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Research progress on the application of laser ablation absorption spectroscopy

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ABSTRACT

Laser ablation absorption spectroscopy (LAAS) is an analytical technique by combining diode laser absorption spectroscopy (DLAS) with laser ablation (LA) technique. It has been developed to atomize various samples and distinguish elements and isotopes directly without chemical separation. This article reviews the principle and recent research highlights of LAAS technique.

Key words: Laser ablation; Plasma; Laser absorption spectroscopy; Isotope analysis

1. INTRODUCTION

Laser ablation (LA) is a process that uses high-energy laser pulses to excite plasma plumes on the surface of a sample [1] [2]. It has been widely used in many fields, including material analysis, laser cutting and soldering, and pulsed laser deposition [3-6]. Laser ablation technology has attracted more and more attention from researchers as a means of sampling and direct atomization due to its wide applicability and other attractive features. Various analytical techniques based on laser ablation have also rapidly developed, such as laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) [7], laser-induced breakdown spectroscopy (LIBS) [8], and laser-induced fluorescence (LIF) [9].

Diode laser absorption spectroscopy (DLAS) is a concentration measurement method based on the principle that the molecules or atoms selectively absorb the laser energy to obtain a high-resolution absorption spectrum and analyze the spectrum to obtain parameter information. Compared with other detection methods, DLAS technology has the following characteristics: strong environmental adaptability, high-sensitivity, high-selectivity, simple operation, and easy

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miniaturization. Presently, DLAS technology is mainly used in atmospheric environment monitoring, industrial process monitoring and elemental analysis [10-15].

Laser ablation absorption spectroscopy (LAAS) is a new technology that combines laser ablation (LA) and diode laser absorption spectroscopy (DLAS) in recent years. Accordingly, LAAS technology has the advantages of both technologies. The advantages of LAAS technology include no need for complex pre-processing of samples, small sample requirements, non-destructive detection, on-line measurement in long-range harsh environments, fast measurement speed, and high spectral resolution.

LAAS technology is currently mainly used for quantitative analysis of elements and measurement of isotope abundance, and gradually expands into more fields.

2. PRINCIPLE OF LAAS

Laser ablation absorption spectroscopy (LAAS) is a new analytical technique which combines laser ablation with laser absorption spectroscopy. As shown in Fig.1, the probe laser passes through the plasma plume stimulated by high-energy laser pulses parallel to the sample surface and reaches the photodetector. The information in the sample can be obtained by measuring the intensity of light passing through the plasma plume.

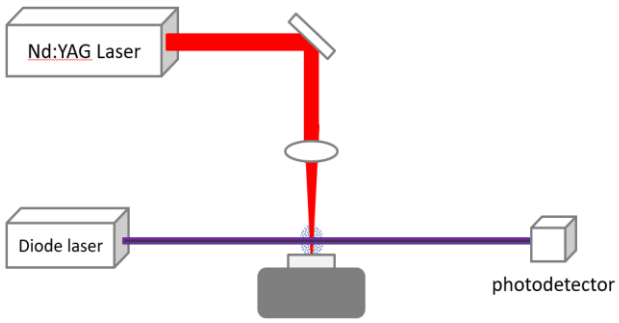

Figure 1

The basic principle of absorption spectroscopy for trace detection is the Beer-Lambert law. After the probe laser passes through the gas sample to be tested, the absorption of a specific component on a given absorption path is measured to obtain information of a specific component. The concentration of the gas is obtained by fitting the molecular absorption line obtained after the measurement with the existing spectral line parameters (or by calibration provided by a standard gas).

The measurement process of direct absorption spectroscopy is shown in Fig.2. After a beam of incident light with intensity $I_0(\nu)$ passes through an optical absorption cell with length L, if the laser passes through one way, the output intensity $I_t(\nu)$ is determined by Beer-Lambert law:

$$I_t(\nu) = I_0(\nu)e^{-\alpha(\nu)L} \quad (1)$$

Where ν is the frequency of laser, α(ν) is the absorption coefficient of sample gas, which is used to characterize the absorption of a certain gas to a certain wavelength of light.

