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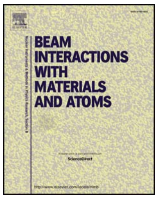

High spatial resolution and high brightness ion beam probe for in-situ elemental and isotopic analysis

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ABSTRACT

A high spatial resolution and high brightness ion beam from a cold cathode duoplasmatron source and primary ion optics are presented and applied to in-situ analysis of micro-scale geological material with complex structural and chemical features. The magnetic field in the source as well as the influence of relative permeability of magnetic materials on source performance was simulated using COMSOL to confirm the magnetic field strength of the source. Based on SIMION simulation, a high brightness and high spatial resolution negative ion optical system has been developed to achieve Critical (Gaussian) illumination mode. The ion source and primary column are installed on a new Time-of-Flight secondary ion mass spectrometer for analysis of geological samples. The diameter of the ion beam was measured by the knife-edge method and a scanning electron microscope (SEM). Results show that an O_2^- beam of *ca.* 5 µm diameter with a beam intensity of ~5 nA and an O^- beam of *ca.* 5 µm diameter with a beam intensity of ~50 nA were obtained, respectively. This design will open new possibilities for in-situ elemental and isotopic analysis in geological studies.

1. Introduction

High spatial resolution secondary ion mass spectrometry (SIMS) has been widely used in earth science research [1–4]. With the continuous development of isotope geochemistry and geochronology, a spatial resolution of several microns or even higher for routine in-situ analysis of mineral crystals with complex structural/chemical features at relatively high primary beam intensity are now required [5–8]. However, there is a trade-off between spot size and ion beam intensity. In general, SIMS analysis of geological material and isotopes requires a nanoampere primary beam intensity to yield enough secondary ions due to the very low concentration of many trace elements. SIMS cannot achieve high analytical precision using a picoampere primary beam. Thus, in order to obtain high analytical precision, a high brightness, high spatial resolution ion source and primary ion optics is one of the most important components in SIMS instruments designed to meet the increasing demands for in-situ microanalysis. Up to now, different types (including cluster ion sources, liquid metal ion sources and plasma source, etc.) of high brightness, high spatial resolution ion sources have been developed for use in different fields of research [9–16]. For geochronological studies, only positive ions of radiogenic isotopes are measured to determine the age of minerals, rocks and ore deposits [13,17–21]. As strongly electronegative O_2^- and O^- ions effectively increase the yield of electropositive secondary ions and reduce the influence of the charging effect on the analysis (which positive cluster ion sources and liquid metal ion sources are not doing), a duoplasmatron ion source is often used as the primary ion source in SIMS instruments and is widely used for in-situ microanalysis of geological material.

At present, NanoSIMS 50L instrument can deliver an O^- ion beam with a duoplasmatron ion source at the target of ~0.3pA at a spot size (16–84% rise distance) of ~170 nm, and 2pA at a spot size of ~340 nm [22,23]. The spot size can reach ~37 nm at target of ~0.15 pA with O^- after installation of a new RF plasma oxygen source from Oregon Physics [24]. However, this ion optical system is under patent protection. Rout et al. [25] updated a duoplasmatron ion source to produce a high brightness proton beam, associated with a tandem accelerator for nuclear physics applications. Though the new filament has greatly enhanced the performance and other source parameters, the hot cathode filament only has a lifetime up to 80 h that is not long enough for use in routine elemental and isotopic analysis. Guharay et al. [23] achieved a high brightness O^- ion beam using a compact gaseous plasma source in order to evaluate the suitability for high resolution SIMS. The ion

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optical column was designed to achieve a sub – 100 nm spot with a beam current of 10pA, which is still not enough for accurate isotopic analysis of geological material. Ishii et al. [26] developed a sub-micron ion beam system using a duoplasmatron ion source and special lens with the double functions of beam focusing and acceleration. The beam width was about 0.43 μm for 30 keV hydrogen ions and yielded 35pA during an experimental time of about 10 min. However, this source also uses a hot cathode filament with short lifetime. Coath and Long [27] designed a cold cathode duoplasmatron ion source for producing a beam of Ar^+ or O^-. According to their theoretical calculation, the O^- beam intensity could achieve a nanoampere level with focused probes about 5 μm in diameter with 15 keV ions. Studies indicate that O_2^- has a better performance in elemental analysis [17,19]. However, those with small beam size do not have enough beam intensity, whereas those with enough beam intensity do not have adequate spot size. As a result, none of the above ion sources can satisfy the demanding accurate in-situ isotopic analytical tasks in geological studies.

The duoplasmatron cold cathode ion source referred to in this paper is based on the design of Coath and Long [27], modified by Australian Scientific Instruments (ASI) and is used to produce O^- and O_2^- ions. The ion source magnetic field is simulated by COMSOL, and the ion optical column is simulated by SIMION. The ion optical system was developed to achieve smaller spots with higher intensity ion beams and allows precise isotopic and elemental measurements from areas several microns in diameter on interplanetary dust particles, meteorites, mineral sections and other geological samples.

