

ESTABLISHMENT OF A COMPREHENSIVE ANALYTICAL SYSTEM FOR STUDY OF TERRESTRIAL AND EXTRATERRESTRIAL MATERIALS AND ITS APPLICATIONS

KITAGAWA, Hiroshi / YAMANAKA, Masahiro / SAKAGUCHI, Chie
/ OTA, Tsutomu / KUNIHIRO, Tak / NAKAMURA, Eizo /
MAKISHIMA, Akio / MORIGUTI, Takuya / KOBAYASHI, Katsura /
TANAKA, Ryoji

(出版者 / Publisher)

法政大学イオンビーム工学研究所

(雑誌名 / Journal or Publication Title)

PROCEEDINGS OF THE 36th SYMPOSIUM ON MATERIALS SCIENCE AND ENGINEERING
RESEARCH CENTER OF ION BEAM TECHNOLOGY HOSEI UNIVERSITY (December 13,
2017)

(巻 / Volume)

36

(開始ページ / Start Page)

1

(終了ページ / End Page)

6

(発行年 / Year)

2018-02

(URL)

<https://doi.org/10.15002/00030353>

ESTABLISHMENT OF A COMPREHENSIVE ANALYTICAL SYSTEM FOR STUDY OF TERRESTRIAL AND EXTRATERRESTRIAL MATERIALS AND ITS APPLICATIONS

Eizo NAKAMURA, Akio MAKISHIMA, Takuya MORIGUTI, Katsura KOBAYASHI,
Ryoji TANAKA, Tak KUNIHIRO, Hiroshi KITAGAWA, Tsutomu OTA,
Chie SAKAGUCHI and Masahiro YAMANAKA

*The Pheasant Memorial Laboratory for Geochemistry and Cosmochemistry (PML),
Institute for Planetary Materials, Okayama University, Misasa, Tottori 682-0193,
Japan*

Abstract

We have devoted the past 25 years to development of the Comprehensive Analytical System for Terrestrial and Extraterrestrial Materials (CASTEM), a unique, extremely well-equipped laboratory for materials science. CASTEM facilities allow for the determination of elemental and isotopic abundances of geochemically significant elements present in samples ranging widely in type and size. Although CASTEM research was initially aimed at investigation of rocks, we have broadened the range of materials investigated to include human tissue and biological materials present in a wide variety of geological settings with various scale ranging from sub-nanometer to 10^{15} m and ages from 4.56 billion years to the present.

I. Introduction

It is extremely important for researchers to continually question the purpose of terrestrial and extraterrestrial materials science. In general, it is to understand the compositions of, and related processes responsible for, “materials” in the solar system and within the Earth. In other words, its goal is to describe the distribution of elements (atoms) over sub-nanometer to 10^{15} m dimensions ranging to the size of the solar system, which developed over 4.6 billion years, and to reveal the processes of material evolution. Somewhat differently stated, it can be said that the most meaningful contribution of terrestrial and extraterrestrial materials science is to extract information regarding physicochemical evolution over this huge range in space and time. This thinking leads us to analyze the broadest range of materials possible, developing new analytical methods

where necessary. This field is not limited to investigation of extraterrestrial materials, such as meteorites, or samples of the type returned by the Hayabusa Spacecraft, and so-called conventional terrestrial materials such as rocks, atmosphere and sea water, but also should involve study of biological materials such as those related to malignant mesothelioma or pulmonary¹⁾ carcinoma and those in rocks and representing geobiological processes. It is necessary to integrate diverse analytical methods and interpret the results comprehensively for the understanding of the origin and evolution of this huge variety of materials. To achieve our goals, we analyze the distribution of elements and isotopes in these materials and integrate this information with knowledge of absolute timing and rates based on age-dating methods utilizing the breakdown of radionuclides. Here we present an outline of CASTEM, is technical core of our research approach, presenting as an example the results of our analysis of particles returned by Hayabusa Spacecraft²⁾.

