# *In Situ* Diffraction Strain Measurements in Aluminum-Mullite Microsphere Syntactic Foams

D. K. Balch,[1] D. C. Dunand[1]
[1] Materials Science and Engineering Department, Northwestern University, Evanston, IL, U.S.A.

## Introduction

Metallic foams are an attractive class of structural materials because of their low densities, high specific stiffnesses, high energy-absorbing capabilities, and good mechanical and acoustic damping capacities, among other attributes [1]. A particular class of foam structure called syntactic foams consists of hollow spheres embedded in a continuous matrix. Metallic syntactic foams containing hollow ceramic spheres can be fabricated by using traditional composite fabrication techniques; they have exceptional mechanical properties [2, 3].

Syntactic foams composed of an aluminum matrix reinforced with hollow silica-mullite ceramic spheres have been fabricated by liquid metal infiltration. By using synchrotron x-ray diffraction experiments to measure the strains present in the ceramic spheres, as demonstrated in Ref. 4, we show that considerable stresses are borne by the spheres during compressive loading. The degree of load transfer from matrix to sphere indicates that careful tailoring of the sphere material and geometry may lead to further improvements in the mechanical properties of metallic syntactic foams.

## Methods and Materials

In-situ x-ray diffraction measurements during compression testing were performed at the DuPont-Northwestern-Dow Collaborative Access Team (DND-CAT) at Sector 5 of the APS at Argonne National Laboratory. A table-top load frame (Interactive Instruments, Inc., Scotia, NY) was used to vary the compressive stress on a syntactic foam sample from 0 to -100 to 0 MPa in steps of 20 MPa, during which diffraction measurements took place at constant applied stress. The general setup for this experiment has been described in detail elsewhere [4]. The $14.98 \times 6.77 \times 6.63$-mm parallel-piped sample, while subjected to a constant uniaxial load, was irradiated for 900 s with a monochromatic 65-keV ($\lambda = 0.019$ nm) x-ray beam with a $0.5 \times 0.5$-mm square cross section aligned perpendicular to the sample compression axis. The 1.66-mm$^3$ diffracting volume contained about 23,000 microspheres. The transmitted Debye-Scherrer diffraction cones from the mullite grains present in this volume were recorded as rings by using a charged coupled device (CCD) camera, as was one Debye-Scherrer cone from a molybdenum powder standard attached to the sample for calibration purposes. The CCD camera (MAR, Inc., Evanston, IL) was positioned at a distance of 708 mm from the sample and had a 132-mm-diameter detector with 16-bit intensity readings over an orthogonal array of $64.4 \times 64.4$-μm pixels. Changes in the diameters of the diffracted mullite Debye-Scherrer rings during compressive loading are used to calculate the strains present in the mullite grains. The analytical procedure used is discussed in detail in Ref. 4, as is the use of the calibration standard to remove errors caused by sample movement or x-ray energy fluctuations.

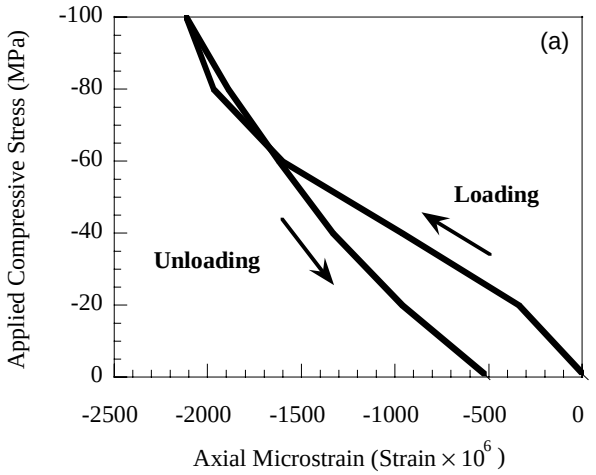
The syntactic foam sample consisted of a commercially pure aluminum matrix containing about 60 vol.% ceramic microspheres. The microspheres (Envirospheres PTY Ltd., Lindfield NSW, Australia) were composed of 45 vol.% crystalline mullite ($3\,Al_2O_3$ and $2\,SiO_2$) and 55 vol.% amorphous silica; they had diameters of 15-75 μm, wall thicknesses of 5-10 μm, and densities of 0.6-0.8 g/cm$^3$. The resulting syntactic foam had a density of 1.41 g/cm$^3$.

## Results

The applied compressive stress vs. mullite lattice strain curves (as measured by using the overlapping {120}/{210} lattice plane reflections in the diffraction experiment) are shown for both the axial and transverse directions in Fig. 1.

## Discussion

Fig. 1 shows the average axial and transverse lattice strains measured in the mullite grains within the microsphere walls as a function of applied stress on the foams by using the {120}/{210} lattice plane reflections. The axial strains (Fig. 1a) are measured in those grains with lattice planes oriented perpendicular to the applied load; i.e., the normal to the analyzed lattice plane is parallel to the applied load. The transverse strains (Fig. 1b) are measured on planes whose normals are perpendicular to the applied load; thus, the axial strains are a result of direct compressive loading, while the transverse strains are a result of Poisson expansion under applied load. In the following discussion, average strains and stresses are considered without accounting for spatial variations of stress within the microspheres. The {120}/{210} rings overlap in the diffraction pattern because of both the similar d-spacings of the planes (0.341 and 0.338 nm, respectively) and geometrical broadening caused by the sample thickness. They were

