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LIQUID METAL SOURCES IN ION MICROSCOPY AND SECONDARY ION MASS SPECTROMETRY

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Abstract

This paper reports on state-of-the-art developments in liquid metal sources and some of the finer points of their operating characteristics that are especially relevant to the satisfactory functioning of analytical ion scanning microscopes equipped with secondary ion mass spectrometers. Such effects include unwanted emissions from the source and their exclusion by means of filters and mass separators in the ion-optical column. The design of the ion optical column is also discussed and some applications of this rapidly advancing form of analytical microscopy are described.

KEY WORDS: Liquid Metal Field Ion Sources. Secondary ion microscopy. Ion microscopy. Sub-micron ion imaging. Needle sources. Capillary sources. Mass filtering. Surface analysis. Ion optics. Ion-specimen interaction.

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Introduction

Liquid metal ion sources (LMIS) go back to the early sixties, when they were investigated in connection with space-thruster research. It was during these investigations that Krohn (1961) remarked on the fact that liquid metal cones, formed at the nozzle of electrically charged capillaries, produced large quantities of ions; but at the time the main interest was in charged droplets, also produced in LMIS. Several research groups investigated "capillary" type LMIS throughout the sixties and early seventies, and although the emphasis gradually shifted from charged droplets to ions, space propulsion remained the main objective. Ions produced by LMIS are still important for space propulsion (Bartoli et al 1982). These early investigations produced some important results that provided a basis in the subsequent "boom" in LMIS, when R.Clampitt's group at the UKAEA Culham Laboratory and, subsequently, their collaborators at Oxford University took a renewed interest in the subject. Notable research teams of the "pre-boom" era were those at the Argonne National Laboratory (Krohn 1961, 1974), at the University of Illinois (Evans and Hendricks 1972) and at Pasadena (Mahoney et al 1969).

It was Krohn and Ringo (1975) and Clampitt et al (1975) that, independently, emphasised the importance of LMIS as high brightness ion sources for ion-optical instrumentation. The brightness of the sources was estimated as being in the range $10^5 - 10^6$ Acm^{-2}sr^{-1}, considerably higher than that of previously known sources.

Needle-type LMIS

The so called "needle" type LMIS, invented and developed at the Culham Laboratory (Clampitt et al 1975, Aitken 1976), has now largely replaced the earlier version that employed a capillary tube filled with liquid metal. The principle of operation of LMIS rests on the formation of an electric field-sustained cone-like protrusion at the nozzle of a capillary or at the apex of a needle covered by a liquid-metal film. The emission of ions, atoms, clusters and droplets takes place at this apex. The ion currents involved are in the microamp range (typically

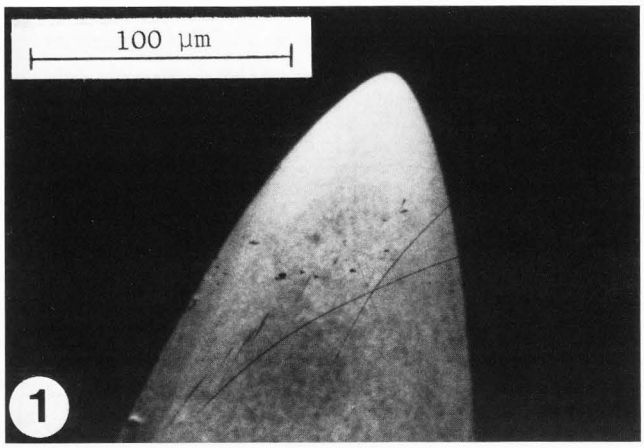
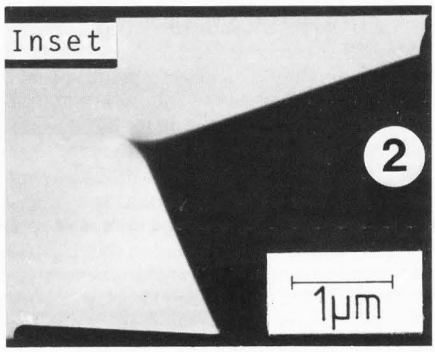
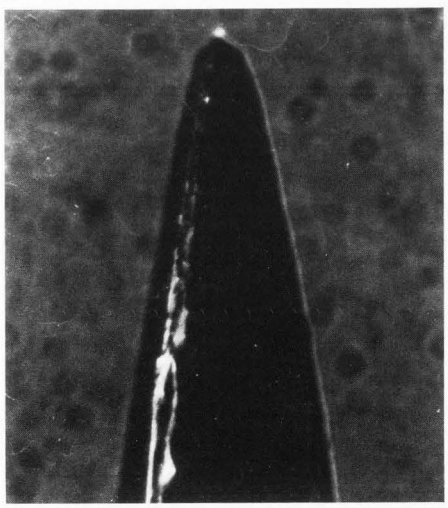


Fig. 1 Unwetted tungsten needle of 10 μm apex radius.





1-100 μA). Figure 1 shows a needle before wetting, and Figure 2 shows needle ion sources during operation. Needle ion sources have some advantages over capillary sources. Accurately shaped needles of specified dimensions are not difficult to manufacture. Furthermore, they can be made from a wide range of materials, thereby extending the range of liquid metals that can be used in LMIS. For example, in order to make a liquid aluminum source, the Oregon group (Bell et al 1982) machined a carbon needle which they subsequently coated with titanium; this was the only way that could be found of preventing a reaction between the aluminum and the substrate. It is hard to imagine, that a liquid aluminum source could be operated in a "capillary" mode - unless, of course, a suitable aluminum alloy could be used with subsequent mass-separation of the aluminum ions. Furthermore, in view of the large volume of liquid exposed to the high field and their very low flow impedance, capillaries are likely to emit a high proportion of undesirable droplets. Nevertheless, with careful design and attention to detail, these difficulties can be overcome and both types of source are in commercial production. In general, for satisfactory performance in a needle-type LMIS, the liquid metal must have a relatively low melting point and an adequately low vapour pressure. It must also wet the substrate, but not react with it.

