Volume 66 • Issue 2 • October 2014

Journal of Achievements in Materials and Manufacturing Engineering

International Scientific Journal published monthly by the World Academy of Materials and Manufacturing Engineering

Investigating porosity of sintering porous copper structure with 3D micro-focus X-ray computed tomography (μCT)

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Received 23.07.2014; published in revised form 01.10.2014

ABSTRACT

Purpose: The paper presents studies on the structure intended to form a heat exchange element made of sintered porous Cu.

Design/methodology/approach: Analyses of Cu foam exhibiting a cellular structure have been carried out using X-ray tomography.

Findings: Samples of copper foam have been imaged at high resolution using a large-field, 3D micro-focus X-ray computed tomography (μCT) system, three-dimensional image blocks derived from the scans were examined as cross-sections along orthogonal planes and as perspective images, maneuvered to be viewed from any angle.

Research limitations/implications: This work presents methods for obtaining pore size distributions for both the micro-pores and their interconnects. It‘s a matter of great significance for recognition of real structure of sintered porous elements and it's utility mainly as heat transfer.

Originality/value: The proposed X-ray method appears to be an excellent tool for determining the 3D structure of the sintered Cu foam. It should be used to improve changes detection in the structure of the foam itself, incurred to the effect of changes process appeared during heat transfer operation.

Keywords: X-ray tomography; Porous materials; Cellular materials; Image analysis; 3D

Reference to this paper should be given in the following way:

W. Depczyński, Investigating porosity of sintering porous copper structure with 3D micro-focus X-ray computed tomography (μCT), Journal of Achievements in Materials and Manufacturing Engineering 66/2 (2014) 67-72.

ANALYSIS AND MODELLING

1. Introduction

For proper understanding of the structure and geometry of the metallic foam there's a need to carry the study with different scales and various research methods. To determine the potential applications the microstructure of the foam solid should be analysed. For this purpose there are used conventional methods standard optical microscopy observations and a SEM. To know the characteristics of the porous structure of these materials is most difficult. This paper describes the study using micro-tomography X-ray μCT. Many authors believe that the tomographic technique will be widely used in the near future for this purpose. The paper also will show obtained 3D images, which may be used to achieve important morphological parameters describing the foams.

Solid foam (SF) are the foams with open or closed cells and their solid element is made of metal, ceramic, or the polymer. They create a wide developing a new category of materials available to engineers. Polymer foams have been used for many years in the packaging industry on industrial scale, for thermal isolation in buildings or applied to increase the comfort of use any kinds of seats.

The literature about this category of materials is relatively rich. The book of Solids by Gibson Ashby [1] for example represents extensive research to understanding the mechanical behaviour of wide SF range. Studies towards the development of metal foams represent a growing range of available publications in both materials science, technology, and theoretical and applied mechanics [1-6].

The authors of the paper [2] had analysed the macroscopic properties of such materials and pointed in two scales of the microstructure which should be detected and analysed:

- the microstructure of material fixed part, indifferent scales,
- the cellular microstructure also referred to recent investigations as a "architecture" in SF.

Researchers, who are developing manufacturing technologies of metallic foams, starting to use the micro X-ray tomography µCT methods as a tool to obtain unique data on the "architecture" of metallic foams and their influence on the properties of such. flow media [7-10].

When using porous materials with high surface developing in heat transfer issues extremely important are in formations about porosity or the surface development [11-14]. In the case of boiling buble pore size distribution determines the characteristics of boiling point – the start and end regarding the heat flow.

2. Experiment specimen and equipment

From the analysis it was evident that sintering of metal powders in a protective atmosphere was the best alternative. A powder mixture of copper and copper oxide was deposited on a copper substrate. The samples were sintered in an atmosphere of dissociated ammonia. The reduction of copper oxides resulted in the formation of diffusion-type pores. The sintering was carried out at a temperature of 950^{o}C for 45 minutes. During sintering, copper oxide reacted with hydrogen:

$Cu_2O + 2H = 2Cu + H_2O$.