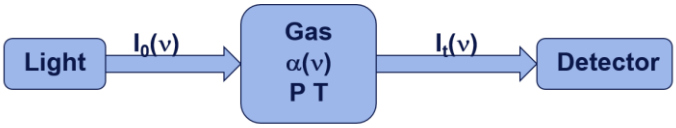

Figure 2

3. RESEARCH PROGRESS OF LAAS

The combination of laser ablation and atomic absorption spectroscopy has been around since the advent of laser ablation (in the early 1960s) [16] [17], but the performance of ordinary hollow cathode lamps or electrodeless discharge lamps as light sources at the time was not very good. The result of the joint use is not very satisfactory. With the development of laser technology, the emergence of diode laser as a light source with better performance provides a bright prospect for the combination of laser ablation and atomic absorption spectroscopy. Diode lasers have many advantages as a source of atomic absorption spectroscopy, such as narrow linewidth, adjustable wavelength, reasonable price, and good spatial resolution. Recently, laser ablation absorption spectroscopy has attracted more and more researchers' attention, and related application research reports have also increased.

3.1 Laser ablation single-beam diode laser absorption spectroscopy (fixed wavelength mode)

Laser ablation single-beam diode laser absorption spectroscopy is one of the first LAAS techniques to be applied. It lays a good foundation for the development of subsequent technologies and also provides a novel and effective way for isotope analysis.

In 2001, A. Quentmeier used laser ablation single-beam diode laser absorption spectroscopy to measure the uranium isotope ratio in solid samples [18]. In the experiments, a diode laser was used for the selective detection of ^{235}U and ^{238}U in laser ablation plasma ignited by a Nd:YAG laser beam focused onto uranium oxide samples. The diode laser was sequentially tuned to the absorption lines of both isotopes 682.6736 nm for ^{235}U, and 682.6913 nm for ^{238}U. Three samples were used with the relative abundance of the minor isotope ^{235}U of 0.204%, 0.407% and 0.714%. The limit of detection of the minor isotope was estimated to be 100 μg g^{-1}. This strategy was checked by determination of the isotope

ratios for the two samples with depleted ^{235}U concentration using the sample with the natural isotope composition 0.714% as a standard. The accuracy and precision for this measurement strategy was evaluated to approximately 10%.

3.2 Laser ablation single-beam diode laser absorption spectroscopy (scan mode)

This method is similar to the above method except that the wavelength scan mode is used. The laser output wavelength is tuned by changing the laser input current or temperature to scan complete absorption lines of the isotope to obtain a high-resolution absorption spectrum, and then analyzing the spectrum to obtain parameter information.

M. Miyabe investigated the temporal evolution of ablation plumes of neutral and singly charged uranium atoms produced by second-harmonic light from a Nd:YAG laser at a fluence of 0.5 J/cm^2. They measured absorption spectra using three samples in which ^{235}U abundances were 0.72 %, 4.85 %, and 9.53 % [19]. The absorption spectrum of uranium was measured by scanning the vicinity of the ^{235}U and ^{238}U isotope absorption line with a laboratory-made external cavity semiconductor laser. The limit of detection (LOD) of the isotope ratio of ^{235}U to be about 0.01 %, the precision of the measured isotope ratio is estimated to be less than 2.5 % relative standard deviation.

3.3 Laser ablation dual-beam diode laser absorption spectroscopy

Laser ablation dual-beam diode laser absorption spectroscopy is developed on the basis of single-beam technology (fixed wavelength mode). Two diode lasers are used as the measurement source. This method can eliminate the influence of signal fluctuation between laser ablation pulses on measurement reproducibility, thereby improving the precision and accuracy of the measurement.

In 2002, on the basis of A. Quentmeier's work, H. Liu proposed a new approach using two diode lasers as the light source to determine the $^{235}U/^{238}U$ isotope ratio by laser ablation dual beam diode laser absorption spectroscopy [20]. In the experiment, two diode lasers cross into the plasma at a small angle (about 4°), and the intersection passes through the plasma center axis. Assuming the plasma is symmetrically extended, the two laser beams will pass through the same path. The precision and accuracy of $^{235}U/^{238}U$ isotope ratio determination were obtained to be 5 % and 2%, respectively , the detection limit of the minor isotope was estimated to be 18 μg g^{-1}. The precision and accuracy of the double beam method are 2 and 4 times higher than that of the single beam method, respectively, and the detection limit is reduced by 5 times. The results show that the double-beam detection method is able to eliminate the influence of the fluctuation of laser ablation pulse signal on the measurement reproducibility, realize the simultaneous determination of primary and secondary isotope absorption signals, and improve the reproducibility of atomic absorption detection. It has greatly improved the precision and accuracy of the single-beam detection method reported earlier.