2. System design

2.1. Ions source simulation

The duoplasmatron cold cathode ion source structure is shown in Fig. 1. O^- and O_2^- ions are generated by the gas discharge of the electrons of the cathode emission and then moved from cathode to anode. Ions are compressed by the intermediate electrode first and focused between the intermediate electrode and the anode electrode by the magnetic field to pass through the anode aperture and are then extracted by the extraction electrode to the ion focus system. The major advantage of negative ion beams in a SIMS primary column is that the surface charging voltage is limited to a few volts for material surfaces analysis. This value is not sensitive to the energy of the sputtering ion beam, even not on an electrically isolated target [23].

Coath and Long [27] investigated the performance of the ion source as a function of gas pressure, arc current, extraction potential, as well as anode to extraction electrode spacing. They pointed out that a high magnetic field is desirable for extracting negative ions, since maximum separation of the electron and negative ion population in the plasma is thereby achieved. The COMSOL simulation model of the ion source is simplified into magnetic material, insulator, copper and gas etc. (Fig. 2a). The direction of the magnetic field lines is mainly along magnet, intermediate and anode electrode permeability magnetic material, and then convergent between cathode and anode aperture from the COMSOL magnetic flux density distribution (Fig. 2b). Fig. 3 shows the variation of the magnetic field strength on the axis of the ion source. The magnetic field strength between the intermediate electrode and the anode is much higher than in the remaining region on the axis of the ion source. Therefore, the ion movement from the hollow cathode to anode electrode is mainly a function of the magnetic field force, whereas the trajectories in the remaining area are mainly influenced by the electric field force.

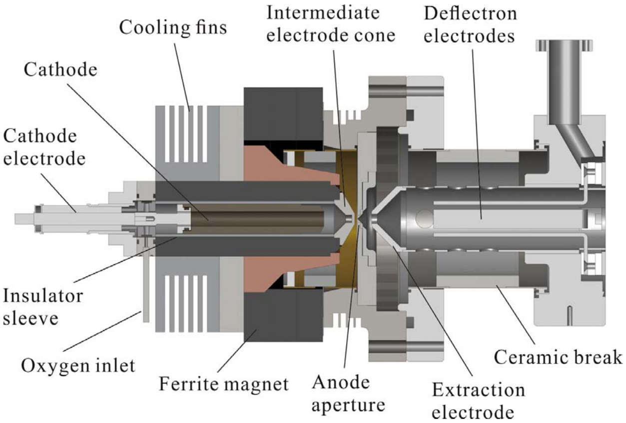

Fig. 1. Duoplasmatron (modified from Coath and Long [27] and Australian Scientific Instruments).

Since the magnetic field intensity is influenced by the magnetic permeability of material for any given magnetic field strength, the variation in magnetic field strength between intermediate and anode electrode aperture was studied by simulating material with different magnetic permeability. The simulation suggests that the magnetic field strength between intermediate and anode electrode aperture drops dramatically with material of magnetic permeability < 1000 and is positively correlated with magnetic permeability from 1000 to 4000; whereas the material with magnetic permeability over 4000, the magnetic field strength between intermediate and anode electrode aperture is almost stable (Fig. 4). Compared with the relative permeability of various materials, soft iron with a purity of 99.8% was chosen as the most suitable high permeability material, which has a relative permeability of ~5000. Moreover, the permeability magnetic material in use in the ion source is far less than its saturation field strength.

The magnetic field between the intermediate electrode and the anode is the key factor influencing the performance of the ion source. The magnetic field simulation model (Fig. 5) is set up to simulate the ions tracing between intermediate and anode electrode aperture under different magnetic field strength. By observing the focusing conditions of simulated ions under different magnetic field intensities, it is shown that the ions are helically focused by the magnetic and electrical fields. The O^- probe diameter on the anode aperture is first decreased and then increased with a continuous increase of the magnetic field strength. The O_2^- probe diameter becomes smaller as the strength of the magnetic field changes from 0 to 4.2 T (Fig. 6). The probe diameter is also controlled by the voltage between the intermediate and anode electrode. We applied a 0.4 T magnetic field intensity to the ion source, based on the maximum magnetic field strength which is limited by the dimension of the source and the angle of divergence of the ions.

2.2. Design of the primary ion optical system

An ion optical system (Fig. 7) was designed assuming a beam originating at an apparent source within the Duoplasmatron with initial ion beam parameters as follows: the initial beam diameter, current, ion energy, divergence angle, and energy spread were 200 μm, 1μA, 15 keV, 0.1°, and 15 eV [28] respectively. These parameters all conform to a Gaussian distribution. All remaining ion optical components of the overall primary column, including Einzel lenses, deflecting systems, a Wien Filter to select ion species, and an ion pulser, were designed to achieve focusing to small spots on the sample surface, with minimization of angular divergence in order to maintain high ion transmission. The entire ion optical system was simulated using SIMION, including matching, intermediate, and focusing lenses. The magnification factor of the first two (zoom) lenses, which focus the beam on a selectable "object" aperture (set at 100 μm), was adjustable between 1 and 2.5. In addition, to allow space for the secondary ion extraction system and the real-time visual observation system, the working distance of the condensing lens was designed to be 31 mm with a magnification factor of approximately 1:11. The intermediate lens has a magnification of approximately 1:2.5, which can be varied slightly. By simulating the ion optical system (Fig. 8) in the SIMION software, pulses and single ions