Some basic mechanisms in LMIS

When the voltage V_o between a liquid-metal-coated needle and a suitably positioned counterelectrode exceeds a critical value, a sudden initiation of ion emission, at microamp. level, occurs. The sharp onset of the current seen in Figure 3 is a consequence of the formation, at this critical voltage, of a cone-like protrusion (Fig.2) at the apex of the needle. The theoretical value of V_{oc}, calculated from a stress balance between the electrostatic forces and those of surface tension at the surface of the liquid film, has been found to agree well with experimentally determined values (see e.g. Mair 1980 and Mair and Mulvey 1984). It should be emphasised that the above theory merely predicts the onset of ion emission. It reveals nothing about the emission mechanism itself. Useful clues as to the nature of the ion emission mechanism emerge, however, from high resolution energy distribution measurements on the ion beam using retarding-field analysers. In addition, such measurements provide essential data for the design of the ion-optical column. Since ions have a

Fig. 2 Gallium - coated tungsten needle, during operation at i = 50 μA. Note luminous spot near the tip (Photo by I. Brown). Inset (courtesy Prof. P. Sudraud): TEM micrograph showing liquid cusp at the apex of a needle at the same current. For i = 10 μA the jet has a diameter of some 3nm near its apex (Benassayag and Sudraud 1985).

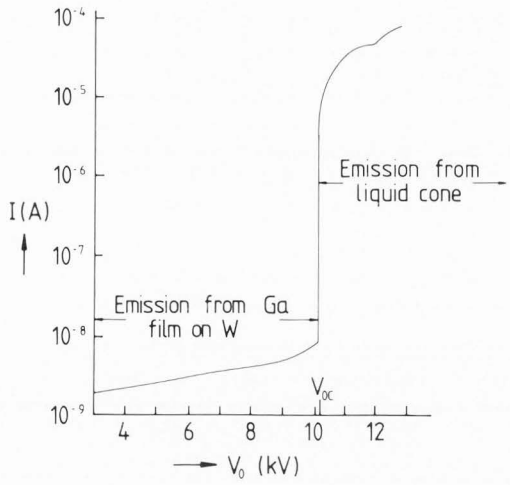


Fig. 3 Illustrating the onset of cone formation at the critical voltage V_{OC} at the apex of a gallium-coated needle at 1000 K (Ohana 1980).

considerably greater mass than electrons, and hence travel more slowly, mutual interactions are more severe and considerable energy spread in the beam might be expected; this gives rise to chromatic aberration in the subsequent lens system, and hence loss of resolution in the ion probe. Moreover, the ion formation process itself will inevitably require the expenditure of energy, so that the mean energy of the ion beam is somewhat lower than that corresponding to the applied voltage. This energy deficit is also important inasmuch as it gives valuable clues as to the mechanism of ion formation. Figure 4a shows typical energy spectra for a gallium ion source; Fig 4b is a plot of the energy spread $\Delta E_{1/2}$ (FWHM) as a function of ion current; finally, Fig 4c shows how the peak of the energy distribution varies with the ion current (i).

The energy spread curve of figure 4b appears to consist of three regions. The linear region in the middle of the curve can be satisfactorily explained by Knauer's (1981) theory, a treatment that attributes the increase in energy spread with current to collisionless Coulomb interactions in the ion beam; the magnitude of the slope of the experimental curve (0.72) is in reasonable agreement with the two-thirds power dependence of the full-width at half maximum (FWHM) on current predicted by the theory. Coulomb interactions in the beam arise from the high current densities involved in LMIS. The high current region, where the curve deviates from linearity, is attributable to gross instabilities (Mair and von Engel 1979, Mair and Mulvey 1984) of the liquid emitter that invalidate the conditions on which Knauer's theory is based (Mair et al 1983a,b). Below an emission current of some 2 μA another type of energy broadening mechanism, different from that suggested by Knauer, seems to operate and no satisfactory explanation has so far emerged. It is interesting to note, however, that this portion of the curve extrapolates back to an FWHM value of 1.2 eV for a current of 1 nA; this result is in satisfactory agreement with the value