3.4 Femtosecond laser ablation absorption spectroscopy

With the development of femtosecond laser technology, high-energy, short-pulse femtosecond lasers have been commercialized, and femtosecond laser ablation has begun to be applied in various fields. The three techniques described

above are based on nanosecond laser ablation, femtosecond laser ablation has fundamental differences from nanosecond ablation due to the lack of laser-plasma interaction, and femtosecond ablation is a cold ablation process that reduces the body of the ablation pit product [21-25]. Also, the combination of femtosecond laser ablation and DLAS technology has gradually attracted the attention of researchers. The data for femtosecond ablation was acquired in a few tenths of a second with no averaging, whereas the nanosecond ablation required several minutes of acquisition due to the lower repetition rate of the laser and averaging over multiple pulses to reduce shot-to-shot noise. The high laser repetition rates in femtosecond laser ablation results in higher signal-to-noise ratios for a given total measurement time.

Mark C. Phillips presented the first measurements of time-resolved atomic absorption spectra of uranium and aluminum in femtosecond laser ablation plasmas[26]. In the experiment, strong atomic absorption signals in femtosecond ablation plumes are observed, and the lower electron density and kinetic temperatures in the femtosecond ablation plumes relative to typical nanosecond ablation plumes results in narrow spectral linewidths occurring at earlier times after ablation. The results will provide valuable data for understanding formation and time-evolution of femtosecond laser ablation plumes and how they differ from nanosecond ablation plumes.

4. APPLICATION OF LAAS

Although LAAS technology has entered the field of researchers as early as the 1960s, the practical application of LAAS technology as an elemental isotope analysis method has only developed in recent years. With the rapid development of laser technology and optical detection technology, LAAS technology has also made rapid development. Compared with traditional analysis technology, LAAS technology has the advantages of no complicated sample pretreatment, minimal sample demand, non-destructive detection, on-line measurement in long-distance harsh environment, fast measurement speed and high spectral resolution. It has been widely used in the field of element isotope analysis.

The application of LAAS in isotope ratio measurement is based on the principle of isotope shift. For different isotopes, due to the different number of neutrons in the nucleus, there are slight differences in the mass of the nucleus and the electron cloud outside the nucleus. This slight difference will lead to the difference in the energy level splitting of the excited state, which is reflected in the spectra, and will lead to slight differences in the absorption and emission line frequencies of different isotopes. It's called isotope shift effect.

Recently, the research on elemental isotope analysis using LAAS technology is mainly concentrated in Germany, the United States, and Japan. Related research in other countries is still quite limited.

4.1 The application of LAAS technology in Germany

In 2001, A. Quentmeier used laser ablation single-beam diode laser absorption spectroscopy to measure the uranium isotope ratio in solid samples [18] [27]. The diode laser was sequentially tuned to the absorption lines of both isotopes

（682.6736 nm for ^{235}U , and 682.6913 nm for ^{238}U ）. Three samples were used with the relative abundance of the minor isotope ^{235}U of 0.204%, 0.407% and 0.714%. The results showed that the limit of detection of the minor isotope was estimated to be 100 μg g^{-1} and the accuracy and precision for this measurement strategy were evaluated to be approximately 10%.

In 2002, on the basis of A. Quentmeier's work, H. Liu proposed using two diode lasers as the light source to determine the $^{235}U/^{238}U$ isotope ratio by laser ablation dual beam diode laser absorption spectroscopy. The precision and accuracy of $^{235}U/^{238}U$ isotope ratio determination were obtained to be 5 % and 2%, respectively , the detection limit of the minor isotope was estimated to be 18 μg g^{-1}. It has greatly improved the precision and accuracy of the single-beam detection method reported earlier [20].

4.2 The application of LAAS technology in the United States

In 1999, L.A. King used LAAS technology to conduct rubidium isotope measurements in solid samples. A Nd:YAG laser was used to produce the plasma on the surface of solid samples placed inside a low pressure chamber with a controlled atmosphere of 150 mTorr to 10 Torr and a narrowband Ti:Sapphire laser was scanned across the 780.02 nm transition of the rubidium isotopes. Measurement results showed that the isotope concentrations were determined in solid calcium carbonate samples to be 2.7±0.2 with a relative standard deviation of 5% and with a detection limit of 25 ppm per laser shot for each isotope [28] [29].