II. Comprehensive Analytical System for Terrestrial and Extraterrestrial Materials

The development of the Comprehensive Analytical System for Terrestrial and Extraterrestrial Materials (CASTEM) began when the first author arrived as an Assistant

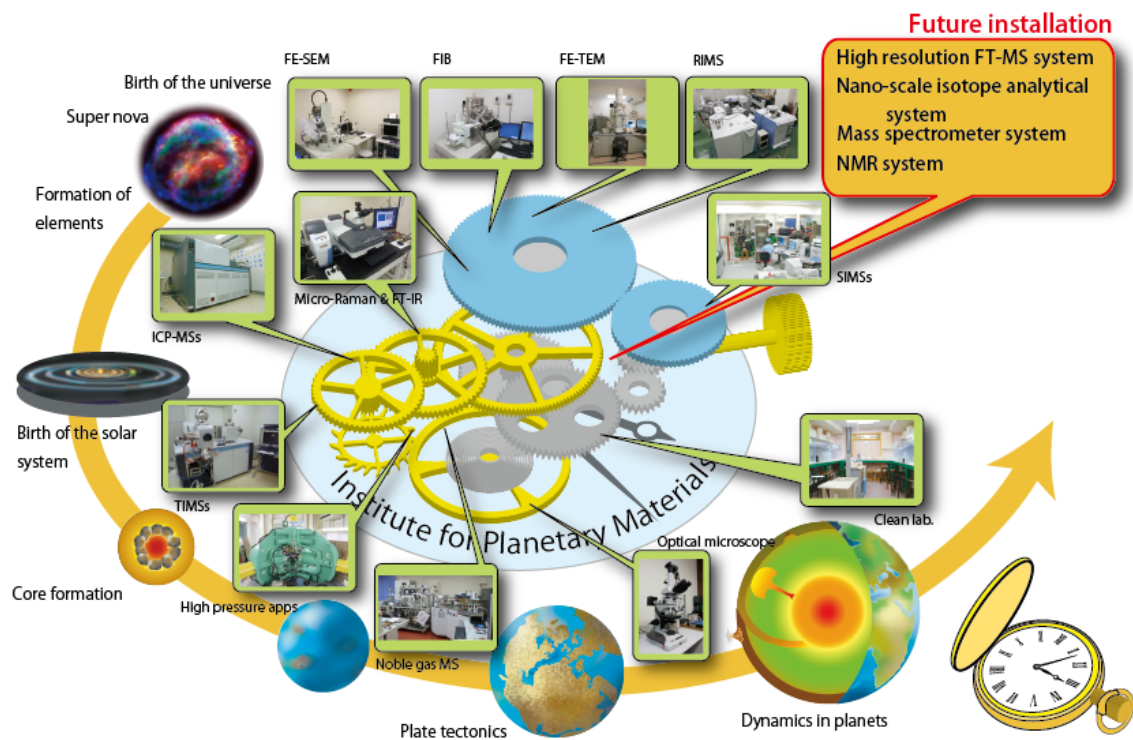


Figure 1 Conceptual image of the CASTEM. The facilities link each other and then form a single virtual system for the comprehensive analysis.

