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Isotope analysis of micro metal particles by adopting laser-ablation mass spectrometry

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1. Introduction

The isotope analysis of microparticles in environmental samples as well as laboratory samples is an important task. A special concern is necessary in particle analysis of swipe samples. Micro particles are normally analyzed either by dissolving particles in the solvents and adopting conventional analytical methods or direct analysis method such as a laser-ablation ICP mass spectrometry (LA-ICP-MS), SIMS, and SNMS (sputtered neutral mass spectrometry). But the LA-ICP-MS uses large amount of samples because normally laser beam is tightly focused on the target particle for the complete ablation. The SIMS and SNMS utilize ion beams for the generation of sample ions from the particle. But the number of ions generated by an ion beam is less than 5% of the total generated particles in SIMS. The SNMS is also an excellent analytical technique for particle analysis, however, ion beam and frequency tunable laser system are required for the analysis.

Recently a direct analysis of elements as well as isotopes by using laser-ablation is recognized one of the most efficient detection technology for particle samples. The laser-ablation mass spectrometry requires only one laser source without frequency tuneability with no sample pretreatment. Therefore this technique is one of the simplest analysis techniques for solid samples as well as particles. In this study as a part of the development of the new isotope analysis techniques for particles samples, a direct laser-ablation is adopted with mass spectrometry. Zinc and gadolinium were chosen as target samples, since these elements have isotopes with minor abundance (0.62% for Zn, and 0.2% for Gd). The preliminary result indicates that isotopes of these two elements are analyzed within 10% of natural abundance with good mass resolution by using direct laser-ablation mass spectrometry.

2. Methods and Results

In laser-ablation experiment, the characteristics of laser-induced plasma depend on the wavelength of the ablating laser. The 2nd harmonic (532 nm) and 3rd harmonic (355 nm) are popular choice of ablation wavelength. In this study the 2nd harmonic of Nd:YAG laser (532 nm) was used for the laser ablation of particle samples. Laser light was mildly focused at the target particle with the beam size of 1.5 mm x 5 mm by using a focusing lens (f=25 cm). Therefore the area of the focal point is ~ 0.55 cm^2. When the laser strikes particles with high intensity for ablation of samples, ~2960 V of high voltage repels the generated ions to the direction of ion reflector. Two additional electrodes, one with 610 V and the other with ground voltage, accelerated the ions the ion mirror. Four deflection electrodes were also installed for the adjustment of ion path in x- and y-directions. The reflected ions were detected by using a dual micro channel plate. The detailed experimental setup of laser-ablation mass spectrometry is shown in Fig. 1.

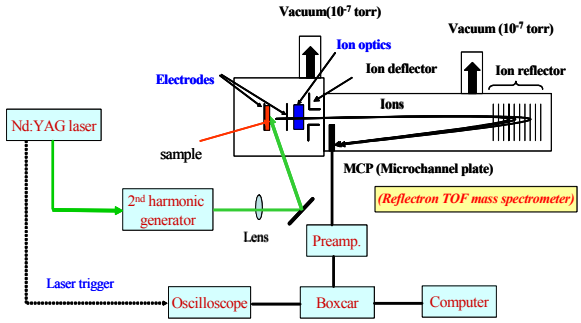

Fig. 1 A schematic diagram of experimental setup for laser-ablation mass spectrometry for particle analysis

Particle samples were prepared by using a Collodion solution to fix the particle on the metal plates. The adopted metal plates are Ta and Zr. The sizes of sample particles were 50~100 μm. Fig. 2 shows the prepared sample plate and the expanded view of loaded particles. The laser beam ablated the Collodion solution as well as particles, but the Collodion did not influence on the mass spectrum of the target particle.