Copper oxide was reduced to pure copper and hydrogen bonded with oxygen to form water vapor. As a result of this reaction, the mixture of copper powder and copper oxide powder was converted into a porous material. The porous sample was sintered and simultaneously diffusion bonded with the copper substrate. The technology is protected by Polish patent No. 199720 [15]. The experiment was performed using electrolytic copper powder with a particle size of 45-63 microns. The copper oxide powder was produced by heating Cu powder in air at 850^{o}C. The particle size of copper oxide powder ranged from 80 to 200 microns.

The samples were sintered in the form of cylinders with a diameter of about 20 mm and a height of about 8.To test µCT was used NIKONXTH225(ST) device with open tube source type -reflective-tungsten. The voltage source was 225 kV and 35 µA. There were taken 3142 projections with 4 seconds interval. The total test time was 4 h.

X-ray spot target 3 µm. Rotate 360 degree.

3. µCT investigation

The sample was on a rotary stage in between an X-ray source and detector. The micro-focus source generates the X-ray radiation and transmits the rays through the sample. A digital flat panel detector captured a 2D images of the X-rays patterns that passed through the specimen, showing different shades of grey depending on material and geometry. Thicker or denser material such as copper translate into darker areas than thin or light materials like air.

To generate a 3D CT volume, a series of sequential 2D X-ray images is taken as the object rotates 360 degrees. The image then go through a reconstruction software algorithm that generates a 3D volumetric map of the object. In addition to the outer surface, the volume contains the internal surface as well as the complete internal structure that is gained through the fourth dimension density.

Reconstruction of an industrial component based on the projected image slices leads to a voxel model (a voxel is the 3D analogue of a pixel), where the grey value of the voxels is a measure of the linear attenuation coefficient of the material. The voxel data is post-processed using algorithms to detect the edges and features of the work piece, allowing dimensional measurement and quality control.

Scheme of investigation process in µCT is shown in Figure 1.

