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Deformation behavior of nanoporous polycrystalline silver. Part I: Microstructure and mechanical properties

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ABSTRACT

This work reports on the microstructure and mechanical properties of porous silver films produced with different sintering parameters. The microstructure is investigated by high-resolution ptychographic X-ray computed tomography and scanning electron microscopy. The mechanical properties are obtained from constant strain rate tensile tests. It is found that for this class of samples both grain size and ligament diameter have little effect on the magnitude of the yield stress. Instead, a clear correlation is found between strength and porosity.

1. Introduction

Sintered porous silver is an attractive material for joining technology and is considered as a candidate to replace high-lead containing solders for die-attach materials in high power electronic packaging [1–7]. A decisive property for a potential application as joining material is the behavior during thermomechanical fatigue. This depends strongly on the role played by the pores during deformation. Porous materials exist in several forms such as foams, cellular materials with truss structure and nanoporous foams [3]. Several models were proposed to make a connection between the macro mechanical properties and the microstructure of porous materials. For instance, Dewey [8] reported a linear relation between the elastic modulus and the porosity, while according to Bert et al. [9], Mackenzie [10], Bal'shin [8] and McAdam [8] the elastic modulus and porosity are related by a power law relationship [8]. Duckworth [11], Spriggs [12] and Wang [13] proposed an exponential relation between the elastic modulus and the porosity. The model proposed by Ashby and Gibson [14] is one of the most widely used for high-porous foams, in which the connectivity of the pore structures is the main determining factor for the mechanical properties. The pore volume fraction or relative density (ρ^*/ρ) is defined as the second principal parameter. The shape, ratio of the thickness to the length and the curvature of the cell edges are other factors that affect the macro mechanical behavior [15–18]. According to Ashby and Gibson [14], the Young's modulus and yield stress of high porous open-cell foams [$(\rho^*/\rho) < 0.3$] are proportional to, respectively $(\rho^*/\rho)^2$ and $(\rho^*/\rho)^{3/2}$ when cell edge bending is the dominant deformation mechanism. There are only few studies reporting on the connection between the porous microstructure and the mechanical behavior of sintered nanoporous silver [19]. The behavior of porous silver membranes under tensile stress was studied in detail by Tanvir Ahmed and Jankowski [20–22]. Plastic deformation was found to occur inside the silver filaments while aligning along the direction of the applied stress. The silver filament size and the porosity were defined as the main characteristic parameters determining the flow stress. In situ X-ray diffraction experiments during tensile deformation of sintered porous silver films have revealed that the deformation behavior strongly depends on the porosity [19]. In films sintered at lower pressure/temperature/time the deformation behavior is mainly controlled by the pores and the local bending of

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the ligaments. In contrast, the deformation in films with higher porosity is predominantly controlled by intra-granular dislocation based mechanisms [19]. In order to study the influence of connectivity, pore size and shape distribution and other microstructural parameters a detailed knowledge of the microstructure is needed. In a first attempt the 3D porous morphology of sintered films was investigated using full field X-ray nano tomography with a spatial resolution of 150–200 nm [19]. However, this limited resolution did not allow observing small isolated pores, which are visible in SEM images. As a consequence, such 3D images are not good enough for finite element microstructure-based modelling. In the present work we take a different approach. The porous structure of the silver foams is characterized with higher resolution using multi-keV ptychographic X-ray computed tomography (PXCT). Scanning X-ray diffraction microscopy (SXDM), also known as ptychography, is a recently developed high-resolution imaging technique that provides images of both the phase and amplitude of the complex transmission function from the measured object [23,24]. In combination with tomography, ptychography has demonstrated to provide quantitative three dimensional electron density maps of the specimen [25,26] with a high resolution without changing the microstructure. A resolution in 3D down to 16 nm has been recently obtained [27]. The technique has been applied to investigate various porous materials such as organic fiber [28], catalyst body [29] and avian eggshell [30]. This work reports on the microstructure and mechanical properties of 8 porous silver films produced with different sintering parameters. The microstructure is investigated by PXCT with a 3D resolution in the range of 35–55 nm and by scanning electron microscopy. The mechanical properties are obtained from constant strain rate tensile tests. In a subsequent paper [31] the high resolution 3D images are used as input in a finite element based model allowing the calculation of the mechanical properties of several samples with different porous structures.

