The Effect of Texturing by Applied Magnetic Field on Grain Growth of Alumina

Introduction: Grain growth is a critical process to both metals and ceramics processing, as grain size plays a major role in bulk material properties, such as fracture toughness. Abnormal grain growth (AGG) is a process by which the growth of a small fraction of grains is incentivized and they grow faster than their neighbors, resulting in a bimodal grain size distribution and heterogeneous bulk properties. Though work has been conducted in this field for decades[1], the cause and mechanism behind AGG are still poorly understood.

The process is particularly import to ceramic materials, as the superior thermal resistance of ceramics lends itself to extreme environment applications. At these elevated temperatures kinetics are accelerated, expediting grain growth and AGG – this is a particular challenge in alumina, which is very susceptible to AGG[2]. Processing of these materials also raises concern, as high sintering temperatures can have the same deleterious effect. As such, it is vital to understand how to control grain growth in ceramics processing and applications.

Textured microstructures with enhanced mechanical properties in materials can be designed by controlling their crystallographic orientation during processing. One technique that has been explored is the application of a magnetic field during processing[3,4]. **This project will investigate how texturing by applied magnetic field in alumina ceramics impacts grain growth** through the use of electron and synchrotron X-ray based techniques, which allow us to track individual grains and grain boundaries. **I hypothesize that a strong applied magnetic field during processing will reduce grain growth and mitigate AGG due the formation of low-angle and, thus, low-energy grain boundaries that have a low driving force to move.** This will be validated by 3D microstructural characterization, allowing the measurement of grain boundary orientation and character for textured samples. These results will be relevant to industrial applications of alumina – including the manufacture of automotive parts and ballistic armor – as microstructure engineering can improve the mechanical properties of ceramic materials and improve stability and lifetime.

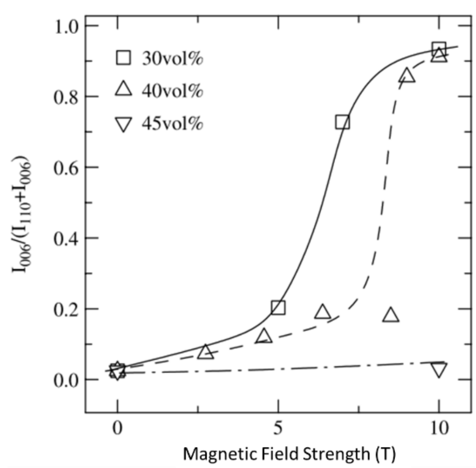

Figure 1. Preferential crystallographic orientation (ratio of 006 signal intensity to total signal intensity) as a function of applied magnetic field strength in alumina with different slip solid loading[4].

Objective 1: Preparation of textured Al_2O_3 samples by thermomagnetic slip-casting process: Alumina samples for this experiment will be prepared via slip casting with high-purity α-Al_2O_3 powder. **Alumina is chosen as a test material due to its impressive mechanical properties and applicability as a structural ceramic, as well as its susceptibility to texturing by magnetic field.** A dispersant will be added to the slip to prevent the agglomeration of particles. The samples will be subjected to an applied magnetic field during casting – this texturing technique has been shown to induce the growth of preferentially oriented grains during annealing[3,4]. Figure 1 illustrates this effect in alumina. Samples will be cast under applied magnetic fields of 0-8 T with 0 T being a control sample. Once cast, alumina green bodies will be sintered to near theoretical density and annealed at temperatures above 1400 ºC for various times.

Objective 2: Electron microscopy characterization of grain growth: From the bulk samples, centimeter-sized sections will be cut and polished for observation under a scanning electron microscope (SEM) to determine the initial average grain size by image analysis software employing the linear intercept method[5]. The samples will then be further annealed under identical time and temperature conditions, and the same method will be repeated to determine the grain growth in each sample. **These results will provide a quantitative measure of grain growth as a function of applied magnetic field during processing and annealing time.**

Objective 3: 3D characterization of crystallinity and grain size distribution: The novelty of this work lies in the use of high-energy X-ray diffraction microscopy (HEDM) to characterize the samples. In this technique, a sample is placed in the path of an incident X-ray beam and rotated while diffraction patterns are collected. In post-processing, these can be indexed to generate a crystallographic map of the measured volume. From the bulk, millimeter-sized samples will be prepared. From 3D crystallographic maps measured by HEDM, true grain sizes (at a resolution of 1 µm) can be determined and a grain size distribution created. The non-destructive nature of this technique is extremely advantageous, as it will allow tracking of individual grains and boundaries across heat treatments. Thus, the slower movement of individual textured low-angle grain boundaries can be observed and quantified. **Via 3D characterization of individual grains and boundaries, these results will verify a) the character and motion of the boundaries as a function of applied magnetic field, and b) whether observed low-angle grain boundaries induced by texturing reduce AGG.**

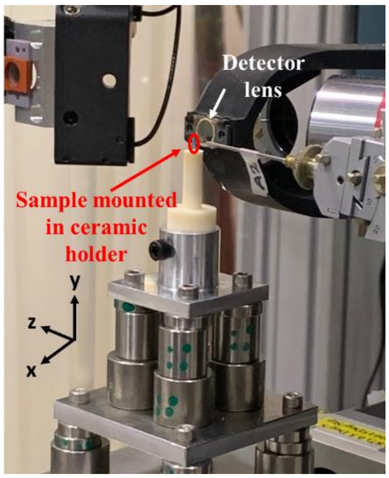

Figure 2. Set-up for HEDM synchrotron measurements, beamline 1-ID at Advanced Photon Source, Argonne National Lab. Incident X-ray beam travels along positive z-direction.

Research Plan: The timeline for this project is one year. Slip casting will be done at Oak Ridge National Lab, which houses a commercially available thermomagnetic system offering up to 8 T magnetic field. The timeline for this step is one week, accounting for travel time, as slip casting is a well-known process that can be modified as needed. SEM will be conducted at the University of Florida, whose Research Service Centers house a TESCAN SEM. The polishing, sample preparation, and data analysis will take 9-10 months. Lastly, HEDM experiments will be run at the NSF-sponsored CHESS synchrotron's Structural Materials beamline, at which HEDM will be available beginning in December 2019. The experimental design considers that each HEDM measurement takes hours, and beamtime slots are limited. As such, high-priority samples exhibiting strongly textured microstructure will be selected for synchrotron measurement.

Broader Impacts: Beyond structural materials, microstructure engineering is essential to functional materials like oxide fuel cells and laser materials. The results of this project will offer quantitative insight into the use of an applied magnetic field to texture alumina ceramics – this fundamental study will serve as a bridge for future industry-specific studies. As such, I would enjoy sharing these results at conferences and with industrial collaborators directly.

1. *Journal of the American Ceramic Society*, *80*(5), 1149–1156. **2.** *Scientific Reports*, *6*(37946), 2–11. **3.** *Scripta Materialia*, *54*(6), 977–981. **4.** *Science and Technology of Advanced Materials*, *7*(4), 356–364. **5.** *Journal of the American Ceramic Society*, *91*(7), 2304–2313.