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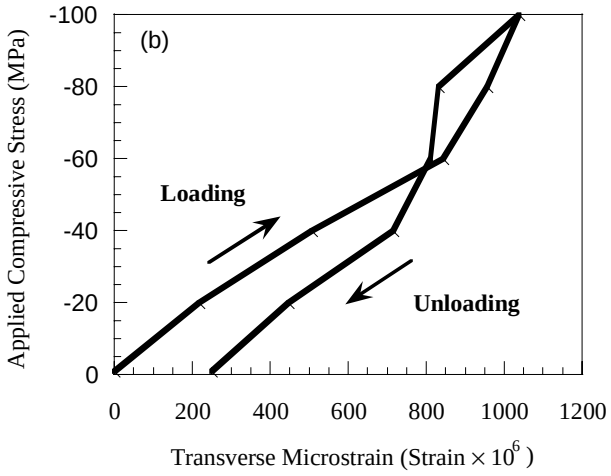


*FIG. 1. Applied compressive stress vs. mullite lattice strain: (a) Axial direction (along loading axis). (b) Transverse direction (perpendicular to loading axis).*

analyzed as a single ring. A separate analysis on the less-intense {110} ring, not presented here for brevity, yielded results in good agreement with the analysis of the {120}/{210} rings.

As seen in Fig. 1, both axial and transverse strains are significant in magnitude, reaching values of -2110 με in the axial and 1040 με in the transverse directions for an applied stress of -100 MPa. In both the axial and transverse directions, the lattice strains increase roughly linearly up to -60 MPa of applied compressive stress. From -60 to -100 MPa of compressive stress, both strain curves deflect upward, indicative of a change in loading of the mullite grains. During unloading, the axial strains decrease smoothly, with a steeper slope than was seen during loading. The steeper slope, as well as the deviations in the transverse strain unloading curve, may result from sphere damage and matrix plasticity altering the partitioning of load within the foam.

The stresses present in the mullite grains can be approximated from the measured strains by using the following equations [5]:

$$\sigma_1 = \frac{E}{1+\nu}\varepsilon_1 + \frac{\nu E}{(1+\nu)(1-2\nu)}\left(\varepsilon_1+\varepsilon_2+\varepsilon_3\right)$$

$$\sigma_2 = \sigma_3 = \frac{E}{1+\nu}\varepsilon_2 + \frac{\nu E}{(1+\nu)(1-2\nu)}\left(\varepsilon_1+\varepsilon_2+\varepsilon_3\right)$$

where $\sigma_1$ = the axial principal stress, $\sigma_2$ and $\sigma_3$ = the transverse principal stresses, $\varepsilon_1$ and $\varepsilon_2$ (= $\varepsilon_3$) = the measured strains, E = 220 GPa = the Young's modulus [6], and $\nu$ = 0.27 = the Poisson's ratio [6]. The calculated stresses during loading are shown in Fig. 2 in the axial and transverse directions, on the basis of the assumption that the strains measured by using the {120}/{210} overlapping rings can be considered the average strains in the mullite.

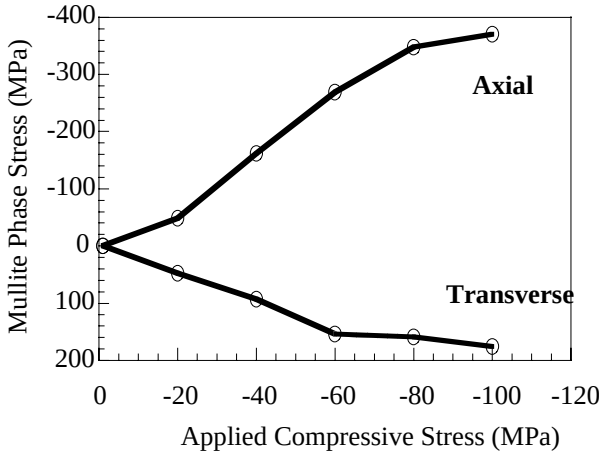


*FIG. 2. Mullite phase stress vs. applied stress during loading.*

It is apparent in Fig. 2 that the stresses behave in a roughly linear fashion up to about -60 MPa but deflect at -80 and -100 MPa to values of lower magnitude than would be expected from extrapolation of the linear region. This behavior is likely indicative of cracking in the sphere walls at the higher applied stresses, which decreases the average stress borne by the spheres. Furthermore, Fig. 2 shows that the mullite stresses are considerably higher than the applied stress. Axial stresses in the mullite grains

reach -370 MPa, with transverse stresses of 180 MPa, at an applied stress of -100 MPa. Loading of the sphere walls is therefore considerable and likely results from both load transfer from the more compliant matrix and direct loading through sphere-to-sphere contact points.

## Acknowledgments

DKB acknowledges the U.S. Department of Defense for support in the form of an NDSEG Fellowship. We thank Mr. P. Seshold of Envirospheres PTY Ltd. for providing microspheres. Diffraction experiments were performed at the DND-CAT Synchrotron Research Center located at Sector 5 of the APS, and the help of the DND-CAT staff is greatly appreciated. DND-CAT is supported by E.I. DuPont de Nemours & Co., The Dow Chemical Company, the U.S. National Science Foundation through Grant No. DMR-9304725, and the State of Illinois through the U.S. Department of Commerce and Board of Higher Education Grant No. IBHE HECA NWU 96. Use of the APS was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. W-31-102-ENG-38.

This report was extracted from the following publication: D.K. Balch and D.C. Dunand, "Mechanical Properties and In-situ Diffraction Strain Measurements in Aluminum-Mullite Microsphere Syntactic Foams Produced by Liquid Metal Infiltration," in *Processing and Properties of Lightweight Cellular Metals and Structures*, Third Global Symposium on Materials Processing and Manufacturing (TMS, Warrendale, PA, 2002), pp. 251–260.

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