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PERFORMANCE OF AN ENERGY COMPENSATOR WITH A LARGE ACCEPTANCE ANGLE

H.O. ANDREN

*Department of Physics, Chalmers University of Technology,
S-412 96 Göteborg, Sweden*

Résumé – Un compensateur en énergie de type Poschenrieder pour une sonde à atomes est décrit. L'angle d'acceptance du compensateur est grand, 0.1 radians. La résolution en masse a été déterminée pour des angles d'entrance et des déficits d'énergie ionique différents, et est comparée à la résolution en masse calculée des erreurs de focalisation du compensateur.

Abstract – An energy compensator of the Poschenrieder type for an atom probe field ion microscope is described. The compensator has a large acceptance angle, 0.1 radians. The mass resolution has been measured for different ion entrance angles and energy deficits, and is compared to the mass resolution calculated from the focusing errors of the compensator.

I – INTRODUCTION

In the atom probe field ion microscope, the mass of individual field evaporated ions is determined by time-of-flight spectrometry. The mass-to-charge ratio of an ion, m/n , is computed from the measured flight time, t , and the known acceleration voltage, U , according to the formula:

$$m/n = 2eU t^2 / l^2 \quad \dots(1)$$

where e is the elementary charge and l the flight distance. In the original and still most used version of the instrument, ions are field evaporated by applying voltage pulses to the specimen [1]. However, it was soon found that the mass resolution of the instrument was much poorer than could be expected from the uncertainties in measurement of U and t [2, 3]. In particular many ions appeared in mass spectra at a too high mass-to-charge value. The resulting tails towards the high mass end of the mass peaks were attributed to energy deficits: many ions were not field evaporated at the pulse peak, and many ions were also accelerated by a falling field [4, 5].

The work of Poschenrieder on time focusing mass spectrometers [6] made it possible to compensate for the energy deficits while retaining geometrical focusing onto the ion detector. The first atom probe with an electrostatic energy compensator of the Poschenrieder type was built by Müller and co-workers, and they obtained a very much improved mass resolution [2]. Since then several energy compensators of the same type have successfully been added to voltage pulsed atom probe instruments.

In Poschenrieder's original publication a numerical example of a compensator design is given. The acceptance angle is 0.04 radians in this example, and it appears that many compensators that have been added to atom probe instruments during the last ten years have had this geometry. Although giving a good mass resolution, this design has a somewhat limited acceptance angle which is a drawback for certain types of analyses, e. g. thin layers.

This paper describes the design and performance of a Poschenrieder energy compensator with a larger acceptance angle, 0.1 radians. The mass resolution has been measured as a function of entrance angle and energy deficit, and the results are compared with calculated time focusing errors.

II – COMPENSATOR DESIGN

The compensator consists of a linear drift path of length g , an electrostatic sector field produced by two toroidal electrodes of deflection angle Φ , radial radius r and axial radius R of the central equipotential surface, and a second

linear path, also of length g (Fig. 1). In order to achieve time focusing and stigmatic imaging, the following relations must be fulfilled:

$$g = 2.33r; \quad c = r/R = 0.249; \quad \Phi = 163.1^\circ \quad \dots(2)$$

[7] – note the numerical error in [6]. For the present design a radius r of 210 mm was selected, which gives a total flight path l of $2g + \Phi r\pi/180$ or about 1570 mm. The electrode separation is 70 mm and the entrance aperture is ϕ 50 mm, which corresponds to a (full) acceptance angle of slightly more than 0.1 radians. The exit aperture is 70 mm wide.

The electrodes have a height of 120 mm and a thickness of about 10 mm, and they were manufactured in the following way: A band was cut out from a 12 mm thick stainless steel plate, bent and welded to a ring. After annealing at 900°C the ring was rough-turned, annealed again at 500°C, and turned to exact measure. The change in shape when cut open was small, accurately measured and accounted for upon mounting. The two electrodes are supported by precision machined glass-ceramic cylinders, which define the position of the electrode surfaces relative to a supporting steel plate with an accuracy of about 0.1 mm.

