 µm.

Image analysis

A three dimensional image analysis software was used. The images were thresholded, in order to extract the pores from the rest of the material. Then the obtained binary images were automatically analyzed, giving individual parameters of the pores (volume, surface, Ferret dimensions) and also the concentration of pores in the studied block.

RESULTS AND DISCUSSION

Validation of the imaging technique

Once the X-ray microtomographic scan performed and the block reconstructed, the sample of alloy B was cut perpendicular to its revolution axis, grinded with SiC paper and polished with a ¼ µm diamond paste. An optical micrograph of the cut surface was performed. The correspondent slice in the tomographic block was then imaged and compared with the optical micrograph in order to assess the ability of microtomography to image porosity.

It can be seen on Fig. 1 that the microtomographic image and the micrograph are qualitatively very similar. The geometry of the pores is easily imaged. The slightly bigger size of the pores on the micrograph comes from a defocusing effect at the edges of the porosity. The white interference fringe on the tomographic image results from the phase contrast [3].

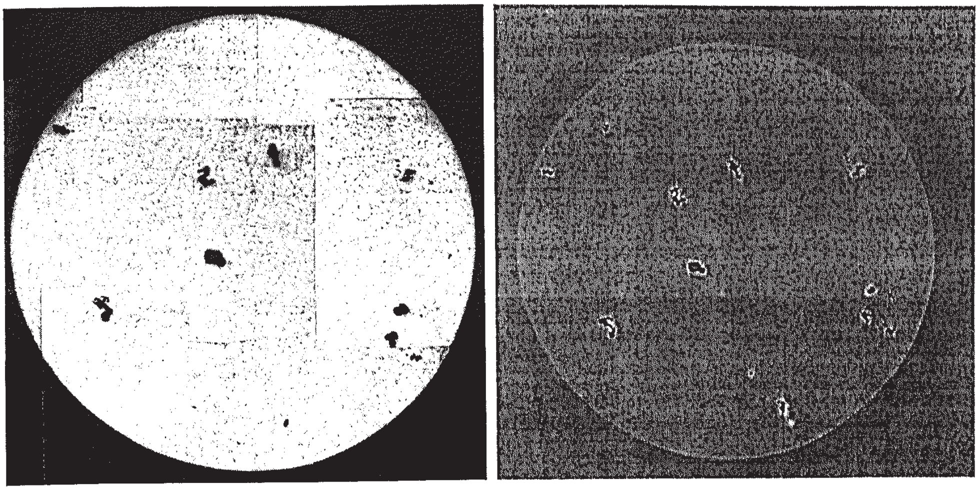

Figure 1 Optical micrograph of a sample of alloy B (left) (diameter of sample : 2.85mm) and the corresponding slice in the tomographic reconstructed image (right). The two images look similar.

Qualitative characterization of the porosity

The reconstructed images have 256 gray levels. The pores appear as dark features. For these reasons, it is possible to image the surface of the pores in three dimensions by taking an appropriate value of gray level and performing a gray level isosurface. This was done for alloys A and B (Fig.2).

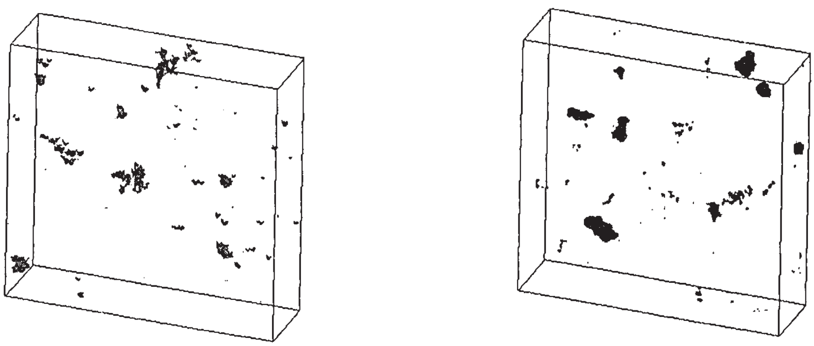

Figure 2 Three-dimensional visualization of the pores boundaries in alloy A (left) and in alloy B (right) (the blocks are respectively 1.5 mm large and high, and 0.425 mm thick)

The validity of the boundary gray level was tested by comparing visually on the same image the trace of the isosurface on a cut pore and the original boundary.

