Application Note TA6009

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TA6009 - TG-MS analysis for debinding process of ceramics

Introduction

The detection or analysis of the gases evolved during a chemical reaction, as a function of temperature, constitute the techniques of thermal analysis called evolved gas detection (EGD) and evolved gas analysis (EGA), respectively.

Thermal analysis using mass spectrometry covers a large number of related and analytical powerful techniques such as evolved gas analysis using mass spectrometry (EGA-MA) including thermogravimetry-mass spectrometry (TG-MS), temperature programmed pyrolysis-mass spectrometry and temperature programmed desorption mass spectrometry.

In conventional EGA-MS, the evolved gaseous products, which are introduced rapidly to MS, are generally ionized by electron ionization (EI) at 70 eV. In this case, a part of the evolved gaseous molecular ion undergoes further decomposition, and observed simultaneously ions. Especially in the thermal processes, since the evolved gases consists of multiple gaseous species in almost all cases, the resulting fragment ions are overlapped, while the fragment ions provide significant information concerning the structure of the molecule, the apparent mass spectra can be quite complicated. In order to differentiate in real-time the multiple organic species that are evolved in the thermal process, one feasible approach is the use of MS with a selective and soft (fragment-free) ionization technique which avoids fragmentation during ionization.

Required materials for debinding are cemented carbide, powder metallurgy, rare-earth magnet, enhanced definition plasma display panel and electronic/structural ceramics. It is important to know the kinds of decomposition evolved gas and its generated behavior when debinding, to obtain good quality material and to establish the efficient debinding process. Performing the simulation analysis of the debinding process from ceramic molding sample employed the skimmer-type differential thermogravimetry-photoionization mass spectrometer (TG-DTA-PIMS), it is useful to control the atmosphere and heating in the actual process.

Instrument: ThermoMass Photo

ThermoMass Photo is an evolved gas analytical system designed for real-time simultaneous measurements of thermogravimetry – differential thermal analysis (TG-DTA) coupled with electron impact ionization (EI) and the fragment-free photoionization (PI) mass spectrometry (MS) that performs measurements as a function of temperature or time.

Measurement and analysis

A comparison between the results from EIMS and PIMS for commercially-supplied alumina ceramics sheet of the debinding process obtained evolved gas in inert atmosphere by TG-EI/PIMS are shown in Figure 1.

The thermal change with debinding is observed as simple mass loss during 300℃ to 500℃. Compared mass spectrum to EI from PI of evolved decomposition component with debinding, the difference of mass spectrum is clear. The conventional EIMS is difficult to analyze evolved gas component qualitatively due to complicate MS generated by the fragmentation ions. On the other hand, PIMS consist only molecular ions due to soft ionization, evolved gas species are m/z 56 (butene) and m/z 100 (methyl methacrylate). Therefore, TG-PIMS is useful method to analyze the debinding process for ceramics.

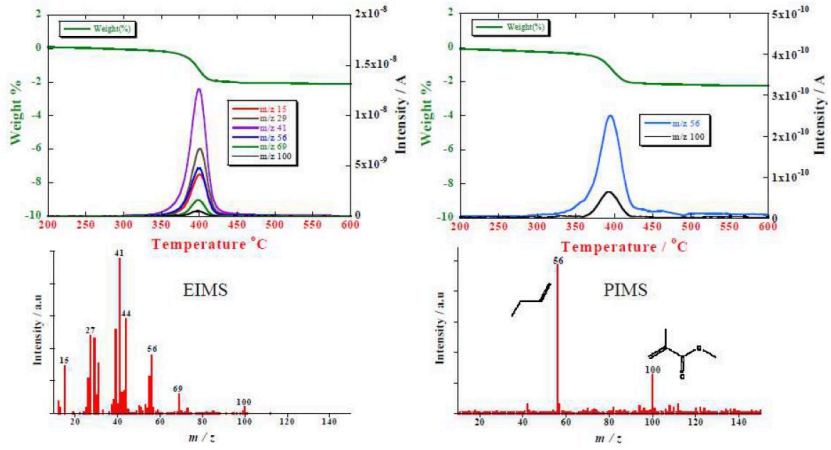

Figure 1: Comparison of TG-DTA-EI/PIMS curves and mass spectra of the debinding process of alumina sheet (Left: TG-EIMA and EI mass spectrum, Right: TG-PIMS and PI mass spectrum)

Related products

ThermoMass Photo

An integrated thermal analysis instrument capable of high-precision mass analysis of evolved gases without breaking the molecules, allowing direct measurement.