

Public Abstract

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Title:COMBINED DILATOMETRY AND MASS SPECTROMETRY OF SINTERING AND EVOLVED GASES OF BARIUM TITANATE AND ZIRCONIUM DIBORIDE WITH SINTERING ADDITIVES

A combined dilatometer mass spectrometer (CDMS) has been shown to be useful in identifying gas phase species and sintering progress during the heating of barium titanate and ultra high temperature ceramics (UHTCs), including zirconium diboride and sintering additives, to high temperature. The development of the CDMS will allow for the observation of how starting powder characteristics affect gas phase reactions, which can then affect the sintering behavior along with the final microstructure and properties of the ceramic. The data provided by the CDMS can be combined with other analytical techniques to generate tailored sintering cycles that optimize desired properties while minimizing production costs.

Carbon dioxide and species related to its cracking in the MS were observed during the heating of barium titanate at low and intermediate temperatures, up to about 800-900 degrees C, and were assigned to the decomposition of organic species and the decomposition of residual barium carbonate from the synthesis of barium titanate. Immediately following the carbon dioxide signals, at temperatures of 850-1200 degrees C, signals corresponding to the evolution of sulfur dioxide were observed.

The UHTCs and common sintering aids heated in the CDMS individually and in mixtures include zirconium diboride, boron carbide, silicon carbide, boron oxide, zirconium oxide, zirconium disilicide, boron nitride spray, and organic binder, dispersant, and surfactant. Low temperature signals below 660 degrees C for these UHTCs are attributed to the decomposition of organics present in the boron nitride spray, binder, dispersant, and surfactant. The high temperature signals above 900 degrees C were determined to be carbon monoxide and carbon dioxide using reference cracking patterns and natural isotope abundances. The carbon sources are identified as arising from unreacted carbon from the synthesis methods for some of the powders and from the carbon inherent in the chemical formula. The oxygen sources were attributed to synthesis reactions for the powders and to the presence of surface oxides. An additional source for carbon monoxide and carbon dioxide production at high temperature for the powders in mixture is the reactive sintering that occurs.

A major result of this work, which has been observed from several diverse ceramic powder systems, has been the detection of many reactions in real time that result in gas phase species occurring at different temperatures throughout the heating cycles. The identification of gas-phase species arising from high temperature reactions and from powder impurities showcases the ability of the CDMS as a routine characterization tool for studying the gas-phase species evolved from ceramic materials at high temperature during sintering cycles.