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3D Microanalysis of Porous Copper Using FIB-Tomography in Combination with X-ray Computed Tomography

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Porous metals with their unique mechanical and physical properties have been the subject of extensive research over the past few decades. While providing high surface-to-volume ratio and low density, porous metals can be tailored to meet the requirements of a wide variety of industrial applications, including filters, cushions, electrodes or heat sinks [1-4]. However, there are still several scientific and technological questions to be answered with respect to understanding and ultimately controlling pore morphology and interconnectivity of porous metals. Correctly evaluating porosity, pore volume and pore size distribution within the volume is a crucial first step when aiming at a correlation between the structure of the material and its mechanical and physical properties. In this context, especially the microstructure of the porous material plays an important role since it has a large impact on its future performance. Therefore, the use of experimental methods which provide as accurate as possible 2D and 3D data from the nm to the mm scale as well as the development and application of accurate image analyzing algorithms are crucial.

In this work we develop a characterization toolbox suitable for analyzing the micro structure of porous copper samples relevant for future industrial application. This comprises (1) the measurement of the microstructure by using focused ion beam- scanning electron microscopy (SEM-FIB) including FIB tomography (FIB-SEM-tomography) for 3D imaging, (2) the application of high resolution X-ray computed tomography (XCT) and (3) the development of suitable image analysis algorithms to obtain accurate results regarding the microstructure of the porous samples from the 2D and 3D grey value data. The goal in the first stage is to gain information regarding the geometry of the sample, the porosity and the 3D distribution of the pores to go towards a better understanding of the physical characteristics of the material.

The sample is basically defined by a thin porous copper film with a thickness of about 30 μm. For the FIB-SEM and FIB-tomography, we use a cross-beam scanning electron microscope from Zeiss (AURIGA® - CrossBeam workstation). The cutting depth chosen for the FIB-tomography data is 100 nm. The chosen ROI is about 15 x 15x 10 μm^3. For the complementary XCT measurements we use a nanotom m from GE. The XCT system has a focal spot size of 800 nm and gives the possibility to measure for these particular samples a voxel size V down to about $V_x=V_y=V_z=500$ nm. The ROI used for the microanalysis is about 400 x 270 x 24 μm^3. The combination of FIB-tomography and XCT gives the opportunity to gain 3D information from the mm down to the nm scale. For the analysis of the 2D and 3D structural data we discuss the use of the commercially available imaging software Avizo® from FEI. For the segmentation of the pores we apply a watershed based algorithm and discuss the use of image post processing and its impact on the results. The thickness of the porous sample was measured by using the XCT 3D data. For statistical reasons, we measured the thickness at 448 points using the grey value data. It is shown that the mean thickness value is about 27.3 ± 2 μm. The first analysis of the obtained 3D data shows a porosity of about 30 %. Complementary to XCT and FIB-tomography, we used a gravimetric method in order to obtain an estimate of the porosity of the material. For this purpose, porous copper samples on silicon substrate of a defined volume are weighed and subsequently etched. The mass difference before and after etching gives the density of the sample. By dividing the calculated density of the porous sample by the density of bulk copper, the average porosity of the sample can be calculated. The difference between the gravimetrical and the 3D evaluation is about 10 %. We conclude that the difference can be explained by the error in the measured mass. Once the porosity can be extracted accurately, the porosity in terms of size and volume distribution in the x-, y- and z-plane and information regarding the interconnectivity of the pores can be evaluated. Further studies to improve the accuracy of the analysis and to relate the findings of the microanalysis to mechanical-, thermal- and electrical properties are planned.

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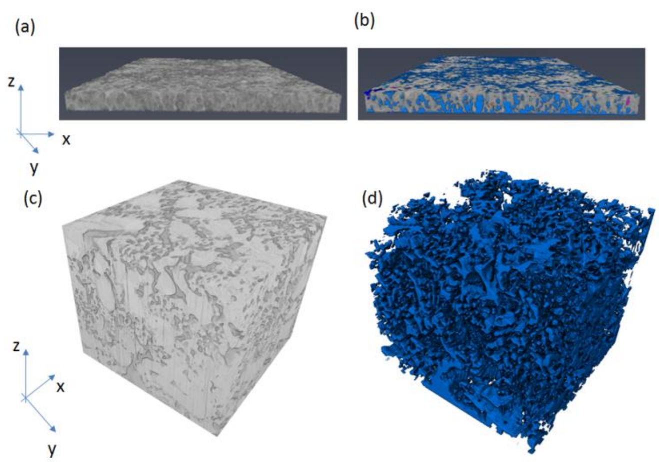

Figure 1. (a) XCT image with $V_x=V_y=V_z=500$ nm. (b) Segmented XCT image. The ROI for the XCT measurements is about 400 x 270 x 24 μm^3. Different color code gives information regarding of how the pores are connected to each other. (c) Reconstructed 3D volume obtained from SEM-FIB-tomography. (d) Segmented SEM-FIB-tomography. The ROI for the SEM-FIB-Tomography is 15 x 15 x 10 μm^3.

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