doi:10.1017/S1431927614008988
Microsc. Microanal. 20 (Suppl 3), 2014

Probing the Structure and Mechanical Properties of Individual $MgAl_2O_4$ Porous Agglomerates and Their Effects on Densification

Jorgen F. Rufner[1,2], Troy B. Holland[3], Ricardo H.R. Castro[1,2], and Klaus van Benthem[1]

[1] Dept. of Chemical Engineering and Materials Science, University of California, Davis, CA, 95616
[2] Neat ORU, University of California, Davis, CA 95616
[3] Dept. of Mechanical Engineering, Colorado State University, Fort Collins, CO 80523

In powder processing agglomeration can be a severe problem limiting achievable final densities and causing irregular microstructures due to residual porosity. Agglomeration can spur from many process aspects from powder synthesis and overly high calcination temperatures to poor green body pressing. Commonly, high uniaxial or isostatic pressure is used to mechanically breakdown agglomerates in an attempt to alleviate the issues mentioned above. Unfortunately, agglomerate breakdown strength is often difficult to determine making it challenging to improve or even determine processing parameters. In this study, $MgAl_2O_4$ nanopowder was synthesized via the Pechini/polymeric precursor method [1] with an average crystallite size of 20nm. The powder was isothermally sintered at 1300 °C for 1, 5, 10, and 20 minutes. The maximum achievable density for the samples was very low, only 55%. SEM imaging of the sintered pellets revealed a microstructure with large amounts of pores, ranging in size from tens of nanometers to microns in diameter. Hydraulic uniaxial compression to nearly 1GPa was applied in an effort to mechanically remove the porosity in the green pellet by deforming/breaking the agglomerates but was unsuccessful. The forces on the pellet surpassed even that of the die causing failure of the die before the agglomerates [4].

For that reason, individual agglomerates were investigated by cross-sectional imaging in a Phillips XL30 SEM (cf. Figure 1), revealing a pore network similar to that seen in the sintered samples with a percent volume porosity of 21%. As agglomerates were unable to be broken macroscopically, for the first time, individual agglomerate mechanical strength and deformation behavior was investigated using in-situ nanoindentation in a JEOL JEM2500 SE/TEM with a flat 1 micron canonical diamond indenter mounted on a Hysitron PI-95 Picoindenter, shown in Figure 2. The benefit of this technique is single agglomerates can be investigated and mechanical properties directly measured as well as high nanoindenter tip pressures become easier to attain due to the tips reduced area.

Through the nanoindentation quantitative load and displacement data was acquired in real time. That allowed for the calculation of elastic moduli and fracture strength values along with the analysis of deformation mechanisms. Across multiple experiments, load and displacement data was converted to stress and strain with the average agglomerate elastic moduli being 4.3 ± 1.1GPa. Stress-strain plots revealed three distinct deformation regions analogous to that of cellular solids described by Ashby and Gibson [2, 3]. The stages observed are correlated to elastic deformation of the material "beams" within the porous agglomerate, pore side wall collapse, and finally further compressive deformation until eventual agglomerate fracture. Considering these agglomerates are not sintered they are surprisingly stiff and strong with fracture strength of over 2GPa, offering potential for use as ceramic foams with large surface areas and high mechanical strength [5, 6]. Without techniques like in situ nanoindentation, quantitative properties from individual agglomerates could not be tied to the macroscopic properties of a powder as in this study.

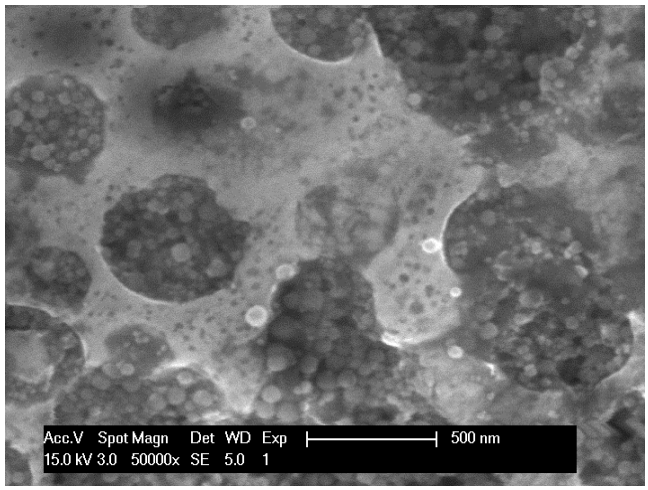

Figure 1: SEM image an of $MgAl_2O_4$ agglomerate cross-sections showing the pore structure on two length scales, numerous small nanometer sized pores amongst larger micron to sub-micron sized pores with.

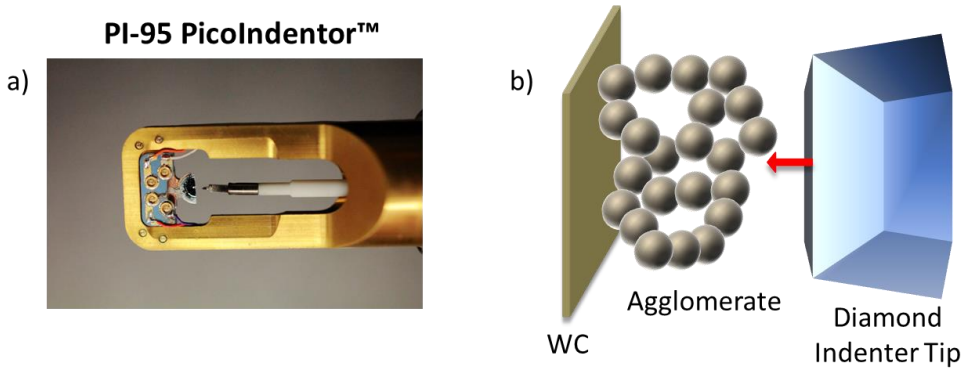

Figure 2: (a) Photo of the Hysitron PI-95 PicoIndentor TEM holder with sample on the left side and white indentation rod extended from the right side of the holder. The indentation setup is schematically drawn in (b) (*figure reproduced from [5]*).

References

[1] M. P. Pechini, "Method of Preparing lead and alkaline earth titanates and biobates and coating method using the same to form a capacitor.", U. S. Pattent, no. 3,330,697, 1967
[2] M.F. Ashby and R.F.M. Medalist, Metallurgical Transactions A, **14** (1983), p. 1755-1769
[3] L.J. Gibson and M.F. Ashby in "Cellular Solids: Structure and Properties", 2001, (Cambridge University Press, Cambridge
[4] J.F. Rufner et al, Journal of the American Ceramic Society, **96** (2013), p. 2077-2085
[5] J.F. Rufner et al, Acta Materialia, *(accepted for publication Jan. 25, 2014),* DOI: 10.1016/j.actamat.2014.01.051
[6] Funding is acknowledged from UC Davis start-up funds, UC Laboratory Fee grant #12-LR-238313, US Department of Energy, Office of Basic Energy Sciences, Los Alamos National Laboratory Material Design Institute, and Office of Naval Research #N00014-11-10788