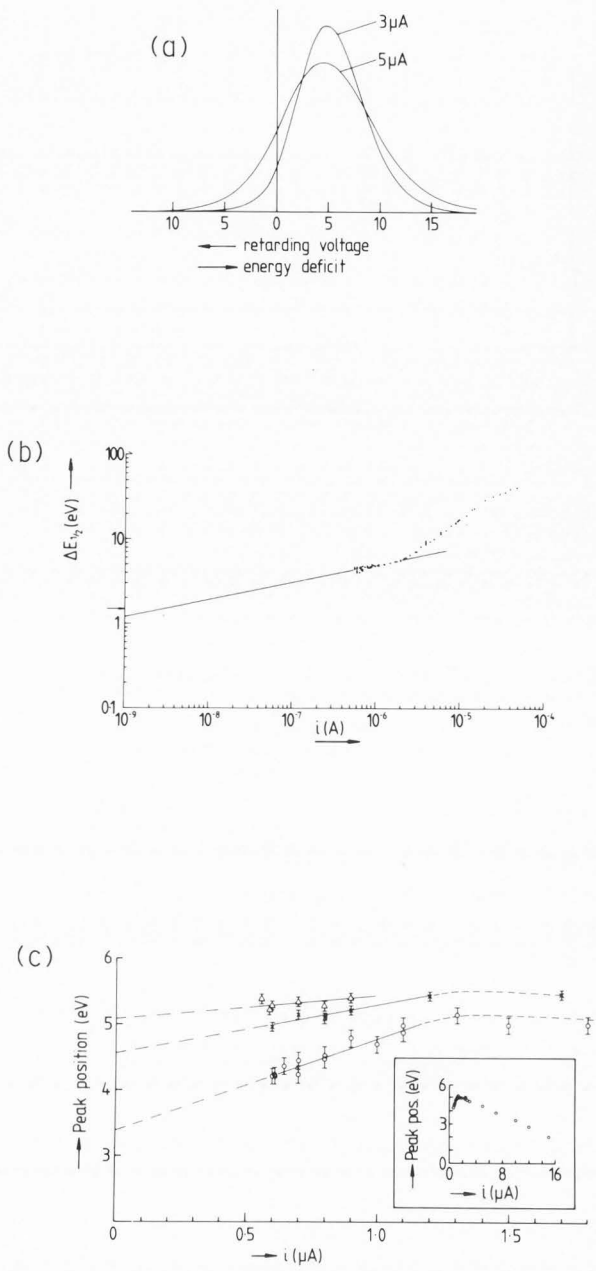


Fig. 4 (a) Energy distributions in a Ga^+ beam. (b) Energy spread (FWHM) vs. current i; arrow indicates the value obtained by Culbertson et al. (1979a,b) for a liquid gallium film on tungsten for $i = 10^{-9} - 10^{-8}$ A. (c) Peak position of the energy distribution vs. i. Note: absolute values of peak positive change with alteration of retarder work function ϕ_C because of contaminants in the vacuum system (eq. (1)). Inset: same as 4c but for a wider current range.

of about 1.5 eV for currents of 1-10 nA obtained for gallium films on very sharp tungsten needles - apparently with no cone formation (Culbertson et al 1979a,b). It is quite clear that in the latter case field-evaporation takes place and the fact that our measurements extrapolate back to the

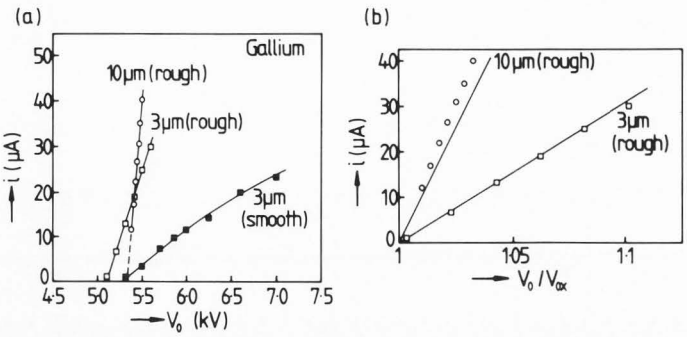


Fig. 5 (a) Experimentally obtained current voltage (i-V_o) curves for gallium needle LMIS. i is the ion current emitted and V_o is the potential difference between emitter and extractor. Results for needles of 3 μm apex are from Kingham and Swanson (1984). (b) Comparison of (i-V_o) curves of low flow-impedance (rough) needles with predictions of equation 2 (solid lines). Symbols as in 5(a). V_{ox} is the voltage for which $i \to 0$.

Culbertson et al result encourages us to think that the same mechanism might also operate in our case (Mair et al 1984).

Additional evidence that the ion generation process is predominantly one of surface field-ionisation (e.g., field-evaporation), rather than field-ionisation in space, emerges from measurements on the energy peak position of the energy distribution:

Figures 4a and 4c (inset) show that with increasing emission current the peak moves towards the potential of the emitter (zero retarding potential), i.e. towards lower energy deficits. For currents greater than 2 μA this behaviour is almost certainly a consequence of the Coulomb broadening of the energy distribution (see Mair et al 1983b), and the linear dependence of the peak shift in this region agrees with recent theoretical predictions (Gesley and Swanson 1984). For very low currents, ($i < 1$ μA) , however, the peak energy deficit increases (Fig 4c). Assuming that no Coulomb broadening (or any other type of broadening arising from particle interaction) occurs for $i < 1$ μA , the shift can conceivably be thought of as being due to the increase with electric field of the heat of evaporation (Λ) and the decrease of the energy barrier (Q) seen by an escaping ion (see, e.g., Forbes and Chibane 1982, Forbes et al 1984) . The so called "critical energy deficit" (ΔE) in field evaporation/surface field-ionisation (which is related to the peak location) is given as (Tsong et al 1977; see also Swanson et al 1980):

$$\Delta E = \Lambda + I - \phi_c - Q \qquad (1)$$

where I is the ionisation potential of the metal and ϕ_c the work function of the retarder. Clearly, as Λ increases and/or Q decreases, ΔE increases. However, the existence of such an emission-related energy spread in the beam would cast doubt on the good agreement with Knauer's theory found for the middle region of Fig. 4b. Any such 'inherent' energy spread would have to be subtracted in quadrature from the measured FWHM values when determining the power dependence of the FWHM on the current i from a log-log plot. It is clear, therefore, that there are still some unanswered questions concerning LMIS. Nevertheless, equation (1) would still be valid. Assuming, therefore, that Q is small (Swanson et al 1980) we calculate that in our experiments $\Delta E \approx 4.3$ eV. This compares satisfactorily with the values of Fig 4c - even allowing for the fact that the peak location does not exactly correspond to the critical energy deficit.

Finally, field evaporation followed by post-ionisation (Ernst 1979) to higher charge states appears to be compatible with mass-spectroscopically determined ratios of the amount of doubly to singly-charged ions in the beam. (Kingham 1983, Swanson 1983). In conclusion, therefore, field evaporation is a strong candidate as the principal mechanism of ion emission in LMIS. However, there are notable exceptions, such as Si^+ produced by a gold-silicon source (Swanson 1983) and U^+ (Van de Walle and Sudraud 1982). In these two cases, judging from energy distribution measurements, spatial field ionisation appears to play a significant, or, for the U source, even a dominant role. Field-ionisation might also contribute, by small amounts, for other liquid metals, particularly at high currents; however, calculations suggest that, for gallium at least, the necessary atom fluxes would have to be produced by a mechanism other than thermal (or classical) evaporation (Mair and Aitken 1984).

