A LITHIUM LIQUID METAL ION SOURCE SUITABLE FOR HIGH VOLTAGE TERMINAL APPLICATIONS*

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Liquid metal ion sources offer potential improvement in lateral resolution over conventionally used gaseous sources for MeV microprobe applications because of their intrinsic brightnesses. The use of a Li liquid metal ion source is particularly attractive because of the increased sensitivity of lithium for detecting hydrogen (hydrogen profiling) and for detecting near surface contaminants through high-resolution Rutherford backscattering spectrometry. However, the technical difficulty of occasional sparking between the needle and extraction electrode requires that the needle of the Li liquid metal ion source be rewetted before reignition can be effected; this problem makes Li liquid metal ion sources of the usual design risky for operation in the insulated high-voltage terminals of accelerators used in such applications. We have designed a reliable, long-lived, Li liquid-metal ion source which has provisions for overcoming this limitation. The design features and performance characteristics of the source are described in this report.

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1.0 INTRODUCTION

High-energy (MeV), low-mass ion beams (H^1 , He^3 , and He^4) are routinely used for analysis of the near-surface regions of materials through the use of Rutherford backscattering spectrometry (RBS), nuclear reaction analysis (NRA), and particle-induced X-ray excitation (PIXE). The use of microfocused ion beams can provide information concerning the depth distributions, as well as lateral mapping of elemental contaminants within the near-surface regions of samples. The lateral resolution in microprobe facilities is limited by the minimum image size required to include ion beam intensities sufficient in magnitude for reasonable counting rates during a measurement. A potential toward improved beam brightness and, consequently, improved lateral resolution, is through the use of the liquid-metal ion source (LMIS). Their use for microprobe applications has been discussed elsewhere.¹

Lithium liquid-metal ion sources add an attractive complement to existing gaseous sources used for ion beam analysis because of improved sensitivities for hydrogen profiling using NRA and higher scattering cross sections for RBS. In this report, a lithium metal ion source is described, which has been operated for extended periods of time in the insulated high-voltage terminal of the Harwell Laboratory microprobe facility 5-MV Van de Graaff accelerator.² An earlier version of the source has been described previously.³

2.0 CHARACTERISTICS OF LIQUID-METAL ION SOURCES

Although the precise ion formation mechanism in the LMIS is still the subject of controversy, generation of ion beams in this source is quite simple. A strong electric

field, applied between a capillary or needle wetted with molten metal at anode potential and an extraction aperture, pulls the metal up into a modified Taylor cone⁴ with a rounded apex. If the field is sufficiently strong ($\sim 10^8$ V/cm) at the surface of the molten cone, ion desorption will occur. In this way, beam intensities of a few to several tens of μA can be formed. The remarkable feature of this source type is that the area of ion emission is extremely small – having a radius of curvature of a few Angstroms. Thus, the LMIS is, in principle, the brightest of existing positive ion sources. The central cores of beams extracted from the source are estimated to have brightnesses of $\sim 10^5$ times those of conventional gaseous discharge ion sources.⁵ However, in order to utilize the source for submicron imaging applications (e.g., microprobe surface analysis, ion implantation, microfabrication, and microetching), the angular divergence and, consequently, ion beam intensity, must be severely limited. This is a consequence of the highly divergent characteristic of ion beams from this source type and the difficulty of accelerating and focusing beams without severely distorting their phase spaces through spherical aberrational effects. The half-angular divergence of LMIS beams ranges from a few degrees at low beam intensities to a few tens of degrees at higher intensities.^{6,7} Inherent in LMIS beams are space-charge effects, which cause increases in their angular and energy spreads.

3.0 THE LITHIUM LMIS

3.1 Source Description and Operational Procedures

The complete lithium LMIS is shown schematically in Fig. 1, while a more detailed view of the source and acceleration electrode system is displayed in Fig. 2. The needle is made from 0.5-mm-diameter tungsten wire, sharpened to a tip radius of 10-20 μm electrolytically in a caustic soda solution. The sharpened wire is installed in

