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Stiffening of sub-micro-porous silver membranes under tensile deformation

H.S. Tanvir Ahmed*, Alan F. Jankowski

Texas Tech University, Mechanical Engineering, Box 41021, Lubbock, TX 79409, USA

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ABSTRACT

Tensile testing of porous silver membranes is used to assess the postulate that the onset of plasticity corresponds with a shear type of deformation. The filament size and porosity take the parametric roles of feature size and free volume that are used to characterize the strain rate sensitivity of strength in nanoscale metals as now adapted for porous membranes. Experimental measurements indicate the porous membranes stiffen under tensile loading as plastic deformation progresses, as evidenced by an increase in the elastic modulus from a value consistent with the C' elastic constant towards the C_{44} elastic constant.

1. Introduction

The plasticity of dense nanocrystalline metals [1–3] is governed by dislocation motion, grain boundary structure, and the free volume to accommodate deformation. An analysis of strain rate dependent strength can be based upon these structural features, wherein the strain rate exponent (m) is determined from a power law variation of stress (σ) as a function of strain rate ($\dot{\varepsilon}$), i.e.:

$$\sigma = c_{\mathrm{m}} \dot{\varepsilon}^{\,\mathrm{m}} \tag{1}$$

where c_{m} is a constant. The plastic behavior of porous ductile solids to rupture can be described [4,5] by considering microvoid nucleation, growth, and coalescence to form macroscopic cracks. Theoretical approaches have been developed [6,7] to account for the effect of void locking by inclusions. The role of porosity in the loss of Hall–Petch strengthening and the reduction of compressive strength of nano-grained ceramics is accounted for [8] through constitutive modeling of individual phase and grain boundaries. Constitutive modeling [9] for power-law viscous solids containing spherical voids yields a generalized elliptic form for the macroscopic strain rate potential with a simple dependence on the void volume fraction.

A combination of mechanical properties makes the porous materials attractive for many engineering applications. Being lightweight and having high surface area per unit volume, porous metal membranes are considered ideal [10] for lightweight structural sandwich panels, energy absorption devices and heat sinks. Use of porous metal coatings is increasing in renewable-energy system applications [11] such as solar cells and hydrogen fuel cells. In low temperature thin film fuel cells [12], the scale of porosity of metal coatings is particularly important for their catalytic performance. Potential future uses of nanoporous materials include electrochemical [13] or chemical [14] actuation, tunable conductors [15,16] and magnets [17,18]. The mechanical stability of the porous coatings is essential in such applications, where deformation may originate as rapid thermal stress–strain cycles. Thus, developing an understanding of the mechanical behaviors of these porous membranes is remarkably important. Although the mechanical properties [10,19] and deformation of porous metal membranes are generally considered under compressive loading, behavior similar to bulk nanocrystalline metals is recently reported [20] for the tensile testing of highly porous silver membranes. The mechanical strength of free-standing silver membranes with constituent submicron porosity was investigated as a function of strain rate from 10^{-4} to 10^{-1} s^{-1} to better understand the operative deformation mechanisms across multiple length scales of structure. Unlike models for porous metal plasticity that require void nucleation and growth, herein the composite structural features that can contribute to the strain rate dependent strength behavior include the porosity, filament (or strut) size, and the constituent grain size. It is found [20] that the strain rate sensitivity exponent for porous silver membranes increases from 0.02 to 0.05 with a decrease in the filament size below 10 μm along with an increase in the porosity from 25 to 50% for submicron pore sizes. A corresponding model was developed [20] to account for how the porosity and filament size affect the strain rate exponent. An increase in the strain rate sensitivity of the porous silver specimens was found to occur with

* Corresponding author. Tel.: +1 806 742 3563; fax: +1 806 742 3540.
E-mail addresses: tanvir.ahmed@ttu.edu (H.S.T. Ahmed), alan.jankowski@ttu.edu (A.F. Jankowski).

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a functional decrease in the filament length scale and an increase in porosity. The interpretation postulated is that the porous membrane will first plastically deform as an open cell structure, and then continue wherein the open cell structure collapses through shear deformation under tensile loading as the filaments (or struts) realign with the load direction through a bending-shearing mode, primarily at the junctions between the filaments. Although the tensile testing was conducted at different displacement rates to assess the strain rate sensitivity, the elastic modulus prior to initial yielding of the monotonic loading appears to be invariant as a function of strain rate. Linear extrapolation of porous data to the fully dense condition produces a modulus value of ~25 GPa that is between the C' and C_{44} elastic constants. The C' elastic constant, which is ½(C_{11}-C_{12}), for silver is calculated to be 15 GPa where the typical values [21] at room temperature for the C_{11}, C_{12}, and C_{44} elastic constants of silver are 124 GPa, 93 GPa, and 46 GPa, respectively. For comparison, fully dense silver samples were tested in tension and the average elastic modulus was found to be ~46 GPa. Since, the linear extrapolation of the elastic modulus of the porous samples did not predict the experimental modulus at fully dense condition quite accurately, a non-linear approach was taken to fit the obtained data set. For additional reference [22], the tensile strength of silver wire annealed at 873 K is 125 MPa whereas the elastic modulus of silver wire strained to 5% then annealed at 523 K is 71 GPa.