Current-voltage characteristics

Fig 5a shows some current-voltage curves obtained with gallium LMIS. It may be deduced from the curves obtained from a source having a needle of apex radius 3 μm , that rate of flow of liquid metal has a profound effect on the rate of rise of the ion current (i) with ion extraction voltage (V_o) : a needle with a smooth lateral surface presents a high impedance to flow and results in a relatively flat i-V_o characteristic, whereas for a needle having longitudinal grooves on its surface, the flow is greatly facilitated and the current rises steeply with voltage. It was shown recently (Mair 1984a,b) that when the flow impedance is negligibly small - as is the case with capillary sources - the i-V_o characteristics are controlled essentially by the effects of space-charges. The relevant equation for cylindrical capillaries of radius R is given by:

$$i = \frac{3\pi(2e/m)^{1/2} RT\cos\phi\,[(V_o/V_{ox} - 1) + (V_o/V_{ox} - 1)^2/2]}{V_o^{1/2}} \qquad (2a)$$

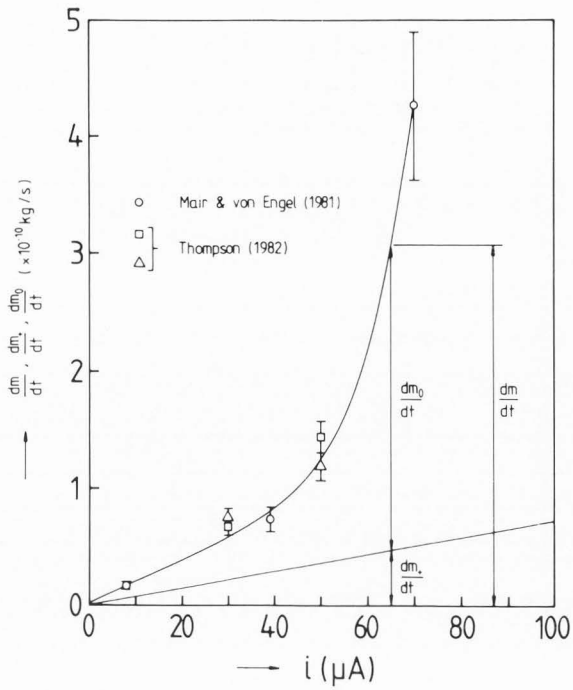


Fig. 6 Non-ionic emission from gallium needle LMIS:
dm/dt=total mass emission rate;
dm_+/dt=mass emission rate of Ga^+ ions;
$dm/dt - dm_+/dt = dm_0/dt$=non-ionic mass emission rate.

Note. The ion beam of gallium LMIS consists almost entirely of Ga^+.

or, for $V_0/V_{ox} \leq 10\%$, to an excellent approximation

$$i = 3\pi(2e/m)^{1/2} RT\cos\phi[V_0/V_{ox} - 1]/V_{ox}^{1/2} \qquad (2b)$$

T being the surface tension, e/m the charge-to-mass ratio of the emitted ions, ϕ the fluid-capillary contact angle (taken as 49.3° (Taylor 1964)) and V_{ox} the extinction potential (i.e. the value of V_0 when $i \to 0$).

Equation (2) holds, strictly speaking, for cylindrical geometry, but should in principle apply to needle-type LMIS of sufficiently low flow impedance. Such needles are likely to be those with rough (i.e. grooved) surfaces. Thus Fig 5b compares equation 2a, or 2b, with the two corresponding experimental curves in Fig 5a described as "rough" (where R in (2a,b) now denotes the radius of the needle apex). Since the needles are not cylindrical, the agreement is perhaps better than might have been expected.

Non - ionic emission in LMIS

It was pointed out in 1979 (Mair and von Engel 1979) that in LMIS, at all current levels, there is a substantial component of the emission that cannot be accounted for by the emission of ions. These observations were based on gallium "needle" LMIS, whose ion emission spectrum was known to consist almost entirely of Ga^+ ions, with very small amounts (<1%) of Ga^{++} and multi-atomic (cluster) ions. Fig.6. shows the total mass emission rate (dm/dt) of gallium LMIS as a function of ion current; also shown is the mass loss rate (dm_+/dt) due to Ga^+ ions It can be seen that, even at small currents, more than half of the total mass emitted is not in the form of ions. Part of this non-ionic mass consists of gallium atoms, the excitation of which gives rise to the luminous spot observed near the tip (Mair, 1980; Venkatesan et al 1981) (Fig 2); the greater part, however, consists of feebly charged droplets that travel predominantly along the paraxial region of the system (Wagner et al 1981, Thompson and von Engel 1982, Papadopoulos et al 1984). The radius of these droplets can be as small as a few nanometers at low currents (Farrow et al 1978), in which case they are often referred to as microparticles (Thompson and von Engel 1982; see also Krohn 1974); at very high currents the radius of the droplets can exceed 1 μm (Prewett et al 1981). It is perhaps worth mentioning that the experimental measurements of Mair and von Engel (1981) were made simply by measuring the mass-loss suffered by the gallium-coated emitter with time, whereas the experiments of Thompson (1982) measured not only the mass-loss of the emitter but also the mass-gain by the target. Thompson found that the difference between these two quantities was not more than the estimated experimental error of ∓ 10%. Moreover, in some of Thompson's measurements, shown by the symbol Δ in Fig 6, collector-released secondary electrons that might otherwise land on the emitter were suppressed; here again the difference between mass loss and mass gain found experimentally was within the general experimental error of the measurements. This finding supports the earlier conclusion of Mair and von Engel (1981) - reached by a series of indirect observations - that "the emission of neutral atoms from the anode of LMIS is a fundamental property of these devices, independent of any heating caused by the impact of secondary electrons". We now know, of course, that the majority of these atoms are not emitted individually, but as part of larger ionic complexes or droplets. The experiments of Wagner et al (1981) produced somewhat different results from those of Fig.6; but in this case too the non-ionic component of the emission was found to be quite substantial, i.e. about 30% of the total mass emitted, even at currents as low as 1.5 μA ; at high currents this proportion increased dramatically, as in the experiments of Mair and von Engel (Fig.6). The findings of Wagner et al. may not, strictly speaking, be comparable to those of Fig.6, for differences in ion source construction - leading to differences in flow impedance - may lead to differences in the ion conversion efficiency of the mass emitted. Experiments performed at the European Space Agency appear to support this view (Bartoli et al 1982).

