doi: 10.1017/S1431927609093234
Microsc Microanal 15(Suppl 2), 2009

Surface Area Determination of Metal Ceramic Composite by FIB Sectioning and BET Measurements

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Surface area determination of porous composite materials are straight forward if the total surface area is of interest. However, to measure each materials surface area contribution to the total often requires more elaborate techniques e.g. chemisorption needs to be material specific and is therefore dependant on the gas medium used. In our case we want to investigate the surface area of a porous metal structure with ceramic particles located the pore walls.The ceramic exhibits a much higher surface area than the porous metal structure and thus its contribution to the total surface area is significant. An as prepared sample cross-sectioned and polished prior to mounting for FIB serial sectioning using a Zeiss 1540XB crossbeam FIB FEGSEM. A 200 by 200 micron area was selected with a milling depth of 50 microns. An image was acquired after each slice with a thickness of 190 nm. The images were then processed to correct for misalignment alignment and intensity bias using in house image processing routines in MATLAB™. A script is applied to distinguish pores from metal. The data was used for 3D reconstruction of the pores (see Fig. 1). From the 3D reconstruction it was possible to calculate both the total pore volume and the surface area of the pore walls. Using a volume of $1.94 \cdot 10^{14}$ nm^3 of the sample the resulting volume specific surface area of the metal was $1.69 \cdot 10^{-4}$ nm^{-1}. Volumes with artifacts such as the surface of the sample and areas where milling-remains block the micrographs have been ignored.

With a measured geometrical density of 5.28 g/cm^3 for the metal sample, the FIB value of the metal volume specific surface area ($1.69 \cdot 10^{-4}$ nm^{-1}) results in a surface density of 0.032 m^2/g. From krypton adsorption measurements a BET surface area of 0.05 m^2/g was recorded for the corresponding sample. The higher value from BET is expected, since the measurement is the total surface area i.e. metal and ceramic whereas the value by FIB is almost exclusively from the metal.
Measuring the BET surface area for comparison of pure metal that has been sintered without ceramic is not optimal as the ceramic acts as a sintering inhibitor resulting in larger surface area. The BET surface area for a single metal phase sample is 0.004 m^2/g i.e. roughly ten times smaller due to sintering.

The conclusion is that FIB sectioning in combination with image analysis provides a good indication on the surface area of the metal. Additional information from the 3D reconstruction such as metal volume or closed pore volume is also valuable in the understanding of material properties.

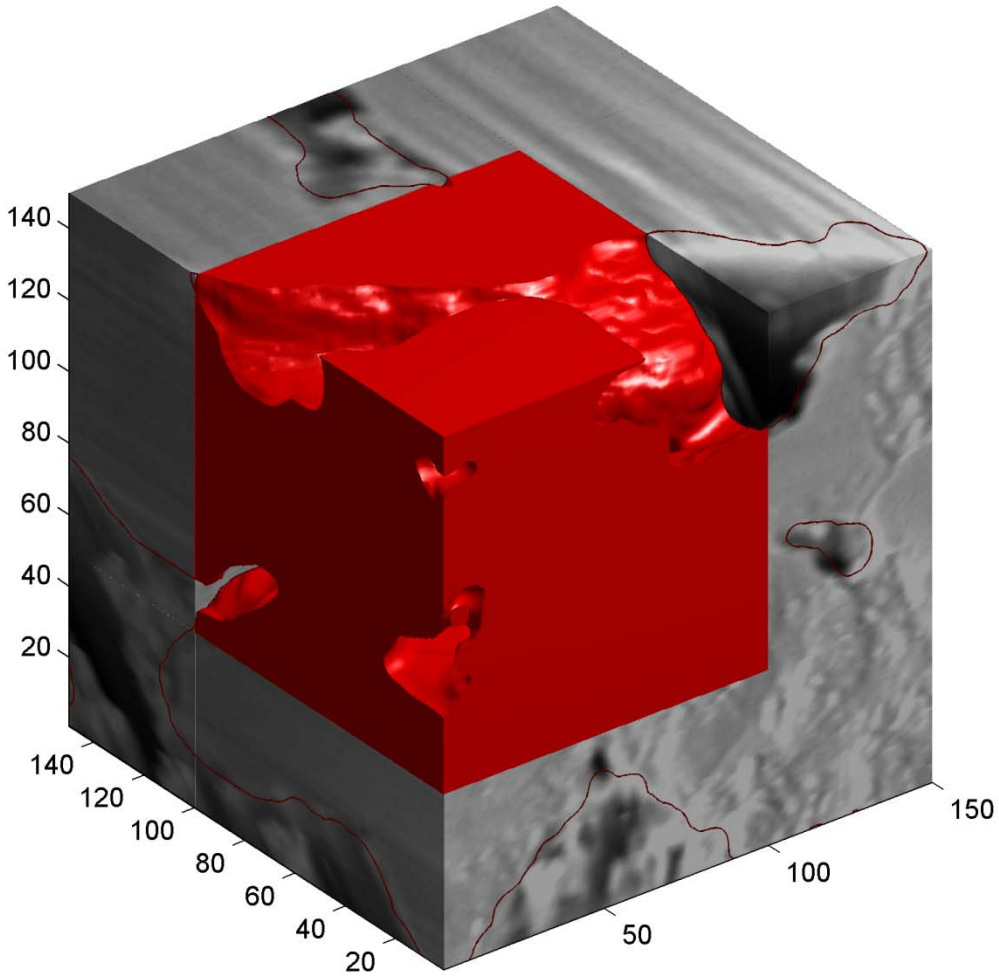

Fig. 1. FIB 3D reconstruction of the porous network. The grey cube illustrates the entire data set after stacking the raw SEM images. Red lines illustrate the metal/pore phase boundaries. The red inset illustrates segmented and reconstructed metal phase revealing the porosity morphology.

https://doi.org/10.1017/S1431927609093234 Published online by Cambridge University Press