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Elastic characterization of nanoporous gold foams using laser based ultrasonics

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ABSTRACT

A resonance based laser ultrasonics technique is explored for the characterization of low density nanoporous gold foams. Laser generated zero group velocity (ZGV) lamb waves are measured in the foams using a Michelson interferometer. The amplitude spectra obtained from the processed time-domain data are analyzed using a theoretical model from which the foam Young's modulus and Poisson's ratio are obtained. The technique is non-contact and nondestructive, and the ZGV resonance modes are spatially localized, allowing for spatial mapping of the bulk sample properties. The technique may be suitable for process control monitoring and mechanical characterization of low density nanoporous structures.

1. Introduction

Nanoporous materials possess unique nanostructures that can be tailored through processing to develop functional materials with unique surface and bulk physical properties [1–3]. Nanoporous metal foams belong to a class of nanostructured materials, produced through free alloy corrosion or electrochemical etching, and possess open cellular nanostructures comprised of monolithic arrangements of nanoscale solid ligaments and voids. The open material architecture of nanoporous foams is produced through selective removal of atoms of less noble species in an alloy solid solution leading to the formation of porosity at the atomic scale. This process is followed by rearrangement and agglomeration of atoms of the noble metal and the formation of interconnected nanoscale solid ligaments and open voids. Nanoporous foams have large surface area to volume ratio, accessible solid-pore interfaces, and exhibit size-dependent and interface-controlled material behavior. Nanoporous foams are promising candidates for applications including; chemical sensing [1–3], electrochemical actuation [4–6], catalysis [7–9], surface-enhanced Raman scattering [10,11], etc.

Understanding the influence of the nanostructure on the bulk properties of dealloyed metals is critical to designing foams that are functionally reliable and have mechanical integrity. This work is concerned with measurements to understand the influence of the nanostructure on the linear elastic behavior of low density open cell foams. Experimental results in the literature show that the Young's modulus (E) of low density macroporous open cell solid is related to the foam density (ρ) by $E/E_s = C(\rho/\rho_s)^n$, where E_s and ρ_s are the modulus and density of the solid skeleton [12]. The constants C and n which varies between 1 and 4, depend on the geometric arrangement of the foam cell elements and the mode of elastic deformation, (i.e. bending or uni-axial compression) [13]. Reports of Young's modulus measurements of nanoporous gold in the literature have significant scatter [14], attributed to different factors, including, variability in the properties of the solid skeleton due to surface stress at the cell interfaces [15], size dependent elastic properties of the solid skeleton [15,16], and residual silver content in partially dealloyed gold foams [17,18]. In addition, artifacts resulting from sample modification due to densification [19] and complicated loading fields particularly in nanoindentation measurements can lead to deviation of the measured properties from their actual values.

In this work, we make measurements of the bulk elastic properties (Young's modulus and Poisson's ratio) of nanoporous gold membranes with different densities using a laser based ultrasonic (LBU) technique. LBU techniques [20] are non-contact and nondestructive, allowing for elastic characterization, free of artifacts due to mechanical loading of the sample. In this work, we obtain the elastic properties of the porous membranes by matching the transit time of bulk elastic waves and the frequency of two zero group velocity (ZGV) Lamb wave resonances in the nanoporous membranes to a theoretical model. The resonance frequencies of ZGV modes in bulk elastic solids have been shown to depend on the plate thickness and Poisson's ratio (or ratio of the bulk wave velocities) [21], and the lateral spatial extent of the resonance modes is

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confined to the wavelength of the Lamb wave mode [21]. Unlike bulk solids, porous plates support two types of Lamb modes namely, structure-borne guided waves, which propagate with either the Rayleigh or shear wave velocities at the high frequency limit, and fluid-borne guided waves whose phase velocity tends to the velocity of the Biot slow wave in the high frequency limit [22]. Since the wavelength of the Lamb waves used in this work (>20 μm) are significantly larger than the pore size of nanoporous gold, the porous sample is treated as an equivalent homogeneous elastic solid with effective bulk properties that depend on the solid structure and the entrained air in the pores. Our results show that the Young's modulus of the nanoporous gold membranes follow a power law dependence on the foam density like microporous foams. The measurement technique may be suitable for process control monitoring during sample fabrication in order to optimize the foam elastic properties.

2. Zero group velocity lamb waves in a bulk elastic plate

Lamb wave propagation is a classical problem of guided wave motion under plane strain conditions in a plate with vanishing tractions at the upper and lower plate surfaces. For a homogeneous and isotropic elastic plate with bulk longitudinal and shear wave velocities, c_L and c_T, the Rayleigh-Lamb dispersion relations [23], yields the wavenumber k and frequency ω for several symmetric S_j and antisymmetric A_j Lamb wave modes, where $j = 0, 1, 2 \ldots$ designates the order of the mode. Some of these Lamb modes exhibit anomalous behavior at frequencies where the group velocity $c_g = d\omega/dk$ is zero while the phase velocity $c_p = \omega/k$ is finite. At these frequencies, strong plate resonances are produced and mechanical energy is confined in the plate close to the excitation source. There have been numerous reports in the literature investigating zero group velocity resonances in bulk isotropic [21,24] and anisotropic plates [25] using lasers, air coupled transducers [26], and mechanical impact [25], as excitation sources.