2. Experimental setup and methodology

2.1. Sample fabrication

Various nano-porous silver films are produced by pressure-assisted sintering. The starting material is bought commercially in the form of 100-μm-thick silver laminates [19], which consist of silver paste printed on a foil with a dimeter of 80 mm and heated to a pre-stage sintering temperature [5]. These laminates are sintered at temperatures between 210 and 300 °C and pressures between 4 and 12 MPa for a duration of 3–10 min, resulting in set of silver films with different microstructures. Eight of these samples (S1–S8) are selected to be investigated by PXCT. S1 and S8 are the samples sintered under two extreme conditions with a pressure of 4 MPa and 12 MPa, temperature of 210 °C and 300 °C and time of 3 min and 10 min, respectively. Table 1 lists the sintering conditions of all 8 specimens.

Table 1
Sintering conditions of the samples investigated by PXCT.

Sample name	Temperature (°C)	Pressure (MPa)	Time (min)
S1	210	4	3
S2	250	4	10
S3	250	8	3
S4	300	8	3
S5	250	8	10
S6	210	12	10
S7	250	12	10
S8	300	12	10

For the PXCT measurements pillars with diameter of 4–5 μm and heights of 15–20 μm are machined out of the silver films using a focused ion beam (Ga^+ FIB model Zeiss NVision 40 CrossBeam). They are attached to a dedicated holder [32] with carbon deposition. The final FIB polishing is performed with a current of 700 pA and a voltage of 30 keV. Fig. 1 displays a representative scanning electron microscopy (SEM, Zeiss Supra VP55) image of a nano-porous silver pillar prepared for the PXCT measurement.

2.2. Grain size distribution

The grain size distribution of the specimens is determined from SEM images taken in the plane of the film. In a first step the grain boundaries and the pores are located using the edge detection routine from the GIMP2 software package [33]. Then the "Particle-analysis plugin" of ImageJ [34] is employed to calculate the surface area of the grains. Finally, considering a circular shape for the grains, the grain size distribution is derived from the measured surface area.

2.3. PXCT measurements and reconstructions

The ptychography measurements are carried out at the coherent small-angle X-ray scattering (cSAXS) beam-line at the Swiss Light Source (SLS) at the Paul Scherrer Institut in Villigen, Switzerland. In ptychographic imaging, the resolution depends mainly on the largest scattering angle at which diffraction signals can be reliably recorded and on the accuracy and stability of the sample position [27]. For these measurements we use a high-accuracy positioning instrument working in air and at room temperature which uses laser interferometry for nanometer-precision positioning of the specimen with respect to the beam-defining optics [27]. A coherently illuminated Fresnel zone plate (FZP) made of Au [35] is used to define the illumination onto the specimen. Sample S5 is measured with a photon energy of 6.2 keV and a FZP with a diameter of 100 μm, an outermost zone width of 100 nm and focal length of 50 mm, providing a flux of 2.3×10^8 photons/s. The sample is placed about 0.85 mm downstream the focus such that the illumination at the sample position has a diameter of about 1.7 μm. A

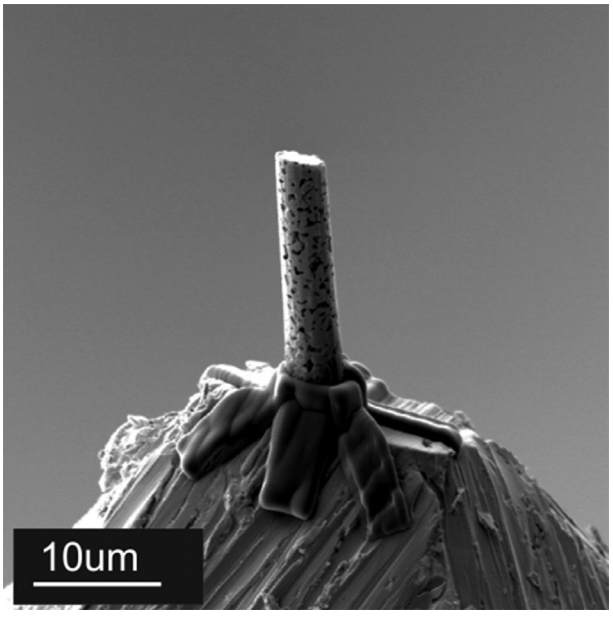

Fig. 1. Representative SEM image of a silver pillar prepared for PXCT.