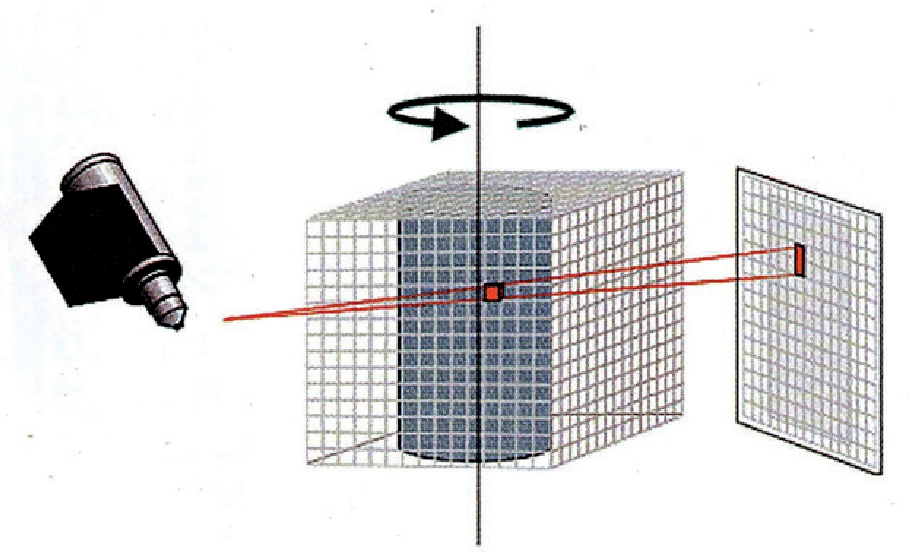

Fig. 1. Scheme of investigation process in μCT

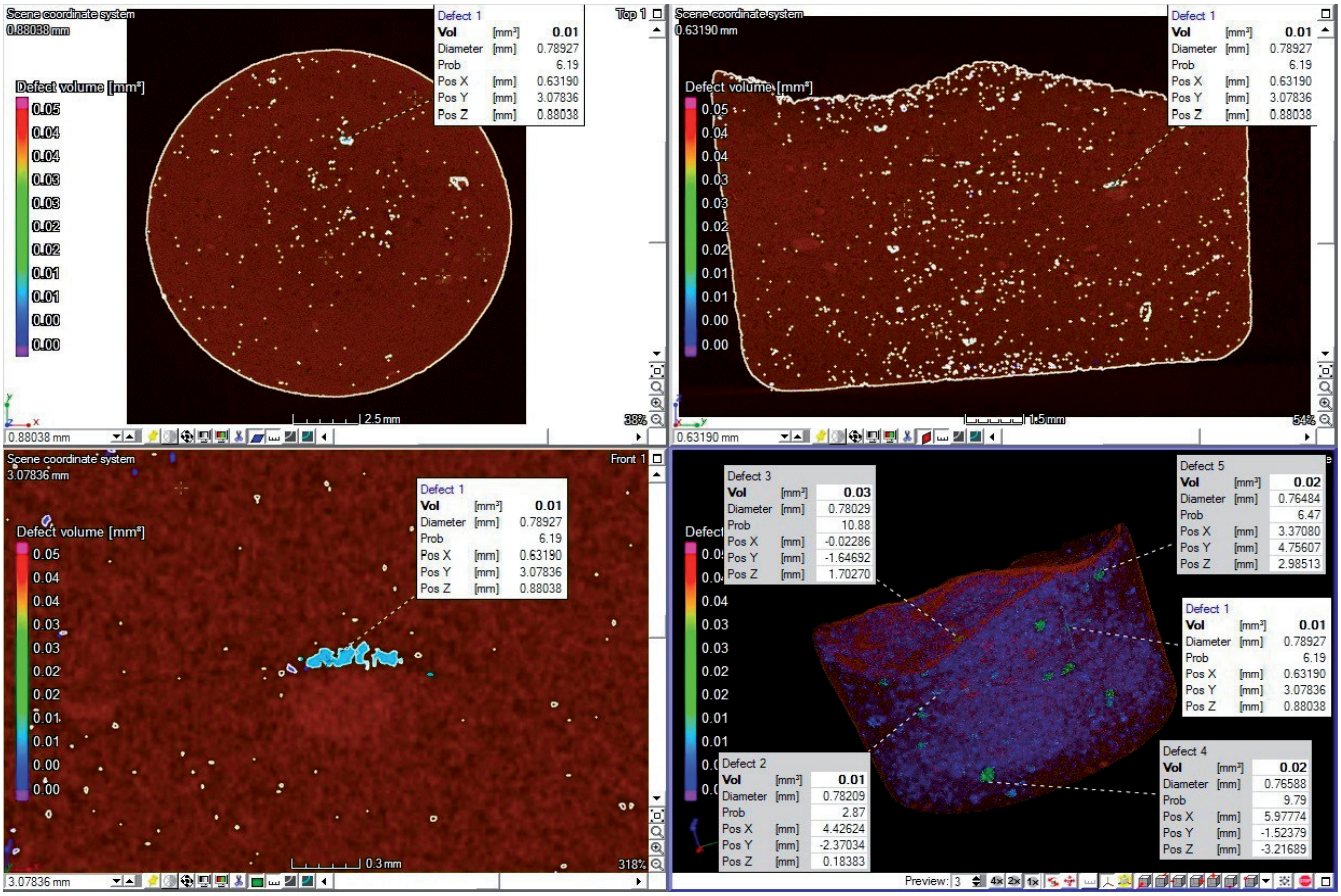

Fig. 2. Cloud of points used to reconstruction process

4. Results and discussion

The μCT technique is developing rapidly. Just a few years before while μCT was a novelty difficult to apply and demanding laborious calculations [3,10]. At the moment, there are many available applications allowing the reconstruction of the material from so called point clouds through at least several available algorithms. one of the commercially available applications is VGStudio MAX 2.2 the Volume Graphics company that provides several algorithms for reconstruction.