Fig. 1 shows numerically calculated dispersion curves for a few Lamb wave modes in a plate with a Poisson's ratio of 0.2. The lowest order modes A_0 and S_0 are continuous over a broad range of frequencies, while the higher order modes originate at fixed cut-off frequencies. At the mode cut-off frequencies, the group velocity is zero and phase velocity is infinite, leading to standing wave resonances arising from multiple reflection of bulk waves in the plate. For instance, the cut-off frequency of the A_1 and S_1 modes are related to c_L and c_T and the plate thickness l by $f_{A1} = c_T/2l$ and $f_{S1} = c_L/2l$, as such, these frequencies are called the shear and longitudinal thickness resonance frequencies. In addition to these resonance modes, zero group velocity (ZGV) points occur on the dispersion curves of higher order Lamb modes where the slope of the curve is zero. These points are indicated by the dotted circles on the dispersion curves for the S_1 and A_2 modes. Unlike the mode-cut-off resonances, the phase velocities at these points are finite, indicating that the corresponding wavelengths of the Lamb modes are finite. Owing to their finite wavelength, these modes are spatially confined and are efficiently excited with localized sources [25–28]. Recently, Clorennec et. al. [21], demonstrated that the frequencies of these ZGV resonances can be related to c_L, c_T and l by, $f_1 = \beta_1\,(c_L/2l)$ and $f_2 = \beta_2\,(3c_T/2l)$ provided that the Poisson's ratio is less than 0.3. In these relations, f_1 and f_2 are the S_1 and A_2 ZGV resonance frequencies, and β_1 and β_2 are unknown constants that depend strictly on the Poisson's ratio [21]. Consequently, the ratio of these frequencies depends on the Poisson's ratio and is independent of the plate thickness. Fig. 2 shows a plot of f_2/f_1 for a range of Poisson's ratios. The curve shows a monotonic decrease in the frequency ratio with Poisson's ratio. Thus, provided that these resonance modes can be excited, the Poisson's ratio of the plate can be directly quantified. In this work, we use the Poisson's ratio of nanoporous gold foams obtained from measurements of the ZGV frequency ratio, as input parameters in a theoretical model developed previously for harmonic generated Lamb waves in an isotropic plate [27], to predict the longitudinal and transverse wave speeds. These properties are used to determine the plate Young's modulus using rule of mixtures formulation for the foam density.

3. Experiment

3.1. Sample fabrication

Nanoporous gold foams of various densities were synthesized by dealloying rolled silver (Ag)–gold (Au) alloys. The dealloying process involves selective leaching of the less noble Ag atoms from the alloy when soaked in an electrolyte, leaving the Au atoms, which aggregate by surface diffusion forming a spongy nanoporous metallic foam [1,29]. The Ag–Au alloys used were fabricated by melting Ag and Au pellets based on four weight percentages of 75–25%, 73–27%, 70–30%, and 67–33%, the larger weight percentage belonging to the Ag pellets. The Ag–Au alloys were melted repeatedly in an arc melter to homogenize the alloy solid solution. The resulting alloys were cold-rolled to a thickness of between

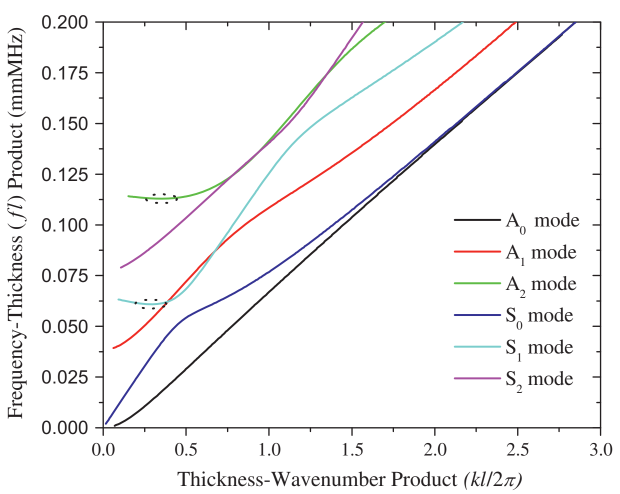

Fig. 1. Dispersion curves for an isotropic plate with a Poisson's ratio $v = 0.2$. l = plate thickness. Dotted circles in the figure show the position of the S_1 and A_2 ZGV resonance modes.

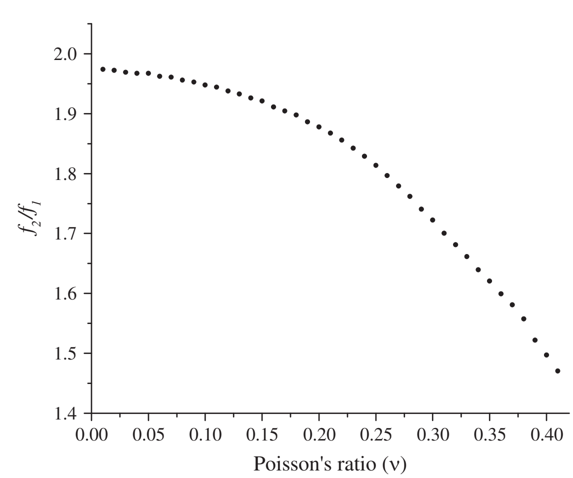

Fig. 2. Frequency ratio of S_1 and A_2 – ZGV resonances as a function of Poisson's ratio.