Professor at the Institute for Study of the Earth's Interior. The aim of CASTEM was, and is, to develop a laboratory environment enabling us to precisely determine the distribution of elements and quantitatively measure isotope compositions to allow us to break new ground in terrestrial and extraterrestrial materials science. The CASTEM has now been under development for a quarter of a century with an extensive and highly specialized clean lab as the hub of the system providing access to methods such as Thermal Ionization Mass Spectrometry (TIMS) and Inductively Coupled Plasma Mass Spectrometry (ICPMS), stable isotope ratio mass spectrometry not only O-H-C-S-N but also for noble gases, and allowing high spatial resolution work using Scanning Electron Microscopes (SEM), a Transmission Electron Microscope (TEM), an Electric Probe Micro Analyzer (EPMA), and two Secondary Ion Mass Spectrometers (SIMS; see **Fig. 1, 2**). The major aim of the clean lab at the center of CASTEM is what we call bulk chemistry analysis, that is, to determine the average chemical characteristics of samples crushed and homogenized. The original clean benches for conducting experiments, clean evaporators for sample heating and drying, and laboratory sample distillation system were designed to minimize blanks (contaminants) to the greatest extent possible. The reason for this great effort regarding reduction of blanks relates to the amount of sample needed for our analysis (commonly near 10 mg) and the commonly low concentrations of the elements to be measured ($\mu\text{g/g}$) – ppb (ng/g). For analyses of extremely small amounts of an element, blanks can easily catastrophically modify our results, rendering them uninterpretable (reference³) for a discussion of typical blanks resulting from handling of rock samples). By use of the clean lab facility, the TIMS and the ICPMS, and using analytical methods developed here, we can conduct quantitative analyses of 63 elements and 11 isotope systems important for terrestrial and extraterrestrial materials science⁴. Several methods that we developed have become the global standard, including those for isotope analyses of boron and lithium. Moreover, we have established radiometric dating by application of systems such as Rb-Sr, Sm-Nd, U-Th-Pb, Lu-Hf, and La-Ce dating. Also, we have developed and put to practical use microscale U-Pb, Mn-Cr and Fe-Ni dating involving extinct radionuclides and U-Th-Ra dating involving radioactive disequilibrium. Thus, we are able, to a greater extent than any other laboratory facility, conduct the dating suitable for the problem being considering, as related to the time scale being considered and the process being investigated.

The major feature of the instruments in CASTEM is that nearly all of them can be employed as one analytical instrument using a Visual Stage System (VSS) designed in our lab involving a two-dimensional coordinate, shared-stage control system based on an image database. Using this system, it is possible to share virtual analytical coordinates

(sample positions) among optical microscopes, the Field Emission-Scanning Electron Microscope (FE-SEM), the Secondary Ion Mass Spectrometry (SIMS), and a laser heating system. It is enabling acquisition of extremely diverse observations regarding the material structure compositions of major and minor elements, and isotope composition, all for exactly the in same location in the sample (with repositioning precision near 1 μm). A recently introduced Focused Ion Beam sample processor (FIB) is also linked to the VSS enabling us to prepare thin film samples based on initial observations by FE-SEM and examine samples using a TEM. This ability to conduct our work with high spatial resolution is critical for our studies of complex physical and chemical structure characteristic of terrestrial and extraterrestrial materials. Moreover, the standard materials used for quantitative and isotope analysis by Secondary Ion Mass Spectrometry (SIMS) are calibrated by use of bulk-sample clean lab methods described above. We can prepare standard materials matching the expected structure and compositions of our unknowns, for example to evaluate matrix effects, thus greatly increasing the reliability of the work conducted at high spatial resolution. Stable isotope and noble gas isotope systems have recently been added to CASTEM and high-precision, quantitative analyses of the isotopes of oxygen, hydrogen, carbon, and nitrogen have become routine enabling us to consider the behavior of light elements and noble gases on Earth and during the evolution of the