Ion optics of LMIS

The ion-optical characteristics of LMIS are closely related to those of thermally assisted electron field emission sources (TF) operating at high probe currents (see El Gomati et al 1985),

where the electron-electron interactions (Boersch effect) play an important part in the beam formation process. This similarity between LMIS and TF sources arises since the effective (gaussian) source size is small (< 50 nm) in both cases. In an LMIS, the gaussian source size is not easy to determine experimentally because of the combined effects of the spherical and chromatic aberration coefficients of the extractor and accelerating electrodes. Since the required probe size is, in general, larger than the gaussian spot size, an ion-optical system incorporating an LMIS must produce a small magnification, perhaps between one and two times. It is this fact that distinguishes LMIS from other forms of ion sources, which usually require a considerable de-magnification in order to form a probe of the required diameter. In practice, the high intrinsic brightness of LMIS cannot yet be fully exploited in ion-optical columns; this arises because the energy-spread in the beam and the inevitable chromatic aberrations of the lens system set an upper limit to the probe current that can be achieved in a probe of given diameter as discussed below.

In the absence of aberrations the current I_p produced by an ion source of diameter d_s emitting into a solid angle α is given by

$$I_p = \pi^2 d_s^2 \, \alpha^2 \beta / 4 \qquad (3)$$

where β is the directional brightness ($Am^{-2}sr^{-1}$). In practice, the independent measurement of d_s and β is difficult in the presence of chromatic aberration and so the term $\pi \, d_s^2 \beta / 4$ is often replaced by the less useful quantity $dI/d\Omega$, or J_Ω, i.e. the current per unit solid angle.

Present LMIS design, still in its infancy, is largely based on experience with electron field emission systems. For detailed reviews, see for example Lauer (1982) and Kasper (1982). Not all the advances in electron sources can, however, be applied directly to LMIS. Thus, the substantial reduction of electron gun aberrations by placing a miniature magnetic lens in the vicinity of the emitting tip (Troyon 1980) is impractical for LMIS simply because the strength of a magnetic lens varies as the charge to mass ratio of the charged particles. Ions are therefore only weakly focussed by conventional magnetic electron lenses. Fortunately electrostatic lenses are equally strong for all charged particles and thus many ideas from electron optics may be transferred directly to LMIS optics.

In order to illustrate this, Figure 7 shows a simple two-electrode arrangement, similar to that of El Gomati et al (1985), consisting of an extractor and an accelerating electrode, focussing the beam of ions emerging from the tip of the LMIS onto a specimen placed some distance away. The tip is located at a distance S_o from the extractor and the specimen is at a distance S_i from the accelerator electrode. The ion beam entering the extractor lens has a semi-angle α_o and the beam converging on the specimen has a semi-angle α_i. For a given object distance S_o, the ion extraction voltage V_o on the extractor is determined by the field required to form the liquid tip; V_o can be varied only over narrow limits. The accelerator electrode then focusses the beam onto the specimen. Thus a two-lens system can be used at only one accelerating voltage. In practice, therefore, three or more focussing electrodes will be needed, depending on the required range of accelerating voltages and what ion-optical properties are to be varied while maintaining the emitting tip and the specimen in the same place. For further consideration of the question of multi-electrode systems see, for example, Heddle and Papadovassilakis (1984).

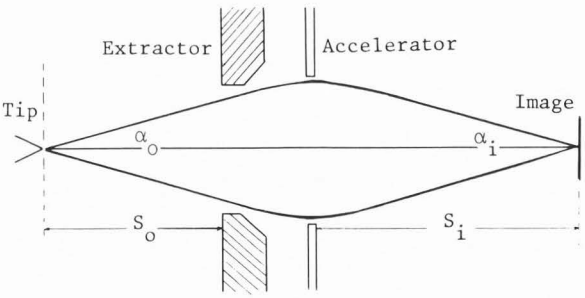


Fig. 7 Schematic arrangement of a simple two-element LMIS, forming an image of magnification M in the specimen plane. α_o - semi-angle of emission in object space, α_i - corresponding semi-angle of convergence in the image.

Referring again to Figure 7, the emitting liquid cone is formed by applying the critical voltage V_{oc} between the extractor and tip. The shape of the extractor electrode is important; a suitable form is that of a moderately thick electrode, flared out into a cone as suggested by Munro (1971) to provide a smoothly divergent trajectory through the aperture with minimum chromatic and spherical aberration. In addition, the extractor should not intercept the incoming ion beam unduly; otherwise backscattered particles may 'poison' the source. After passing through the extractor, the ions are further accelerated by the accelerator electrode held at a potential V_i with respect to the tip and converged onto the specimen. The magnification M of the system is given by the Helmoltz-Lagrange equation

$$M = \frac{\alpha_o}{\alpha_i} \left(\frac{V_o}{V_i} \right)^{1/2} \qquad (4)$$

where the term $(V_o/V_i)^{1/2}$ corresponds to the ratio of the refractive indices in the object and image spaces. In practice the ratio (V_o/V_i) is around 0.1 so that the magnification M is reduced by a factor of three. The magnification is thus a function not only of the object and image distances S_o and S_i but also of the potentials V_o and V_i. Detailed calculations of the optical properties, including the magnification for such a two electrode system have recently been made by El Gomati et al (1985). Under present conditions in ion microscopy and ion beam

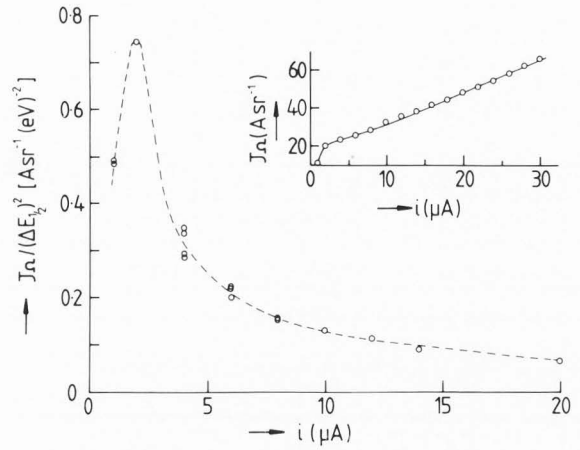


Fig. 8 Experimental chromatic angular intensity $J_\Omega/(\Delta E_{1/2})^2$ vs. i for gallium LMIS. Curve is based on the data of Fig. 4b and that of Swanson et al (1979) (see inset).