In 2009，B. A. Bushaw used laser ablation dual-beam diode laser absorption spectrometry to measure ^{152}Gd:^{160}Gd isotope ratios in micron-size particles [30] [31]. Two wavelength-tunable external cavity violet diode lasers (405 nm and 415 nm, respectively) are used to selectively excite each atomic isotope transition and measure the resulting isotope absorption. The diode lasers are tuned to specific isotopes in two different atomic transitions at 405.9 nm (^{152}Gd) and 413.4 nm (^{160}Gd), and directed collinearly through the laser ablation plume. The results show that single-shot detection sensitivity approaching the femtogram range and relative isotope ratio uncertainty better than 10%. This study laid the foundation for developing an engineered, onsite safeguards approach featuring continuous, automated aerosol particulate collection with uranium $^{235}U/^{238}U$ isotope ratio analysis [32].

In 2011, based on the above research, they invented a system and process with high accuracy and high precision destructive analysis measurements for isotope ratio determination of relative isotope abundance distributions in liquids, solids, and particulate samples [33]. The invention utilizes a collinear probe beam to interrogate a laser ablated plume. Isotope ratios and particle assays that can be determined with relative precision better than about 10%. In 2013, they made improvements based on the original technology. Isotope ratios that can be determined at approximately 1% or better precision and accuracy (relative standard deviation) [34].

In 2014, N. R. Taylor used a two-beam differential laser absorption technique to measure ^{238}U absorption spectra with high signal-to-noise ratios in atmospheric pressure laser-induced plasma [35]. A spectral linewidth (FWHM) of 2.23±0.13

GHz was found for the ^{238}U line in dry air at 760 Torr. And the result was significantly narrower than the 10 GHz linewidths observed in recent LIBS studies on atomic and ionic transitions near 424 nm [36].

In 2016, Mark C. Phillips presented the first measurements of tunable laser absorption spectroscopy in femtosecond laser ablation plasmas [26]. Time-resolved absorption spectra of uranium and aluminum are measured with high spectral and high temporal resolution. The results showed that strong atomic absorption signals in femtosecond ablation plumes are observed over time scales from 100's of ns to 10's of μs. The results also provide valuable data for understanding formation and time-evolution of femtosecond laser ablation plumes and how they differ from nanosecond ablation plumes.

4.3 The application of LAAS technology in Japan

In 2009, M. Miyabe conducted research on lanthanide elements using LAAS technology (scan mode) [37]. To develop high isotopic selectivity and detection sensitivity, they performed absorption spectroscopy of lanthanide elements under various ablation conditions (ablation pulse energy, ambient gas and its pressure) and observation conditions (atomic transition, geometry, distance to sample surfaces, observation time delay). Some analytical performances of this method were determined under optimum conditions and were discussed. In 2010, this research team investigated the time evolution of the neutral and singly-ionized cerium atoms in the ablation plume by the similar mode and obtained some experimental conditions for isotope analysis of atomic cerium from the detailed plume evolution [38].

In 2012, they studied the plume dynamics of uranium and determined suitable experimental conditions for isotopic analysis of uranium [39]. Using the optimum experimental conditions, they evaluated the calibration curve linearity, limit of detection, and precision for three samples having different abundances of ^{235}U.

In 2017, they performed a spectroscopic study of plutonium atoms and ions by employing the LAAS technique with a remote analysis system installed in a glove box [40]. Using the optimum experimental conditions, they obtained a limit of detection of 30-130 ppm and a relative standard deviation of approximately 6% for an abundance of ^{240}Pu of 2.4%. The results demonstrated that laser ablation absorption spectroscopy is applicable to the remote isotopic analysis of highly radioactive nuclear fuels and waste materials containing multiple actinide elements.

5. CONCLUSIONS

For some time, LAAS technology has gradually matured, and new research results and applications have emerged. It has become a fast, convenient, accurate and reliable isotope analytical technique. These LA-based analytical techniques have many potential applications in the field of nuclear technology because of their capability for rapid and remote (contactless) analysis of radioactive samples. Applying LAAS technology to the remote isotopic analysis of highly radioactive nuclear fuels has become a hot topic. Based on the many advantages of LAAS technology, LAAS technology will create more value in more fields through the unremitting efforts of researchers.

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