Metallic Aluminum Aerogels from Magnesiothermal Reduction of Alumina Aerogels

Moriah C. Buckwalter, Ryan T. Nelson, Lauren E. Randaccio, Justin S. Griffin, and Stephen A. Steiner III

Aerogel Technologies, 1 Westinghouse Plaza, Boston, Massachusetts 02136, USA
mbuckwalter@aerogeltechnologies.com

Aerogels comprising reduced metal species offer unique technological potential, as they combine high surface area, high particle curvature, low density, electrical conductivity, and catalytic and chemical reactivity behaviors of metals in a single material envelope. Metallic aerogels show promise for applications including high-power-density air battery and supercapacitor electrodes, high-surface-area fuel-metal scaffolds for energetic materials, and precursors for production of other non-oxide ultraporous scaffolds. Leventis et al. synthesized metal aerogels of iron, nickel, and copper by carbothermal reduction of oxide aerogels, however for elements that do not have a carbothermally reducible oxide, a more powerful reducing agent such as magnesium can be used. Magnesiothermal reduction of nanostructured silicas into isomorphic silicon nanostructures has been previously reported in the literature by Bao et al. Aluminum represents a more challenging target, however, as the melting point of aluminum is very close to the melting point of magnesium thereby complicating magnesiothermal reduction. Additionally, selective etching of MgO from the resulting nanostructured aluminum material is extraordinarily vexing.

We synthesized aluminum aerogels via a carefully optimized magnesiothermal reduction of alumina aerogel precursors synthesized via epoxide-assisted gelation using $AlCl_3 \cdot 6H_2O$ and epichlorohydrin in a water/ethanol mix, supercritically dried from CO_2. The alumina aerogels were then magnesiothermally reduced at temperatures near the melting point of Al in a stainless-steel tube-furnace flow reactor. The resulting aluminum aerogels exhibit surface-readable Al^0 chemistry by XPS and serve as molecular hydrogen generators on contact with water and acids. Parameters underlying the magnesiothermal reduction process including magnesium-to-alumina ratio, reaction temperature, and time were explored. Methods for removing residual magnesium and magnesium oxide from the resulting aluminum aerogel including the dissolution of the magnesium and MgO in acid will be discussed. The properties and morphology of the resulting aluminum aerogels as characterized by nitrogen sorptimetry, XPS, XRD, and SEM will be discussed.