microfabrication, the probe sizes and currents that are required, together with the large energy spread (5 eV) in the ion beam cause the chromatic disc of confusion to be much larger than the gaussian size of the image, thereby setting a limit to the current that can be produced in a probe of given diameter. The current I_p leaving the source in a cone of semi-angle α_o is given by

$$I_p = J_\Omega \pi\alpha_o^2 \qquad (5)$$

The diameter d_c of the disc of confusion in the Gaussian image plane, due to chromatic aberration is given by

$$d_c = 2MC_c\alpha_o \frac{dV}{V_o} \qquad (6)$$

where M is the magnification of the source at the image plane. C_c is the chromatic aberration coefficent and dV/V_o is the relative variation of the energy of the ion beam at the specimen. If the disc of confusion due to chromatic aberration is large compared with the gaussian image size, the current density J_p at the specimen will be given, to a first approximation, by the quotient of the current I_p and the area of the disc of confusion. Hence J_p is given by

$$J_p = \frac{1}{M^2} \frac{J_\Omega}{(dV)^2} \frac{V_o^2}{C_c^2} \qquad (7)$$

The chromatic aberration coefficient depends directly on the focal length of the probe forming lens and is approximately equal to it for a weak lens. It should also be remembered that the coefficient C_c is defined for infinite conjugates, i.e. a parallel incident beam. As the magnification approaches one (the value commonly used in LMIS) the value of C_c approximately doubles. This will reduce the value of J_p by roughly a factor of four. On the other hand, it is possible to reduce the effective value of dV by weakening the focussing lens so that changes in energy of $\mp$ dV/2 rather than from 0 to dV occur about the de-focus position. Thus the value of J_p as given in Equation (7) should be approximately correct for most practical situations. The other important factor in Equation (7) is the term $J_\Omega/(dV)^2$, conveniently expressed in μA sr^{-1}(eV)$^{-2}$. In the absence of other effects this quantity is equivalent to $J_\Omega/(\Delta E_{1/2})^2$, and is a distinctive property of a given design of LMIS. Figure 7 shows its form as a function of beam current for a gallium needle LMIS. The data were taken from the J_Ω vs i curves of Swanson et al (1979) and the energy spread measurements of Mair et al (1984) given in Fig 4b. It can be seen from the curve of Figure 8 that the value of $J_\Omega/(\Delta E_{1/2})^2$ increases rapidly up to a value of some 2 μA, after which it decreases rapidly for higher currents as losses in the beam increase. This transition point coincides with the characteristic kink in the J_Ω vs i curve shown in the inset, and also with the kink of the energy spread curve of Fig. 4b. This curve indicates that this transition point marks the optimum operating conditions for the gallium LMIS of Swanson et al (1979); and in spite of likely variations in angular intensity amongst gallium LMIS of different design the general shape of the curve in Fig. 8 is probably typical. As is clear from this figure it is desirable to avoid operating in the higher current regions. The current Ip in a probe of diameter d at the specimen is given by

$$I_p = J_p\pi d^2/4 = \frac{\pi d^2}{4M^2} \frac{J_\Omega}{(dV)^2} \frac{V_o^2}{C_c^2} \qquad (8)$$

Equation (8) is of course subject to the same constraints that apply to Equation (7), namely that it is valid for a beam focussed onto the gaussian image plane and for magnifications large compared with unity. In practical LMIS neither assumption is adequately fulfilled. However, if the ion beam is optimally underfocussed so as to reduce the chromatic disc and if the magnification is around unity, Equation (8) should still give a close approximation to the current attainable in practice. Equation (8) is similar to that derived by other authors, although in the case of Swanson (1983) the crucial term $J_\Omega/(dV)^2$ appears as J_Ω/dV - a typographical mistake, probably, although the author appears to carry on with the mistake in the discussion following his derivation, and, like Prewett (1984), he does not appear to appreciate the existence of an optimum operating current.

LMIS as ion microprobes

Ions have been used extensively in recent years in a variety of applications, and the development of LMIS as sources of high brightness, with sub-micron spot sizes, has added impetus to this trend. As a result, scanning ion beam microscopy has been greatly stimulated as a complementary technique to scanning electron microscopy. In addition, very large scale integrated circuits pose many challenges for scanning electron microscopy. Thus, LMIS have

opened up new possibilities in ion beam lithography (IBL) , ion implantation, mask repair and scanning ion microscopy (SIM), together with secondary mass spectrometry (SIMS). Moreover, a recently reported technique for the fabrication of U.V. photomasks by ion implantation (Stangl et al 1983) emphasises the great potential of LMIS and of FIB systems in general.

Considerable progress has been made in IBL in recent years. Ga^+ and Si^+ beams (Seliger et al 1979, Cleaver et al 1983a,b) have been used in ion-optical columns employing electrostatic lenses and a magnetic filter (Cleaver et al 1983b) to produce submicron-line detail (see also Kurihara 1985, Kato et al 1985, Yamaguchi et al 1985, Arimoto et al 1985, Hamadeh et al 1985). Very recently, features as small as 20 nm have been produced using 30 keV protons (Adesida et al 1985). The magnetic filter is employed in order to select from the beam the desired ionic species for the target. Thus, for example, a silicon LMIS, produces a beam that consists mainly of Si^{++} , with a comparable proportion of Si^+ and substantial amounts of silicon cluster ions (Prewett et al 1983). Some mass separating systems, however, are ineffective against feebly, charged microparticles, or droplets, and atoms (see above) that travel quite near to the axis (Thompson and von Engel 1982, Wagner et al 1981, Papadopoulos et al 1984). It appears that one very effective way of eliminating these unwanted species - the only way in the case of atoms - is by bending the incident ion beam, and this is exactly what the magnetic filter of Cleaver et al (1983b) does. The advantage of ions, over electrons, in IBL and implantation comes from the reduction of the so-called "proximity effect". This effect arises from scattering of the beam particles inside the target, or resist, and results in deterioration of the line profile and hence control of minimum spacing between adjacent lines (Phang 1979). As seen in Fig 9, "proximity effects" are much reduced with ions - although, with simple LMIS systems, the quasi-Gaussian shape of the beam will annul, to a certain extent, this advantage, and, hence, as in electron beam lithography, some beam-shaping will be necessary. In addition,"ion beam writing", and this refers to both IBL and implantation, has serious limitations with regard to writing speed and hence production time requirements. In fact, the technique appears, at least in its present form, not to lend itself readily for large scale production (see Prewett 1984).