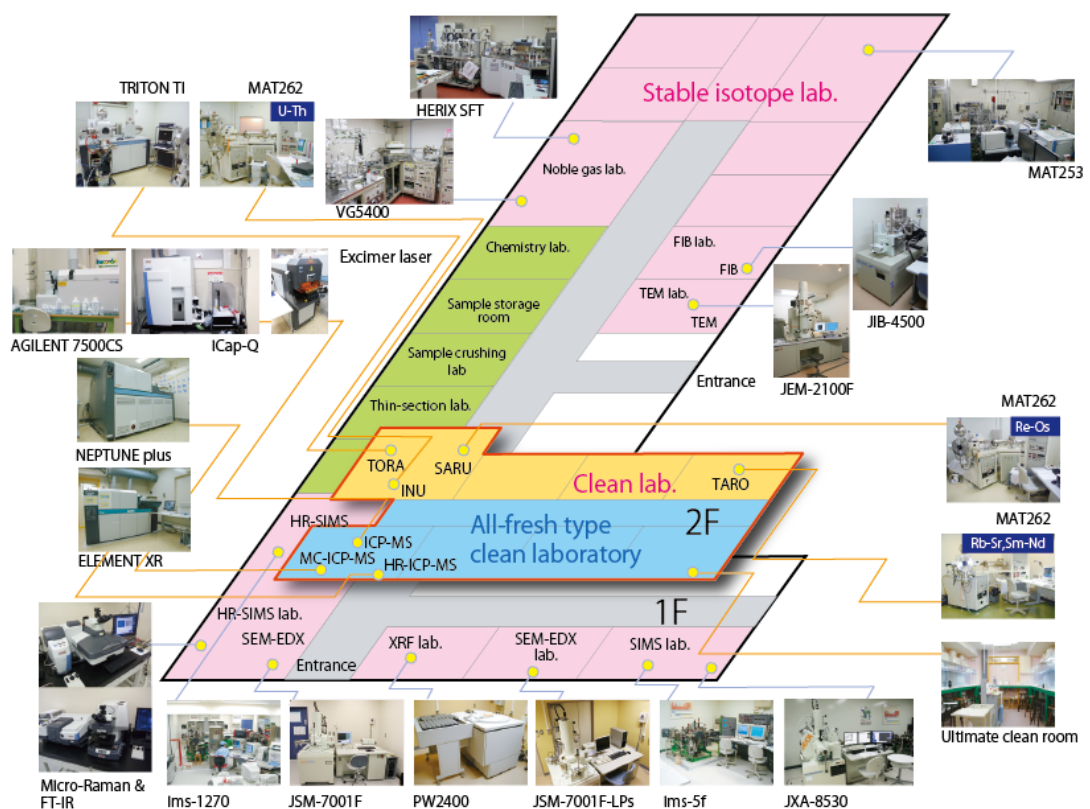


Figure 2 The facility layout of CASTEM 2017.

solar system. We expect these capabilities to extend our research to areas such as environmental and life science, allowing us to broaden our perspective of terrestrial and extraterrestrial materials science. We also have engaged in upgrading our ability to deduce absolute ages of volcanic eruption by K-Ar dating, using our noble gas isotope analytical system, and we are able to determine eruption ages of tens of thousands of years younger than what has previously been measurable. Such a capability allow us to quantitatively assess the dynamics of magmatic systems, from the generation of the magmas to their eruption, thus informing the public regarding volcanic eruption hazards.

III. Summary

To summarize, the CASTEM initially consisted of only one thermal ionization mass spectrometer (in 1987) and the present configuration and success of the lab were achieved through the motivation, originality, and ingenuity of researchers who have realized the great value and novelty of such an undertaking and involving well-sequenced introduction of key analytical instruments. As a result, it became possible to extract information sufficiently comprehensive for thorough investigation of terrestrial and extraterrestrial materials science.

Reference

- 1) E. Nakamura, A. Makishima, K. Hagino and K. Okabe: Accumulation of radium in ferruginous protein bodies formed in lung tissue: association of resulting radiation hotspots with malignant mesothelioma and other malignancy. *Proc. Japan Acad. Ser. B* **85**, 229, 2009.
- 2) E. Nakamura, A. Makishima, T. Moriguti, K. Kobayashi, R. Tanaka, T. Kunihiro, T. Tsujimori, C. Sakaguchi, H. Kitagawa, T. Ota, Y. Yachi, T. Yada, M. Abe, A. Fujimura, M. Ueno, T. Mukai, M. Yoshikawa and J. Kawaguchi: Space environment of an asteroid preserved on micrograins returned by the Hayabusa spacecraft. *Proc. Nat. Acad. Sci. USA*. **109**, E624, 2012.
- 3) E. Nakamura, A. Makishima, T. Moriguti, K. Kobayashi, C. Sakaguchi, T. Yokoyama, R. Tanaraka, T. Kuritani and H. Takei, Comprehensive geochemical analyses of small amounts (<100 mg) of extraterrestrial samples for the analytical competition related to the sample return mission MUSES-C, *Inst. Space Astronaut. Sci. Rep.*, **SP. 16**, 49, 2003.

- 4) A. Makishima and E. Nakamura, Determination of Major, Minor and Trace Elements in Silicate Samples by ICP-QMS and ICP-SFMS Applying Isotope Dilution-Internal Standardisation (ID-IS) and Multi-Stage Internal Standardisation, *Geostand. Newsl.* **30**, 245, 2006.