IN-SITU ELECTRON MICROSCOPY STUDIES OF ELECTRIC FIELD ASSISTED SINTERING OF OXIDE CERAMICS

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A wide range of studies shows a dramatic effect of applied electric fields or currents on the sintering behavior of oxide ceramic powders. However, the mechanisms accounting for the so-called flash sintering remain elusive despite the wide application potential. Using in-situ scanning and transmission electron microscopy, we aim to gain insight into the atomic origins of sintering behavior, as well as of the high conductivity states that occur in conjunction with flash events during field-assisted sintering.

We investigate the sintering dynamics of ZnO green bodies with a density between 50% and 70% and ZnO thin films with and without electric fields and under different oxidizing and reducing gas pressures. Specifically, we use a specially designed SEM heating stage to study the evolution of microstructure and morphology, including grain/void morphology, segregation, and precipitation, both with and without applied fields and with and without gas pressures up to 2 mbar. The in-situ TEM sintering studies, also under controlled electric field and gas pressure, allow us to detect chemical segregation and valence changes (using EDX and EELS) near the sintering boundaries. By gaining access to structural and chemical information down to the atomic scale, we hope to determine how the electric field causes flash sintering.

Several conventional sintering experiments (no field, only heating) have been performed which confirm that sintering can be instigated and observed in the TEM. Furthermore, experiments have been performed with additional applied voltage / current to cause the flash sintering effect. Initial tests indicate that it is difficult to reach sufficiently high current densities to cause flash sintering in free standing green body specimens without causing phase changes due to Joule heating induced temperature increase. Therefore, we have started to grow thin layers of ZnO on Al_2O_3 and YSZ substrates which are both good thermal conductors thereby reducing the Joule heating. Since both substrates are poor electrical conductors, the applied current passes mainly through the ZnO layer. Furthermore, the thin film geometry allows more uniform current densities in the ZnO and facilitates the alignment of grain boundaries for high resolution TEM investigation. Results of studies on the dynamics of grain boundaries during sintering with and without applied current (Figure 1) will be discussed.

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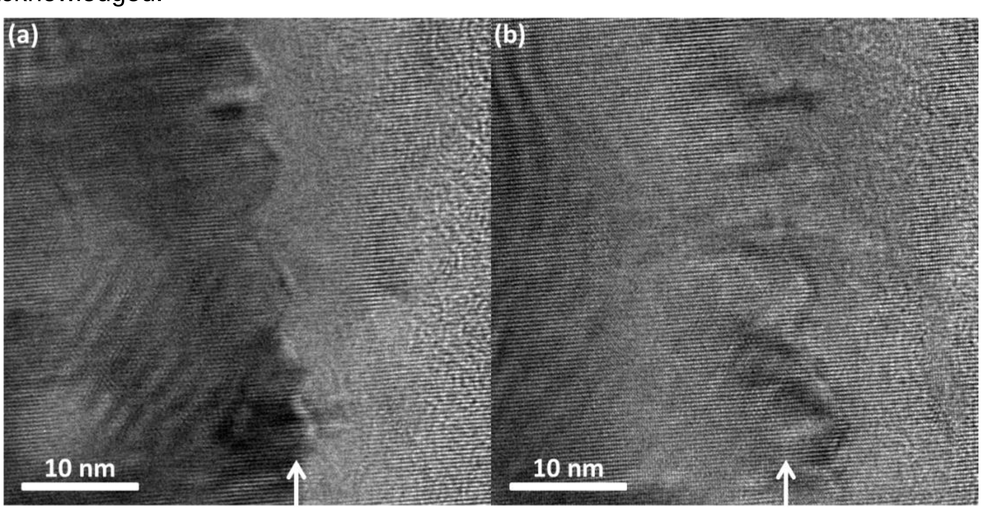

Figure 1 – Grain boundary migration in a ZnO film under an applied electric current and without additional heating. Images from an in-situ video show that the facetted grain boundary moves from its initial position (a) (marked by arrow) a distance of approximately 4 nm in 160 s (b).