Apart from mask defect correction (Wagner 1983, Brown and Wagner 1983, Prewett 1984, Heard et al 1985) that holds encouraging prospects for LMIS systems, SIM and SIMS appear to be the areas where LMIS are being used more and more with impressive results.

It was in 1980 that Prewett and Jefferies (1980) reported on the use of a 6 kV LMIS Ga^+ beam for analytical microscopy. Various groups have since been involved in the subject, and focused ion beam (FIB) systems with voltages exceeding 50 kV have been operated at high lateral resolution (50 nm) (see, e.g., Levi-Setti et al 1983a, 1984b). Lower energy microprobes, in the 10 kV range, have higher surface sensitivity, but poorer resolution (<1 μm) (Waugh et al 1984a).

The advantage of LMIS lies in their higher ion-optical brightness (current density per unit solid angle) compared to that of the traditional gas sources such as the Duoplasmatron. In Fig.10. the characteristics of LMIS and the Duoplasmatron are compared. It is clear from this figure that for low probe sizes the Duoplasmatron is very inferior to LMIS. Currently, spot sizes around 50 nm are readily produced using LMIS (see, for example, Levi-Setti et al 1984b, Waugh et al 1984b). In a closely related field the fine imaging capability and greater ease of controlling spatial position of LMIS-based FIB systems for in-situ ion milling and controlled specimen preparation has been demonstrated (Waugh et al 1984b). These properties make possible applications such as depth profiling and trace element analysis of implanted species (Prewett 1984), while caesium LMIS for negative SIMS (Fig 11) offers the prospect of analysis of arsenic-implanted regions (Prewett et al 1984).

The value of high resolution SIMS with LMIS is greatly increased if the various images, e.g. the ion induced secondary electron image and the associated SIMS scanning images for various elements can be precisely correlated. This can be done most expeditiously by means of a frame-store, a facility now becoming more common in SEM and TEM instruments. Figure 12 shows a high resolution secondary electron micrograph of a recording head device with 2 μm tracks. This image can be accumulated and saved in the frame store and subsequently overlayed, with high spatial accuracy, by other image information from the SIMS signal. A further advantage of this technique is that no image information is lost and the state of the surface can be monitored and recorded as it inevitably alters under the ion bombardment.

The Chicago group, whose initial - and very considerable - work concerned a detailed examination of image contrast studies with LMIS FIB systems (Levi-Setti, 1983; Levi-Setti et al 1982; 1983a,b; 1984a), have recently reported on SIMS imaging microanalysis, and have obtained high quality elemental maps with a lateral resolution of 40 nm using a 40 kV Ga^+ probe (Levi-Setti et al 1985). Progress with imaging of biological samples has been made recently using gallium LMIS (Levi-Setti et al 1983a, Waugh et al 1984a), where the implanted gallium appears to remove, in some cases, the necessity for coating with conductive material; indeed, this can also be the case with insulating materials in general. The danger of unwanted artifacts arising from precipitation of insoluble material when excessive doses are applied has already been pointed out in the literature (Ishitani et al 1981, Moore and Prewett 1984). In one instance the existence of micrometre size droplets on a silicon target was reported (Gnasser et al 1982) - although, admittedly, this observation was made with a capillary type LMIS, where the large liquid cone might be more susceptible to break-up. It is quite obvious that care should be taken to

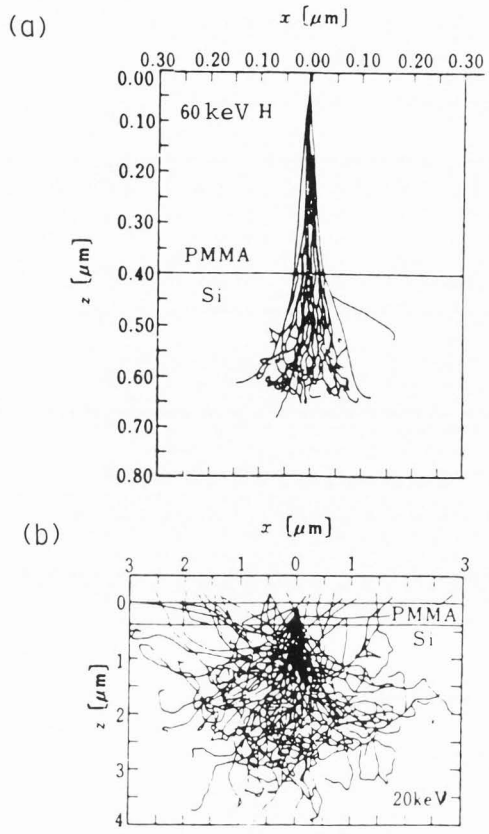


Fig. 9 Comparison of trajectories of 20 keV electrons and 60 keV H^+ ions in 0.4 μm thick PMMA on Si substrate (after Gamo and Namba 1984).

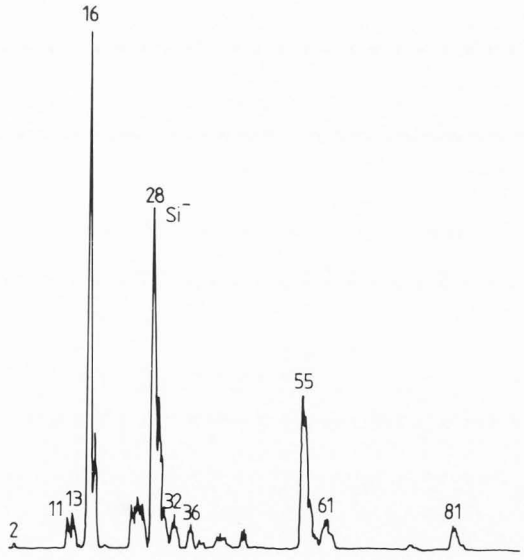


Fig. 11 Negative ions SIMS spectrum of Si target using caesium LMIS (Prewett 1984).