A simple numerical calculation solving Laplace's equation for the potential between the electrodes (assuming for simplicity $R = \infty$) showed that fringe effects above and below the flight path have to be corrected for (Figure 2). Six compensating wires of ϕ 2 mm stainless steel were therefore mounted, 1 cm apart, between the electrodes at their upper and lower edges. The wires are held by glass-ceramic supports, and care was taken to shield as much of the glass-ceramic as possible in order to prevent charging up effects. With this arrangement the potential deviates less than 1 % from the intended value within a volume of ϕ 50 mm around the central path (Fig. 2).

The potential in the toroidal field between the electrodes can be approximated by the following equation [8]:

$$\varphi(u,v) = 2U_0 [u - (1+c)u^2/2 + cv^2/2] \quad \dots(3)$$

where c is defined in Eq. 2, $u = (\rho - r)/r$ is the normalised radial coordinate and $v = z/r$ is the normalised axial coordinate, measured from the plane of the central path. U_0 is the accelerating voltage, which means that ions which do not suffer any energy deficits will have an energy of neU_0 . With this equation the required voltages on the electrodes and compensating wires can be calculated. The large separation between the electrodes means that rather high voltages have to be used, $-0.37U_0$ and $+0.30U_0$ on the inner and outer electrodes, respectively, with correspondingly higher requirements on high voltage supplies, feedthroughs and insulation.

The energy compensator was mounted onto an atom probe instrument which has been described elsewhere [9]. A laser beam was used as an aid during mechanical alignment of the ion optical axis of the compensator along the geometrical axis of the instrument (defined by the movement of the probe hole as the tip-to-channel-plate distance is changed).

TABLE I - Measured width of two Ni^{2+} mass peaks in atom probe spectra. Five different combinations of tip-to-screen distance and pulse amplitude were used.

Column	1	2	3	4	5	6	7	8
Experiment number	Tip-to-screen (cm)	Pulse fraction (%)	Peak width at base Ni-58 (u)	Ni-60 (u)	Width at half maximum Ni-58 (u)	Ni-60 (u)	"65% of ions" width Ni-58 (u)	Ni-60 (u)
1	11.5	15	.17	.15	.03	.03	.03	.03
2	8	15	.15	.17	.06	.06	.04	.04
3	4	15	.19	.18	.01	.08	.07	.07
4	2	15	.24	.25	.01	.08	.07	.07
5	8	29	.26	.26	.01	.01	.04	.06

III - RESULTS

Experiments were performed with a specimen of pure nickel. The specimen temperature was 90 K, the pressure less than 30 nPa, the evaporation voltage about 8 kV, and the evaporation pulse amplitude 15% of the standing voltage. The entrance angle to the compensator was varied between 17 and 100 mrad by varying the distance between the channel-plate image intensifier, with its $\varnothing$ 2 mm aperture, and the specimen from 11.5 to 2 cm. In addition, with an entrance angle of 25 mrad a pulse amplitude of 29% was used.

For each set of parameters about 1200 ions were recorded. Examples of mass spectra obtained are shown in Figure 3. The width of the two largest Ni^{2+} mass peaks, from Ni-58 and Ni-60, was determined from the spectra both at half maximum and at the base. The results are given in Table I. As can be seen from the Table the peak width at half maximum is not a good measure of the shape of mass peaks, since due to the limited resolution of the timer this width can vary considerably if most ions "hit" one timer channel or if most ions are distributed evenly over two channels. In addition therefore the width of that part of each peak, around the peak maximum, in which 65% of the ions occurred was determined and is given in Table I ("65% of ions").

As a comparison a spectrum of pure nickel, obtained with a straight atom probe using similar conditions (15% pulse fraction and 67 mrad entrance angle), is shown in Figure 4.

IV - CALCULATION

Measurement errors

Differentiating Eq. 1 we obtain for the error in mass-to-charge ratio:

$$\Delta(m/n)/(m/n) = 2 \Delta t/t + \Delta U/U \quad \dots(4)$$

Neglecting for the moment the focusing errors of the compensator, the error Δt in Eq. 4 is simply the uncertainty in time measurement of the spectrometer, Δt_m . In the present case the time is measured with a 200 MHz timer. Therefore the timing error consists of the sum of the timer start error and the timer stop error. Both are stochastic variables with constant probability, the former over the interval (-5, 0) ns and the latter over the interval (0, 5) ns. The standard deviation of such a variable is $\sqrt{(25/12)}$ or 1.44 ns, so that the sum has the standard deviation $\sqrt{(25/6)}$ or 2.04 ns.