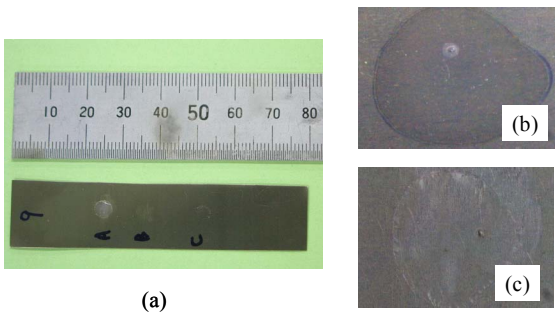

Fig. 2 A photograph of the loaded particle sample on the zirconium plate

As was indicated, the size of laser beam was 0.55 cm^2, while the size of the sample particle is about 100

μm. Therefore it is very important to make the laser beam uniform in spatial profile and to hit accurately the sample particle. Since our experimental setup did not have a microscope, a trial and error type effort was put in order to ablate target sample. Fig. 3 is the Laser ablation mass spectrum of Zn particle with laser power of 50 mW. Only ions of Na, K and Zn are identified. In Fig. 3-(b) the detailed mass spectrum of Zn particle is presented. Four isotopes are clearly identified and the calculated abundance is listed in Table 1.

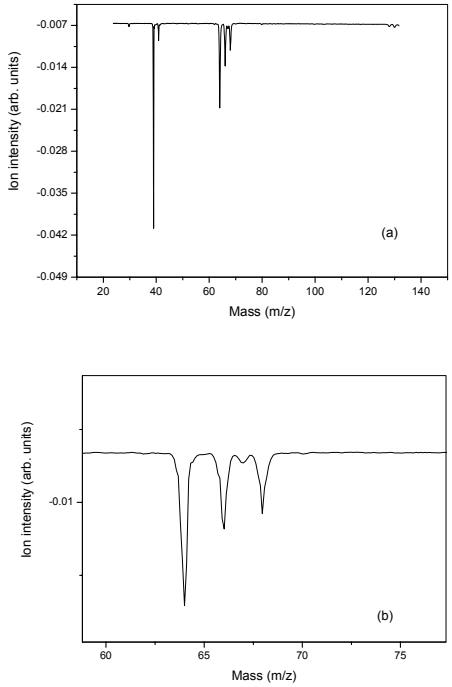

Fig. 3 A laser-ablation mass spectrum of zinc particle.

Table 1. Measured isotope ratio of Zn particle

	$^{64}Zn_{(\%)}$	$^{66}Zn_{(\%)}$	$^{67}Zn_{(\%)}$	$^{68}Zn_{(\%)}$	$^{70}Zn_{(\%)}$
Natural abundance	48.63	27.9	4.1	18.75	0.62
Data1	49.48	26.61	4.17	18.80	0.93
Data2	51.70	26.78	3.43	17.55	0.53
Data3	51.40	26.69	3.87	17.61	0.42
Averaged value	50.86	26.69	3.82	17.99	0.63

The mass spectrum of Gd particle (size : less than 10 μm) is also presented in Fig. 4. The Gd particles are so small that several particles are loaded with Collodion. The Gd has seven isotopes (m/z=152, 154, 155, 156, 157, 158, 160), and the figure shows all seven isotopes. The calculated abundance based on the measured mass spectra agrees within 10% of the natural abundance.

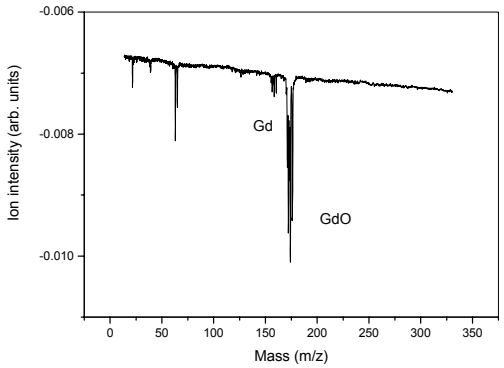

Fig. 4 A laser-ablation mass spectrum of Gd particle.

3. Conclusion

We have demonstrated the isotope analysis of micro particles by adopting laser-ablation mass spectrometry. The measured isotope abundance agrees within 10% of the natural abundance. This technique may be applied for the isotope analysis of swipe samples. Further improvement of the developed technique is in progress in order to have better accuracy and precision of the measured isotope ratio.

Acknowledgement

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