DEVELOPMENT OF A COMBINED DILATOMETER AND MASS SPECTROMETER SYSTEM FOR STUDYING GAS PHASE CHEMISTRY AND KINETICS DURING SINTERING

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ABSTRACT

Details on the components and materials comprising the combined dilatometry mass spectrometry (CDMS) apparatus are provided; including recent modifications, operating procedures and information on major system failures. The CDMS is then used to study the gas phase reactions that occur during the sintering of strontium titanate. Two families of peaks are identified based on isotope ratios, cracking patterns, and comparisons to the decomposition of model compounds, as carbon dioxide and sulfur dioxide. Quantitative analysis was performed by comparing the primary peak size from the strontium titanate sample to the decomposition of the model compounds, showing that these two gases can account for between 60 to 200% of the observed weight loss. This range is because it is unclear whether the carbon and sulfur reacted with oxygen occurring in the crystal matrix, or with trace oxygen in the atmosphere.

The model-free technique for determining the apparent activation energy of sintering proposed by Su and Johnson is modified using an approximation developed by Lee and Beck. The analytic equations are compared to numeric integration to find the activation energy of sintering for simulated data. All equations are proven to be highly accurate, two of them more so than numeric integration when noise is present in the data. Additionally, if an equation for the mechanism is known, the approximation can be substituted for the integral so that the full equation can be mathematically manipulated to facilitate analysis of the assumed mechanism.