a close-fitting stainless steel hypodermic needle which provides a guide for insertion of the needle through the molten Li metal into the beam formation position and for retraction into the metal for wetting or rewetting the needle. The needle assembly is passed through guide holes in a stainless steel reservoir containing molten lithium until the tungsten tip penetrates the liquid lithium. The reservoir is maintained at the operational temperature by an ohmic heater. The whole assembly is mounted on an electrically insulated vacuum chamber flange. The tungsten needle is initially positioned within 1.5 mm of the extraction electrode. The tip can thus be withdrawn into the melt for rewetting, if necessary, by means of a remotely actuatable solenoid. This device was found to be essential for reliable long-term operation of the source within the insulated gas high-voltage environment of the 5-MV Harwell Van de Graaff accelerator due to the inability to reignite the source following occasional sparks to the needle. The source is loaded with a short length of 3-mm-diameter lithium wire previously cleaned in a solution of 10% CH_3OH in CCl_4 and quickly transferred in atmosphere to the reservoir of the source. The reservoir is heated to a temperature of about 550°C and the tip immersed for about 30 minutes in the liquid lithium in order to wet the tungsten. For source operation, the lithium temperature is lowered to a value between its melting point of 180°C and 300°C , and the tip moved to a position less than 1 mm (typically, 0.5 mm) from the extraction electrode aperture. The lower operational temperature was found necessary in order to reduce vaporization and subsequent condensation of lithium in the extraction and acceleration electrodes, which eventually leads to sparking.

The source acceleration electrode system is similar to that used for a gallium LMIS described in previous reports.^{8,9} Ions are extracted by maintaining the tip at 5-10 kV positive with respect to the extraction electrode. A schematic of the power supply arrangement is shown in Fig. 3. After extraction, the ions are accelerated

further by 10-20 kV by a potential maintained between the extraction electrode and an acceleration grid, passed through a molybdenum aperture plate, and focused using a gridded einzel lens into an electron-suppressed Faraday cup. It was found necessary to incorporate a 50-M Ω resistor in the supply to the source emitter in order to obtain stable emission currents over long periods. This resistor also served to quench any long-duration sparks which occur occasionally, thus limiting damage to the emitter tip.

3.2 Source Performance

To date, the source has produced Li ion beam intensities up to 15 μ A at an operating reservoir temperature of $\sim$ 300°C. The gap distance between the emitter and the extraction electrode has been varied between $\sim$ 0.25 and 1 mm. The field strength required to effect ignition is found to be approximately constant at 20-25 kV/mm for this range of settings. A typical voltage-Li current characteristic curve for an extraction gap of 0.4 mm, is shown in Fig. 4. Useful Li ion beam intensities are typically found to be 10% of the total anode supply current, which includes secondary electron emission.

The dependence of Li⁺ ion beam intensity on reservoir temperature has not been fully examined. However, observations during cooling of the source from 300°C to the solidification point for Li (180°C) show a reduction in ion beam intensity of only a few percent. Ion emission instantly ceases upon lithium solidification.

Analysis of beam using a 90° magnet revealed a variety of ion species, including ⁶Li⁺, ⁷Li⁺, ⁷Li₂⁺, and ⁷Li₃⁺ in relative abundances of 6%, 85%, 6%, and 2.6%, respectively. The observed ion beam intensities were generally stable with fluctuations of a few percent per hour. Up to the present time, the longest period of

continuous operation has been 30 hours, during which the source operated stably in the terminal of the Harwell Van de Graaff accelerator. However, the source has run intermittently for 50 hours before malfunctions occurred which were due mainly to Li vapor deposition. The ability to remotely withdraw the needle into the molten Li for rewetting, often necessary after sparking to the needle of the source, has proved to be an important practical attribute of the source, which makes the Li LMIS a viable option for use in high-resolution RBS and NRA (hydrogen profiling) analyses of samples for which the source must operate stably and reliably for extended periods of time in the insulated gas environment of the high-voltage terminal of an electrostatic accelerator.

Efforts are presently under way to estimate the brightnesses of Li^+ ion beams extracted from the source.

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Figure Captions

1. The Li LMIS equipped with a solenoid for retracting the tungsten needle into the molten Li for wetting/rewetting the needle.
2. The Li LMIS and acceleration electrode system.
3. The power supply arrangement for extracting and accelerating ion beams from the Li LMIS.
4. Li^+ beam intensity versus acceleration voltage.

Fig. 1

Fig. 2

Fig. 3

Fig. 4