It can be qualitatively seen from Fig. 2 that alloys A and B contain pores families which are very dispersed in size. The biggest pores are bigger in alloy B than in alloy A. Furthermore, the big pores of alloy B are rounder than the ones of alloy A, which appear to have a tortuous surface.

Quantitative characterization of the porosity

The porosity concentrations in alloy A and B were found respectively to be 0.24 % and 0.46 %. The actual volume of each pore V is given by the 3D image analysis software. We then calculated the equivalent size s, which is defined as the diameter of a sphere with the same volume V :

$$s = 2\sqrt[3]{\frac{3V}{4\pi}} \qquad (1)$$

The distributions of s were calculated for two blocks having approximately the same dimensions as in Fig. 2. All the pores having fourteen voxels or less were eliminated from the total population, corresponding to an equivalent size lower than 20 μm.

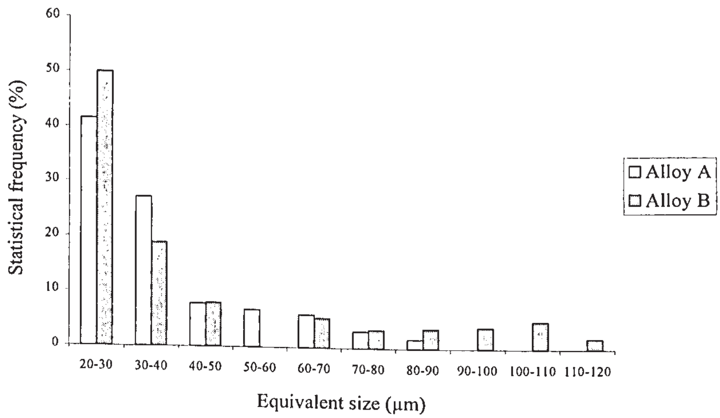

Figure 3 Statistical frequency of the pore size parameter s in alloys A and B

The pores cut by the boundaries of the three dimensional analyzed blocks were also eliminated. This introduces a bias which needs to be corrected : bigger pores are more likely to touch the boundary of the image and to be bypassed in the counting. A pore having in three perpendicular directions the maximal dimensions Δx, Δy and Δz in an image with dimensions x, y and z, will be counted more than once [4] :

$$count = \frac{xyz}{(x-\Delta x)(y-\Delta y)(z-\Delta z)} \qquad (2)$$

The results including the correction explained above are shown in Fig. 3 and show that the size distibutions of alloys A and B have the same shape. Both are approximately of exponential type : there are a lot of little pores and few big ones. But the size distribution of alloy B exhibits a longer tail indicating that the biggest pores of alloy B are bigger than in alloy A.

However, one can see on Fig. 2 that both alloys contain round and tortuous pores. Tortuous pores are believed to be due to solidification shrinkage while pores which have a somewhat round shape are due to degasing. In fact the relative roles played by degasing and shrinkage in the formation of

pores are still not elucidated and a pore should be formed by a combination of both phenomena. In order to study the eventual correlation between formation mechanisms and pore form, we need at least one parameter which characterizes the form of a given three dimensional pore.
A sphericity parameter λ taking into account the actual surface S and the actual volume V of a pore, was defined in a first approach to quantify the tortuousness of pores :

$$\lambda = \sqrt[6]{\frac{36\pi V^2}{S^3}} \qquad (3)$$

This dimensionless parameter is 1 if the pore is spherical and decreases when the surface goes away from an ideal spherical shape. For instance, the parameter λ has been calculated for the pores shown on figure 5. In this case, the values obtained for the round pore and for the larger tortuous one are 0.75 and 0.50, respectively.