The objective of the tensile experiments proposed in this study is to assess the postulate that the initial yield point of porous metal membranes under tensile loading can correspond with a type of shear deformation. The tensile testing will again be conducted at different displacement rates to assess the strain rate sensitivity from 10^{-4} to 10^{-2} s^{-1}. For the mode of plastic deformation to change, the elastic modulus measured from tensile loading should change. To assess the change in modulus with plastic deformation, the loading curve will be interrupted [23,24] after initial yielding. Full unloading and reloading cycles will follow the initial onset of yielding to probe whether or not the plastically deformed porous membrane structure has indeed stiffened by changing its deformation mode from primarily mixed mode shearing towards uniaxial. Thus, it is suggested that an increase in the modulus will progress with the amount of plastic deformation until the ultimate strength level is reached. Thereafter, localized necking will reduce the cross-section so that further deformation will provide a decrease in the engineering stress and computed elastic modulus.

2. Materials and methods

2.1. Materials

The tensile specimens [20] are die cut from 25 mm diameter ($2r$) porous silver membranes of 0.9997 purity that are 57–79 μm in thickness (h_{ts}). The nominal pore sizes (h_{np}) are 0.22, 0.45, 0.80, and 3.0 μm. The average foil thickness (h_{ts}) is measured using a micrometer from a stack of 10 foils and confirmed using scanning electron microscopy (SEM) images [20] of foils viewed in cross-section. The membrane density (ρ) is determined from a microbalance measurement of weight (w) as

$$\rho = w(\pi r^2 h_{ts})^{-1} \quad (2)$$

The porosity (p) is computed as the ratio of the actual density (ρ) in comparison to the full density of silver (ρ_{Ag}) that equals 10.5 g/cm^{-3} as

$$p = 1 - \rho(\rho_{Ag})^{-1} \quad (3)$$

The porous silver membranes were imaged [20] with an SEM in plan view (as seen in Fig. 1) and cross-section to provide surface morphology and structural features including some definition

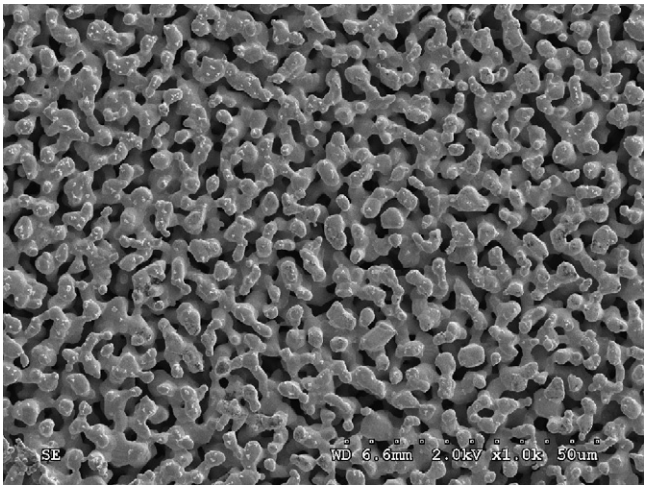

Fig. 1. SEM image of a 0.8 μm pore size membrane in plan view. The scale shows 50 μm.

of grain size within each filament (or strut). The linear intercept method is applied [20] to determine the average filament size (h_f) from the plan view images as follows. The average length for the intercept points of the filaments along each of six radial traces (equally spaced at 60°) is measured from three (or more) random points with the plan view image. The grain size (h_g) is measured as the average of smallest widths measured for each filament, assuming that the filaments (or struts) have a bamboo-like structure. The average grain size is found to be 2.47 ± 0.19 μm, irrespective of the nominal pore size for each sample. The dimensions for h_{ts}, p, and h_f are listed in Table 1 for the membranes of each nominal pore size.

2.2. Porosity effect

The cross-sectional area (A_c) is evaluated by assessing the dimensional nature to the porosity of the membrane. A cross-sectional area (A_c) equals a product of the nominal cross-sectional area (A_n) corrected with the porosity (p) according to the expression [10] for open cell porous materials as

$$A_c = A_n(1-p)^n \quad (4)$$

The exponent (n) accounts for the dimensional nature of the membrane. For a two-dimensional pore structure, i.e. pores that transit through the thickness only, n equals one. For a three-dimensional pore structure, i.e. pores which transit in-plane as well as through the thickness, n equals 1 ½. This later case is found [20] for the silver membranes of this study.

2.3. Tensile testing

The tensile bars from the porous and fully dense samples have a gage length (z_g) of 10 mm and a 3 mm width. The tensile specimens are mounted using detachable clamps with serrated grip surfaces and are tested using a universal tensile testing machine. The strain rate experiments are conducted by moving the crosshead of the linear actuator a fixed displacement (Δz) at different speeds. A total displacement of 10 mm occurs during a displacement time (Δt) that is varied from 10^2 to 10^4 s. The normal tensile load (P) is recorded as the crosshead position (z) is measured as a function of time (t) using a displacement transducer, i.e. a linear variable differential transformer (LVDT). The yield stress (σ_y) of the sample is determined from a plot of the engineering strain (ε_e), i.e. $\Delta z/z_g$, versus the engineering stress (σ_e). The elastic modulus (E) is calculated