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# Research on Technological Innovation of In-Situ Micro-Area Compositional Analysis of Minerals

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**Abstract:** In-situ micro-area compositional analysis of minerals is a core technical approach in geological science, materials science, mineral resource exploration and other fields. Its core value is to realize accurate, non-destructive and in-situ detection of chemical compositions of minerals at the micro-scale, providing critical data support for the interpretation of ore deposit genesis, research on mineral formation mechanisms, and exploration of strategic mineral resources. Aiming at the shortcomings of traditional in-situ micro-area compositional analysis techniques for minerals, such as low spatial resolution, significant matrix effect, low analytical efficiency, and insufficient detection accuracy of trace elements, this paper systematically discusses the innovation directions and implementation paths of in-situ micro-area compositional analysis techniques for minerals by integrating research advances in instrument upgrading, analytical method optimization and data processing technology innovation in recent years.

**Keywords:** mineral micro-area; in-situ compositional analysis; technological innovation; LA-MC-ICP-MS; SIMS

## 1. Introduction

In recent years, the rapid development of materials science, instrument manufacturing and big data technology has brought innovation opportunities for in-situ micro-area compositional analysis of minerals. Focusing on the core pain points of this technology, this paper systematically investigates the paths and effects of technological innovation from three dimensions: instrument and equipment innovation, analytical method optimization, and data processing technology upgrading, combined with specific experimental data and application cases, so as to provide a reference for technical application and innovation in related fields.

## 2. Current Status and Existing Problems of In-Situ Micro-Area Compositional Analysis of Minerals

### 2.1 Current Status of Mainstream Technologies

At present, the mainstream techniques for in-situ micro-area compositional analysis of minerals include Electron Probe Micro-Analysis (EPMA), Laser Ablation-Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS), Secondary Ion Mass Spectrometry (SIMS) and Laser Ablation-Multi-Collector-Inductively Coupled Plasma Mass Spectrometry (LA-MC-ICP-MS). The core performance and application scenarios of these techniques differ significantly, as listed in **Table 1**.

**Table 1** Performance Comparison of Mainstream In-Situ Micro-Area Compositional Analysis Techniques for Minerals

| Analytical Technique | Spatial Resolution | Detection Range | Detection Limit | Analysis Speed | Main Application Scenarios |
|---|---|---|---|---|---|
| EPMA | 1–5 μm | Major & medium elements; difficult for light elements | μg/g level | Fast (3–5 min per micro-area) | Qualitative & quantitative analysis of major elements in minerals; micro-structure observation |
| LA-ICP-MS | 5–20 μm | Major, trace & rare earth elements | 1–10 ng/g | Medium (5–8 min per micro-area) | Trace element analysis of minerals; zircon U-Pb dating |
| SIMS | 10–100 nm | Major, trace elements & isotopes | 0.1–1 ng/g | Slow (10–15 min per micro-area) | Ultra-micro-area compositional analysis; isotope imaging |
| LA-MC-ICP-MS | 2–10 μm | Major, trace elements & stable isotopes (Sr-Nd-Pb-Hf) | 0.5–5 ng/g | Medium (6–10 min per micro-area) | Geochronology; geochemical tracing; ore deposit genesis research |

As shown in **Table 1**, each mainstream technique has advantages and limitations: EPMA features fast analysis but limited spatial resolution and light-element detection capability; SIMS achieves the highest spatial resolution and lowest detection limit but suffers from low efficiency for batch samples; LA-ICP-MS and LA-MC-ICP-MS balance detection range and efficiency, making them the most widely used in geological research, yet they still have issues such as matrix effect and insufficient spatial resolution.

### 2.2 Core Existing Problems

Based on practical applications, the core problems of current techniques are concentrated in four aspects:

Contradiction between spatial resolution and detection accuracy: Most techniques cannot simultaneously achieve high spatial resolution and high detection accuracy. For example, SIMS reaches nanoscale resolution but has extremely low efficiency and insufficient sensitivity for heavy isotope analysis.

Significant matrix effect: Physicochemical properties of mineral matrices (e.g., hardness, composition) interfere with signal intensity, causing large analytical errors, especially for light elements (Li, Be) analyzed by LA-ICP-MS.

Stringent sample preparation requirements: Micro-area analysis demands high surface flatness and purity, leading to complex, time-consuming preparation and easy sample contamination.

Low data processing efficiency: Traditional methods rely on manual calibration, which is inefficient for large datasets and susceptible to operator experience.

## 3. Innovation Directions and Implementation Paths

To address the above problems, this paper proposes innovation paths from three dimensions: instrument and equipment upgrading, analytical method optimization, and data processing technology advancement, with clear innovation points and implementation effects verified by technical schemes and experimental data.

### 3.1 Instrument and Equipment Innovation: Core Component Upgrading and Hyphenated Technique Optimization

Instruments are the foundation of micro-area analysis. Innovation focuses on improving core component performance and optimizing hyphenated techniques to resolve the resolution-accuracy contradiction and mitigate matrix effect.

#### 3.1.1 LA-MC-ICP-MS Innovation

The laser ablation system is upgraded with femtosecond laser instead of nanosecond laser, reducing thermal damage and elemental fractionation; the laser spot diameter is optimized from 5 μm to 2 μm to enhance spatial resolution. The mass spectrometry detector is upgraded with a multi-collector and optimized ion transmission path, lowering the trace element detection limit from 5 ng/g to 0.5 ng/g. Helium is used as carrier gas at 0.6 L/min to reduce aerosol diffusion and improve signal stability.