In the present case the flight time, t , is about 6.8 μs . The maximum timer error, Δt_m , is of course ± 5 ns or about $\pm 0.7\%$, which gives a maximum measurement error $\Delta(m/n)_m$ of ± 0.043 u (atomic mass units). In comparison with the timer error, the error $\Delta U/U$ due to the uncertainty in voltage determination is small and will be neglected in the following. The full maximum measurement error is therefore $\Delta(m/n)_m$, and this is entered in Table II, where we shall collect calculated *maximum* errors. We will then compare these with measured widths at the *base* of the mass peaks.

The standard deviation, 2 ns or about 0.3 %, corresponds to a standard deviation for m/n of $\sigma_m = 0.017$ u. It can be shown that for the sum of two stochastic variables of constant probability over an interval, 65% of the ions occur within $\pm$ one standard deviation. We enter σ_m in Table III, where we shall collect calculated "65% of ions" errors. These will then be compared with measured *half maximum* and "65% of ions" peak widths.

TABLE II - Calculated maximum measurement errors and focusing errors for the experimental conditions in Table I

Experi- ment	$2\alpha = 2\beta$ (mrad)	δ (%)	$\Delta(m/n)_m$ (u)	$\Delta(m/n)_\alpha$ (u)	$\Delta(m/n)_\beta$ (u)	$\Delta(m/n)_\delta$ (u)	$\Delta(m/n)_{\alpha\delta}$ (u)	$\Delta(m/n)_{tot}$ (u)
1	17	41	± 0.043	-0.001	+0.002	+0.043	± 0.001	$-0.05 \pm .09 = .14$
2	25	41	± 0.043	-0.002	+0.005	+0.043	± 0.001	$-0.05 \pm .09 = .14$
3	50	41	± 0.043	-0.007	+0.019	+0.043	± 0.002	$-0.05 \pm .11 = .16$
4	100	41	± 0.043	-0.030	+0.076	+0.043	± 0.004	$-0.08 \pm .17 = .25$
5	25	79	± 0.043	-0.002	+0.005	+0.157	± 0.002	$-0.05 \pm .21 = .26$

Focusing errors

Maximum errors

The second order approximation for the electric field in the toroidal compensator is given by Eq. 3 above. Using this approximation it is possible to compute the flight time of an ion of energy $U_o(1+\delta)$, (where $\delta = \Delta U/U_o$ is the energy deficit), radial entrance angle α and axial entrance angle β , to second order in α , β , δ and ion source width u_o and v_o [10]. (The compensator is free from time focusing errors to first order in these quantities.) Neglecting terms in u_o and v_o , which are very small for a field ion specimen, we have (Eq. 11 in [10]):

$$\Delta t = (1/v_g) [\alpha^2 F + \alpha \delta G + \delta^2 H + \beta^2 L] \quad \dots(5)$$

where v_g is the velocity of an ion of energy U_o , and F , G , H and L are constants. With the numerical values given in [10] we obtain, independent of flight distance and electrode separation:

$$\Delta(m/n) = (m/n) [-0.407 \alpha^2 \pm 0.069 \alpha \delta + 1.05 \beta^2 + 0.865 \delta^2] \quad \dots(6)$$

It is straightforward to calculate $\Delta(m/n)_\alpha$ and $\Delta(m/n)_\beta$ from the above formula and the known entrance angles α and β . The results are given in Table II. In order to calculate $\Delta(m/n)_\delta$ and $\Delta(m/n)_{\alpha\delta}$, δ must be estimated. From the spectrum in Fig. 4, obtained without any energy compensator, it can be deduced (by extrapolation of the approximately exponential peak tail) that the mass peak Ni-58 would have a width at the base of about 1.2 u for our particular experiment conditions and 15% pulse fraction, if no energy compensation were made. This gives a value for δ of 41 % at 15% pulse and—assuming that δ increases linearly with pulse fraction—of 79% at 29% pulse. The expected $\Delta(m/n)_\delta$ and $\Delta(m/n)_{\alpha\delta}$ when using a compensator can then be calculated from Eq. 6, and they are given in Table II together with $\Delta(m/n)_{max}$, the sum of all maximum errors computed.