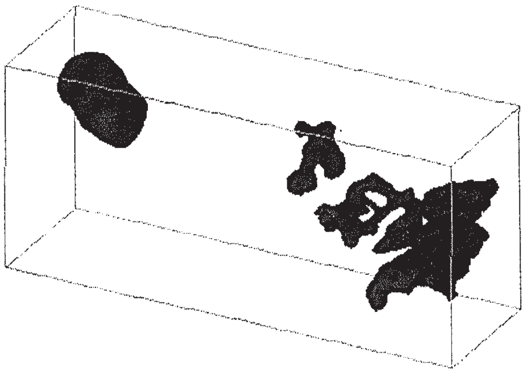

Figure 4 Reconstructed block of alloy B containing a round pore and two tortuous ones (the dimensions of the block are 186µm, 272 µm and 592 µm respectively)

The distributions of the λ parameter were calculated for both alloys for pores bigger than 40 µm. The results are shown on figure 5. It can be seen from this figure that the pores of alloy B are more spherical than the ones of alloy A.

Discussion

The noxiousness of a pore can be considered in terms of geometry, environmental microstructure and nature of the atmosphere trapped in the pore (vacuum, gas or air). The following discussion deals with the geometrical effects only as geometrical features are the only parameters assessed by our dataprocessing technique so far. In a general way, the local stress concentration coefficient K_t can be expressed as :

$$K_t = 1 + \alpha\sqrt{\frac{s}{\rho}} \qquad (4)$$

where s is the size of the pore perpendicular to the loading axis, α a geometry-dependent parameter and ρ the local radius of curvature.
Quantitative image analysis of the 3D blocks obtained by X-ray microtomography show that the pore size in alloy B is larger than in alloy A and that, besides, the shape of the pores in alloy B tend to be more spherical than in alloy A. This last result indicates that the pores of alloy A are likely to

exhibit on average, smaller values of ρ. Fatigue tests with a load ratio of 0.1 have shown that alloy A has better fatigue properties than alloy B over a wide range of stress levels. This seems to indicate that for the investigated alloys, fatigue life is mainly governed by the size of the pores rather than by their global shape. This can be explained considering high stress levels applied during the fatigue tests. In these experimental conditions, the initiation stage is hardly observed for fatigue cracks and the pore size simply adds to the crack size. Therefore statistically the initial stress intensity factors for the cracks in alloy B are larger than in alloy A, resulting in shorter fatigue lives. At low stresses however, crack initiation is an important part of the fatigue life and there might be a competition between rapid crack initiation/high propagation length (for a given critical crack length) in alloy A and slow initiation/low propagation length in alloy B.

At that stage, however, it must be emphasised that the parameter λ which aims at characterizing the tortuousness of internal pores describes incompletely their geometrical noxiousness. In fact, the deduced information is global and we need local information. For instance, it is possible that big pores in alloy B are noxious because of local regions with small values of ρ. More work is currently being carried out to obtain local values of the pore surface curvature from reconstructed images. This parameter should allow a better description of the pores and allow a better correlation with fatigue properties.

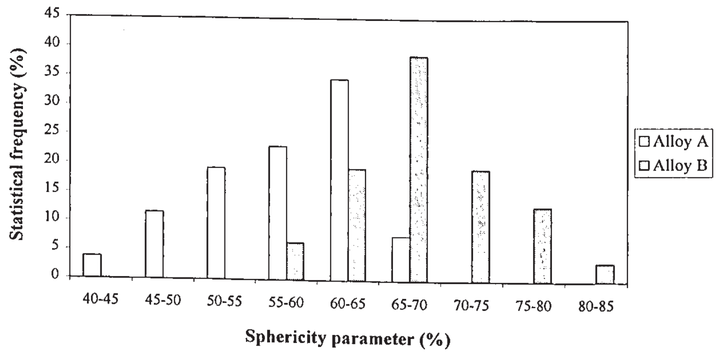

Figure 5 Distributions of the sphericity parameter λ for alloys A and B

CONCLUSION

This paper presents preliminary results on the use of X-ray microtomography for characterizing porosity in Al cast alloys. Pores were shown to be imaged easily by this new technique. Quantitative parameters characterizing size or shape of internal pores could be measured on 3D reconstructed images and correlated to fatigue properties. For the two investigated alloys fatigue properties appear to be dependent on the size rather than the shape of the pores. More work is required to define precisely the noxiousness of the individual pores.

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Acknowledgements

J.C. Ehrström and F. Cossé from Pechiney are fully acknowledged for having provided the material.