Basic algorithm operates upon the principle of analyzing the entire point cloud, in terms of the limit set for gray level for the environment detail "background" and checks the gray level of a given voxel. If this level corresponds to the level of air (low values of the gray level), it recognizes it as a defect – the emptiness (it's smaller than the defined limit).

Advanced algorithm "Default (v2.1)" is suggested by the producer as the most optimal. This algorithm "Default (v.2.1)" operates for further analyzes of the adjacent areas to detected defects with making a few iterations (Fig. 2).

As a result of the transformation 3D point cloud Figure 3 were obtained.

Subsequently the reconstruction allowed us to receive "an image" of the sample created on the basis of available algorithms (Fig. 4).

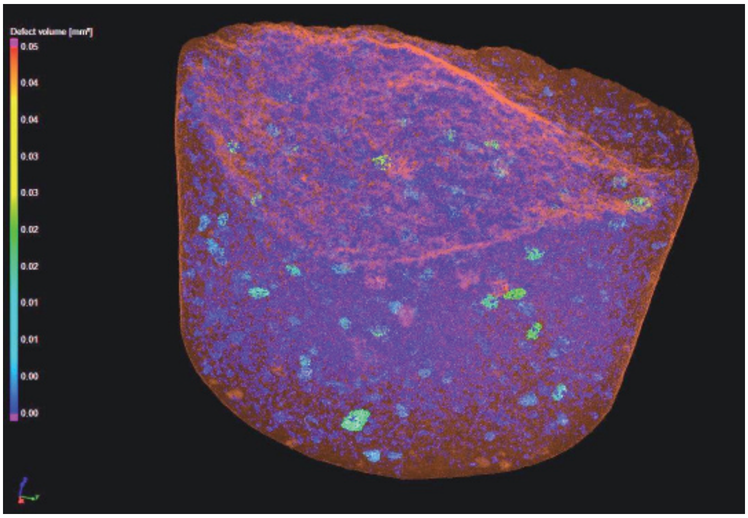

Fig. 3. 3D point cloud transformation

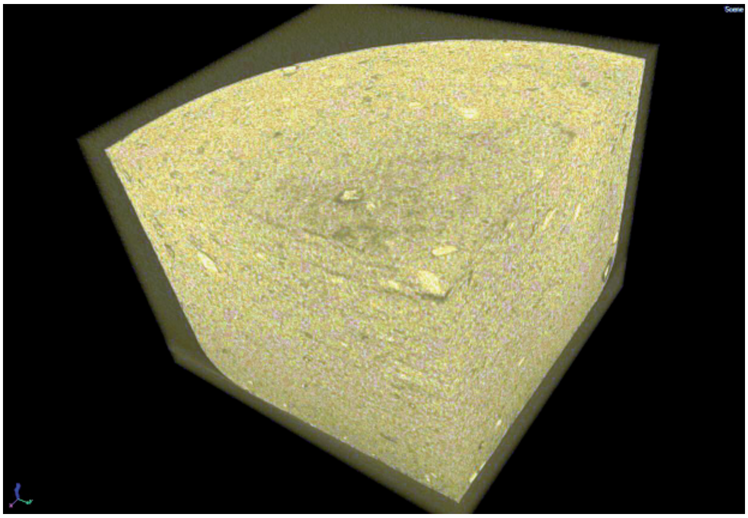

Fig. 4. Reconstructed portion of the element

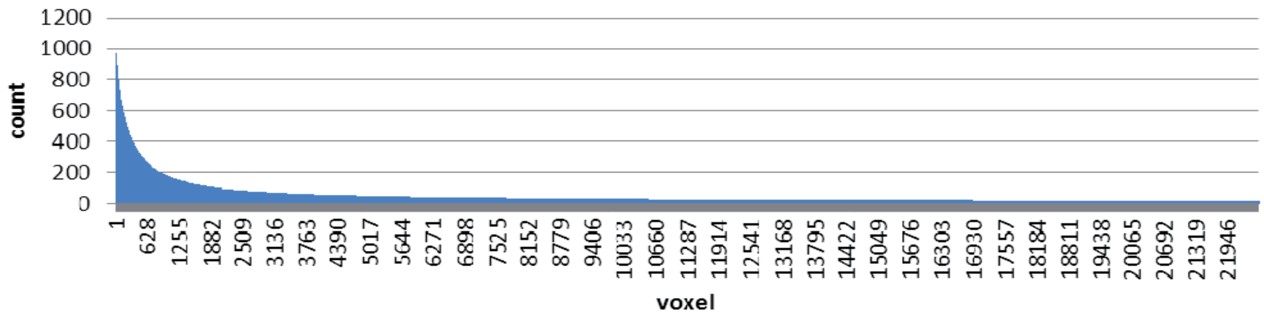

Fig. 5. Size distribution of the pores in a volume (voxels) of the test element

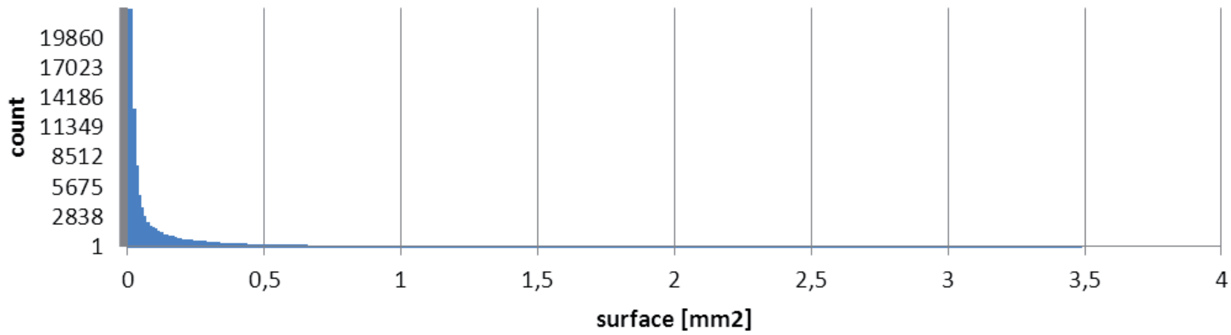

Fig. 6. Surface area distribution of the pores in a volume of the test element

After the reconstruction there are available options such as:

- automatic and fast detection, analysis and visualization of pores or inclusions in the material,
- volume, position, size and surface area are determined for every individual pore/inclusion,
- color-coding of pores or inclusions according to volume,
- defect size statistics, total percentage of porosity and pore volume histogram,
- analysis of the entire object or Regions of Interest.

For the works on materials for heat transfer extremely important is a pore size distribution in the volume of the heat exchanger [11-14]. As a result of reconstruction there were obtained the size distribution of the pores in a volume of the test element (Fig. 5) and surface area distribution of the pores in a volume (Fig. 6).

5. Conclusions

This technic will allow prediction of phase transport properties from direct computations on digitised images of the pore-space in porous materials with important implications to mass transfer processes.[8,10] X-ray micro-tomography are powerful non-destructive measurement techniques allowing to obtain pertinent information relative to the 3D internal structure of porous materials [9]. Structural parameters like porosity, mean pore size and specific surface area have been computed on 3D images obtained by micro tomography [6]. These geometrical parameters have been introduced in models reported in literature to predict thermal effects boiling in the foam. The results of this kind of opportunity for detailed modelling heat exchange as the basis for future experiments and applications for use metallic foam.

The µCT methods provide more accurate results with the whole volume of the sample than those applied so far with image analysis method based on microphotography. Image analysis methods are adequate for small volume observations.

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