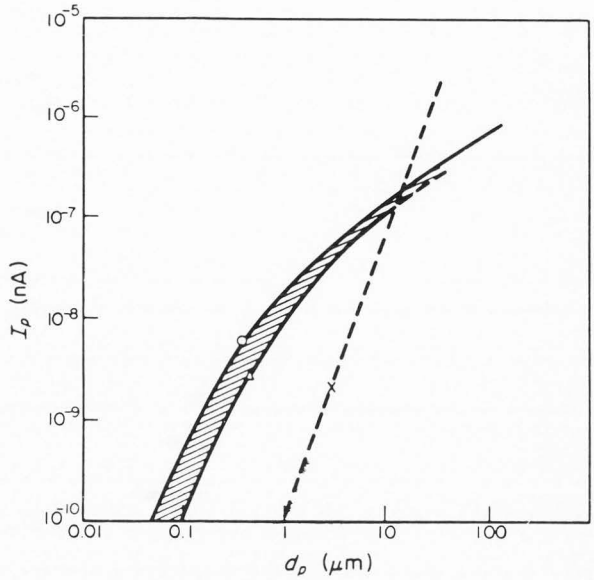


Fig. 10 Typical probe current as a function of spot size for a Duoplasmatron (dashed curve) and for LMIS.

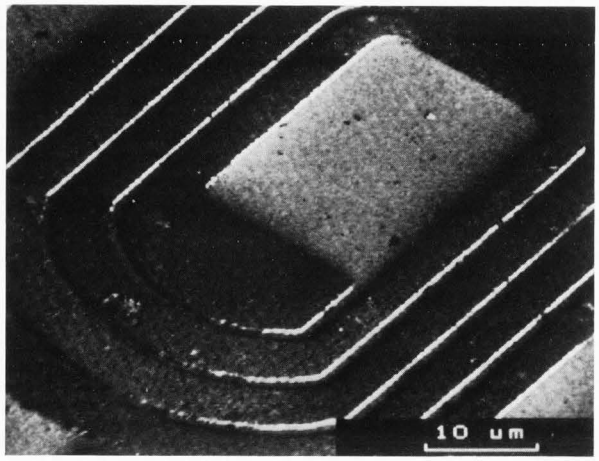


Fig. 12 High resolution LMIS microanalysis of a recording head device with 2 μm tracks. Ion-induced secondary electron image held in frame-store prior to precise overlay of SIMS data. Micrograph by courtesy of VG Scientific.

prevent, or at least minimise, unwanted material, in the form of microparticles, droplets, etc., reaching the sample. Ways of doing this might include "anchoring" a small liquid cone on a relatively sharp needle, which might also help because it would reduce the flow impedance of the needle (Bartoli et al 1982) , and/or the use of beam deflection filter systems. Operation at low currents, apart from other advantages already mentioned, also reduces the proportion of the non-ionic emission (see above).

Another problem that can hardly be avoided in LMIS is the lateral movement of the jet at the

apex of the liquid cone (Aitken 1976, Clampitt and Jefferies 1978, Gaubi et al 1982, Benassayag and Sudraud 1984), which in turn can enlarge the apparent source size. Theoretical analyses of electrified cylindrical jets indicate that both axially symmetric ("varicose") instabilities,which lead to break-up of the jet, and unsymmetric ones, which cause lateral movement of the jet, are possible (Taylor 1969).

Conclusion

Liquid metal sources have already earned a place for themselves in the field of high resolution SIMS. Further technical development is needed in order to extend the available range of suitable ions. The use of frame-stores and other image-gathering systems developed for electron microscopy will greatly facilitate the efficient handling of the large amount of data generated by ion probes of 50 nm or less in diameter. The sensitivity of LMIS & SIMS is already higher than that of other analytical techniques, and further improvements can be expected.

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Discussion with Reviewers

M.J. Bozack: In the discussion related to Figure 4(b), which shows the dependence of energy spread vs current for a Ga LMIS, you argue for the existence of field evaporation by extrapolation of data over three orders of magnitude. How do you justify such a gross extrapolation? Does not data exist in this region? What about observations from other ion sources which suggest that the curves approach 5 eV at low currents?

Authors: The observation in question is simply an interesting one, although some unfortunate phrasing in the latter part of the discussion may have given the wrong impression; we have slightly rephrased this sentence now. It is worth noting, however, that the statistics involved in the curve fitting and extrapolation is quite good, with over forty experimental points used in the current range of ~ 0.5 - 2 μA. Incidentally, the extrapolation may not be quite over three orders of magnitude, given that the result of Culbertson et al corresponds to a value of current somewhere between 10^{-9} - 10^{-8} A and our lowest value of current was about 5 x 10^{-7} A.

There can be no data below about 0.5 μA (or, possibly, somewhat less) with "conventional" LMIS, for this is the lowest obtainable current before the liquid cone collapses. Experiments in the current range you are interested in would have to be carried out with liquid-metal-film-covered needles that are sharp enough to prevent sudden cone formation; in such a case, "jumps" between emission regimes would not occur, leading, at a sharply defined onset, to orders of magnitude increase in the current (see Aitken and Mair 1980, Clampitt 1981).

We have not seen any published information on any LMIS, other than Ga, whose curves "approach" 5 eV - or any other value for that matter.

M.J. Bozack: Has anyone systematically studied mass loss in a LMIS as a function of source construction and surface roughness?

Authors: No, not to our knowledge. We understand, however, that some experiments are being carried out at the moment by Mr. S. Papadopoulos, who is presently attached to Bell Laboratories from Oxford University.

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