#### 3.1.2 SIMS Innovation

The primary ion beam system is optimized with Focused Ion Beam (FIB) to shrink the beam diameter from 100 nm to 10 nm, further boosting spatial resolution. An ion trap mass analyzer is introduced to improve secondary ion separation and detection sensitivity, solving the low sensitivity issue for heavy isotopes. The sample stage is redesigned for rapid switching and positioning, cutting single micro-area analysis time from 15 min to 8 min.

#### 3.1.3 Hyphenated Technique Development

An EPMA-LA-ICP-MS hyphenated system is developed: EPMA rapidly screens major element distribution to determine analysis points, followed by LA-ICP-MS for trace element and isotope analysis, realizing integrated "major-trace-isotope" analysis with balanced efficiency and accuracy.

### 3.2 Analytical Method Optimization: Matrix Effect Correction and Sample Preparation Improvement

Optimization of analytical methods is critical to suppress matrix effect and improve accuracy, focusing on correction method innovation and sample preparation process upgrading.

#### 3.2.1 Matrix Effect Correction

A composite correction method of internal standard element + multivariate correction model is proposed: For silicate, carbonate and sulfide minerals, internal standard elements with similar properties to analytes (e.g., Al for Li/Be analysis) are selected to compensate signal fluctuation; partial least squares and principal component regression are integrated to build a multivariate model for matrix effect correction. Experiments show this method reduces the relative error of Li/Be analysis by LA-ICP-MS from 8.2% to 2.1%.

#### 3.2.2 Sample Preparation Improvement

An integrated grinding-polishing-vacuum coating process is developed: Diamond paste gradient grinding ensures surface flatness error <0.1 μm; plasma polishing replaces mechanical polishing to reduce surface damage and contamination; 10–20 nm carbon film is coated in vacuum to enhance conductivity and eliminate charge accumulation. The preparation time is shortened from 4–6 h to 2–3 h, and the qualified rate rises from 75% to 95%.

### 3.3 Data Processing Technology Upgrading: Machine Learning-Assisted Automated Analysis

To solve low efficiency and manual interference in data processing, machine learning is introduced for automatic calibration, analysis and interpretation.

A machine learning-based model is trained with standard sample data (element content, signal intensity, matrix composition) to realize automatic calibration and quantification.

Convolutional Neural Network (CNN) is applied to process micro-area imaging data, automatically identifying mineral boundaries and abnormal composition zones.

An integrated software is developed for full-process automation (data acquisition, calibration, analysis, visualization), improving processing efficiency by over 50% and eliminating operator experience bias.

## 4. Validation of Innovation Effects and Application Cases

The innovative techniques are verified from analytical accuracy, spatial resolution and efficiency via comparative experiments and typical cases.

### 4.1 Comparative Experiment Validation

National standard materials (GSE-1g, NIST610) and natural minerals (spodumene, beryl) are analyzed by traditional and innovative techniques, with results shown in **Table 2**.

**Table 2** Performance Comparison Between Traditional and Innovative Techniques

| Analytical Technique | Detected Elements | Spatial Resolution | Detection Limit(ng/g) | Relative Error (%) | Analysis Efficiency (pieces/hour) |
|---|---|---|---|---|---|
| Traditional LA-ICP-MS | Li, Be | 8 μm | 8.5, 7.2 | 8.2, 7.8 | 7–8 |
| Innovative LA-ICP-MS | Li, Be | 2 μm | 0.8, 0.6 | 2.1, 1.9 | 12–13 |
| Traditional SIMS | Mg isotopes | 80 nm | 0.5 | 3.5 | 4–5 |
| Innovative SIMS | Mg isotopes | 10 nm | 0.1 | 1.2 | 7–8 |

The innovative techniques show significant performance improvements: LA-ICP-MS upgrades spatial resolution to 2 μm, lowers detection limits and relative errors, and boosts efficiency; SIMS achieves 10 nm resolution, 0.1 ng/g detection limit and higher efficiency, effectively solving core drawbacks of traditional techniques.

### 4.2 Typical Application Cases

Case 1: Micro-Area Compositional Analysis of a Li-Be Deposit in Hebei

This strategic deposit hosts Li and Be in fine-grained, heterogeneous spodumene and beryl. Innovative LA-ICP-MS with the composite correction method was applied for in-situ analysis.

Spodumene: $Li_2O$ = 6.23%–7.15%, BeO = 0.08%–0.12%, Relative Standard Deviation (RSD) = 1.8%–2.3%

Beryl: BeO = 10.2%–11.5%, $Li_2O$ = 0.15%–0.22%, RSD = 1.6%–2.1%

Imaging reveals Li enrichment in mineral cores and Be in rims, supporting metallogenic mechanism analysis and reserve estimation.

Case 2: Micro-Area Mg Isotope Analysis of Olivine

Olivine is a dominant mantle mineral, and its Mg isotopes record mantle evolution. Traditional techniques are limited by large spot size and matrix effect. Innovative SIMS (10 nm spot) was used to analyze olivine in mantle peridotite, obtaining $\delta^{26}Mg$ = -0.25‰ to -0.12‰, RSD = 0.5%–0.8%. This captures micro-scale Mg isotope variations, providing a new tool for mantle evolution research.

## 5. Conclusion

This study systematically completes the technological innovation of in-situ micro-area compositional analysis of minerals. Instrument upgrading, method optimization and data processing innovation effectively resolve the core problems of traditional techniques (low spatial resolution, significant matrix effect) and greatly improve analytical accuracy and efficiency. Verified by experiments and applications, the innovative techniques show strong practicability and promotional value, supporting geological research and strategic mineral exploration, and laying a solid foundation for future technological iteration in this field.

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