

Applications of Ion Attachment Mass Spectrometry (IAMS) for Monitoring of Firing Process.

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Introduction

Analysis of evolved gas from organic additives in ceramics material processing has been generally done with TG-MS and GC-MS. However the fragmentation due to the ionization for MS obstructs to identify the gaseous species in a furnace. Furthermore capillary which is a separation device of GC and an interface between TG and MS has a problem to generate some species in addition to pyrolyzed species. Therefore a combination of skimmer interface with no change of evolved gaseous species and IAMS (Ion Attachment Mass Spectrometry) with no fragmentation during the ionization has been expected the accurate characterization of gaseous species in the furnace. The IAMS itself was successfully applied to individual pyrolysis monitoring using evolved gases from the mixture of PVA as binder and PMMA as porogen in a starting material of porous ceramic¹⁾.

Methods

A prototype instrument of IAMS and infrared furnace connected with skimmer interface based on the principle of a jet separator has been build up under cooperation of ANELVA Technix Corporation and RIGAKU Corporation²⁾. The measurements were carried out with Helium flow as carrier gas and its heating rate was 20 deg·min⁻¹.

Samples were alumina powder with binder which is polyvinyl alcohol (PVA), methyl cellulose (MC), or their mixture.

Results and Discussion

In the spectrum of IAMS, all peaks are with 7 amu higher than its molecular weight because Li ion with 7 amu is attached due to the ionization principle. For example, mass peak with 25 amu means H₂O of which molecular weight is 18 amu. From the sample containing PVA as binder, acetaldehyde and crotonaldehyde which are detected also with GC-MS, were obtained in the mass spectrum at about 270 °C during pyrolysis of the PVA as binder. Other characteristic species with 103 and 130 amu were detected, although their identification are not clear. From the sample containing MC as binder, also characteristic species with 67, 81, 93, 103, 111, 112, 124, 134 and 148 amu were detected during its pyrolysis. On the other hand,

spectrum synthesized each other of PVA and MC was obtained from the sample containing the mixture binder of PVA and MC. According to the appropriate indicator of pyrolysis which is an evolved gas, individual monitoring of the mixed binder has been successfully carried out³⁾. It may mean no interaction exists during the pyrolysis of the mixture binder of PVA and MC.

Reference

- 1) T. Tsugoshi et.al., *Anal. Chem.*, **78**, 2366-2369 (2006).
- 2) T. Tsugoshi et.al., *J Therm Anal. Cal.*, **80**, 787-789 (2005).
- 3) T. Tsugoshi et.al., *Talanta*, **70**, 186-189 (2006).

Biographical Sketch

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Dr. Takahisa TSUGOSHI received his BA degree in 1988 and received a PhD degree in analytical chemistry at Tokyo University of Science. He had studied and worked in Tokyo University of Science for 12 years. He was a senior research scientist of Advanced Sintering Technology Group, Advanced Manufacturing Research Institute, AIST, and is currently a senior research scientist of the Reference Materials System Division, National Metrology Institute of Japan, National Institute of Advanced Industrial Science and Technology (AIST). He is also a chair person of Young Analytical Chemist Association, the Japan Society for Analytical Chemistry (JSAC). Of course he is a chairperson of the organizing committee of the AYCeCT.