"65% of ions" width

For the case of *measurement* errors it was possible to compute the "65% of ions" width of the mass peaks. This quantity was also measured. Let us therefore also estimate the "65% of ions" width caused by the *focusing* errors because of the spread in entrance angle of the ions and their energy deficits.

Since ions having a large radial entrance angle α have a shorter than normal flight time, and ions with a large axial entrance angle β have a longer than normal flight time, ions originating from two diagonals of the probed circular area suffer no flight time error (Figure 5). The loci of equal time error are hyperbolas, and by calculating the area between these the fraction of ions which have less than a certain $\Delta(m/n)$ can be computed. The $\Delta(m/n)$ corresponding to 65% of the probed area for some of the experimental conditions are given in Table III.

For 15% pulse fraction, the energy deficit which 65% of the ions do not exceed can easily be deduced from the spectrum in Fig. 4, and the corresponding $\Delta(m/n)$ computed from Eq. 6 is given in Table III together with an estimated value for 29% pulse, again assuming a linear increase in energy deficit.

TABLE III - Calculated width of mass peak within which 65% of the ions occur. The calculation was made for three of the experiments in Table I. The dominating contribution to the peak width is underlined.

Experiment	$2\alpha = 2\beta$ (mrad)	δ (%)	Calculated "65% of ions" width (u) due to: measurement ($2\sigma_m$) α and β δ
2	12.5	41	<u>.04</u> .002 .001
4	100	41	<u>.04</u> <u>.06</u> .001
5	12.5	79	<u>.04</u> .002 .003

V — COMPARISON WITH MEASUREMENTS

Full peak width

The calculated total maximum errors, i.e. the sum (with proper signs) of maximum measurement errors and focusing errors, are given in the last column of Table II. These should be compared with the measured widths at the base (full peak width) of mass peaks Ni-58 and Ni-60, columns 3 and 4 in Table I. The agreement is good. It can be noted that the timer resolution and the energy deficits limit the mass resolution, measured at the peak base, to about 1/180 at small entrance angles and energy deficits, whereas a large entrance angle or energy deficit gives a mass resolution of about 1/115.

"65% of ions" peak width

The mass resolution measured as peak width at half maximum is of course much higher. However, the simple width at half maximum is not a good measurement of the peak shape, as is evident from columns 5 and 6 in Table I. As an example, deducing a mass resolution of $.01/29 = 1/2900$ from the Ni-58 peak at large entrance angle or energy deficit would be meaningless. But the "65% of ions" width seems to be a reproducible measure, which then should be compared with the calculated errors in Table III. It is not a straightforward procedure to add these errors, but the dominating error in each of the three cases in Table III (underlined) seems to agree quite well with the measured "65% of ions" width in the last two columns of Table I. The "65% of ions" widths correspond to a mass resolution of between about 1/410 and 1/960, and are apparently mainly caused by timing errors, except at large entrance angles.

VI — SUMMARY

1. An energy compensator with a large acceptance angle, 0.1 radians, was constructed. From a design point of view the major difference relative to a smaller acceptance angle compensator is that higher voltages have to be handled.

2. The full peak width (peak width at its base) was measured for different entrance angles and energy deficits, and compared with calculated maximum measurement errors and focusing errors. The agreement was good. For Ni at 8 kV, using a timer with 5 ns resolution and a flight path of 1.6 m, the full peak width corresponds to a mass resolution of between 1/115 and 1/180 for the different cases.

3. To estimate the peak width at about half maximum, the mass range within which 65% of the ions occurred was found to be a suitable quantity. Again the agreement between measured "65% of ions" widths and calculated errors was good. The mass resolution measured in this way varied between 1/410 and 1/960.

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