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Filament temperature stabilizer for a thermal ionization mass spectrometer

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Abstract

A major enhancement in temperature stabilization of a filament has recently been achieved by means of the Halas–Kamiński bridge, in which the reference resistance of one leg is directly proportional to the filament temperature. In this article we describe a novel version of this type of electronic circuit which feeds dc power voltage to the filament via a switching transistor. The switching frequency is regulated by means of a triangular waveform generator incorporated into the system. The duration of the heating peak (square wave) is automatically adjusted properly once at each break between two subsequently appearing heating peaks, which is the interval when the bridge imbalance signal is measured. At the end of each break the bridge imbalance signal is kept on the output of the sample-and-hold amplifier. This signal compared with the generator output voltage provides the square wave for the switching transistor. The application of this circuit for a thermal ionization mass spectrometry is simple and straightforward. The filament temperature is set by two decade low-ohm resistors. Long-term variance of the ion current is approximately 5 times lower than that obtained by use of a commercial voltage stabilizer for the filament supply. It is shown theoretically that stabilization of the filament resistance results in the lowest possible variance of temperature. (Int J Mass Spectrom 181 (1998) 167–171) © 1998 Elsevier Science B.V.

Keywords: Ion source; Filament temperature; Mass spectrometry; Thermal ionization; Temperature stabilization; Surface ionization

1. Introduction

In thermal ionization mass spectrometry the ions are produced on the surface of a hot metal with high melting point (usually W, Ta, Re, Ir, Pt). The current density which can be drawn from the surface at equilibrium conditions is determined by the well-known Saha–Langmuir formula [1,2]:

$$j^+ = \frac{\nu}{1 + \frac{g^0}{g^+} \exp \frac{(E_i - \varphi)}{kT}} \quad (1)$$

where ν is the flux density of particles supplied to the surface, g^+ and g^0 are statistical weights of ground state of ion and neutral particles, (for alkali metals the ratio g^+/g^0 is equal to 1/2), respectively, E_i is the ionization energy of the particle, φ is the work function of the metal. Formula (1) is strictly valid only when the surface coverage is small.

Inasmuch as both the numerator and denominator in formula (1) are strongly temperature dependent, a precise filament temperature stabilization is greatly needed for the production of stable ion beams by the surface ionization process.

In the case of a single filament ion source the flux density, ν, is driven by the surface diffusion from the

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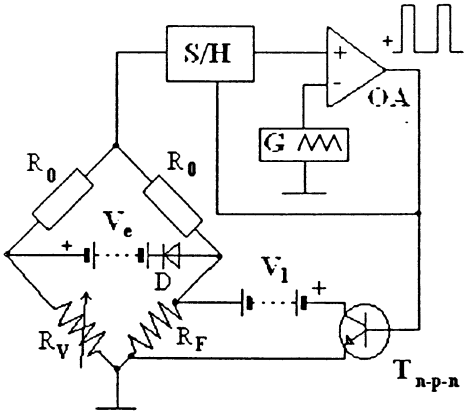

Fig. 1. Conceptual diagram of using the Halas–Kaminski bridge for constant-resistance operation of a filament (R_F). S/H is a sample-and-hold amplifier, G is a triangular wave generator. Note that $R_0 \gg R_V \approx R_F$ to avoid power dissipation from V_e source.

cooler ends of the filament toward its center. The filament center should be kept at a temperature where the analyzed atoms can be effectively ionized, which means that the surface coverage is far below the monolayer at the filament center. This condition is fulfilled for potassium at temperatures between 1000 and 1500 K.

It is the purpose of this article to describe a novel temperature stabilizer of a filament of the surface ionization source. The ion source is applied in our laboratory for potassium content determination by the isotope dilution technique [3].

2. Stabilizer arrangement

The stabilizer design is based on the well proven idea of the Halas–Kaminski (HK) bridge [4–7]. A conceptual diagram of the stabilizer is shown in Fig. 1. The HK bridge contains a dc excitation voltage source (V_e). The excitation current passes through a diode and the bridge resistors but not through the transistor T. R_F and R_V denote, respectively, the resistance of the filament and a variable resistor used for temperature setting. We will assume that the filament is made of a pure metal like W, Ta, Re, Pt, which has positive and significantly large temperature coefficient of electrical resistivity.

If $R_V \leq R_F$ then the bridge signal supplied to a noninverting sample-and-hold amplifier (S/H) is negative or zero. In this stage the transistor does not conduct because its base is kept at a negative potential of the operational amplifier (OA) output. The noninverting input of OA is connected to the output of S/H amplifier whilst the inverting input of OA is fed by a positive signal from a triangular wave form generator (G).

In the reverse situation, i.e. $R_V \geq R_F$ the imbalance signal generated by the bridge is positive and has the magnitude:

$$V_{\text{in}} = \frac{V_e - 0.7\ \text{V}}{4} \frac{R_V - R_F}{R_V} \tag{2}$$

This signal is amplified by S/H which is held at the output when the negative potential taken from the output of OA turns to positive. The comparator OA produces a positive output voltage if the generator signal is below the voltage held by S/H.

In this fraction of the cycle transistor T conducts. Hence the dc power voltage supply, V_1, is connected in parallel to R_F (through T which plays the role of a switch). Note that no significant current from V_1 can pass through the remaining parts of the bridge due to the presence of the diode D. Because V_1 considerably exceeds V_e, the excitation current cannot pass through the low-resistance legs of the bridge (R_V and R_F) when R_F is supplied from V_1. This is the reason for using a sample-and-hold amplifier instead of a normal operational amplifier.

As a result of this action of the circuit (shown in Fig. 1) the series of heating pulses is supplied to R_F with frequency driven by the generator but their duration is driven by the output voltage of the S/H amplifier. The bridge is always kept close to the balance state, i.e. $R_F = R_F$. If for some reason (e.g. voltage V_1 starts to diminish) R_F resistance becomes somewhat lower than R_V, then the bridge imbalance signal becomes somewhat higher, which results in a longer duration of the heating pulses. Note that part of the circuit presented in Fig. 1 comprising the comparator OA and generator G is a typical solution of a pulse-width-modulator (cf. [8]).

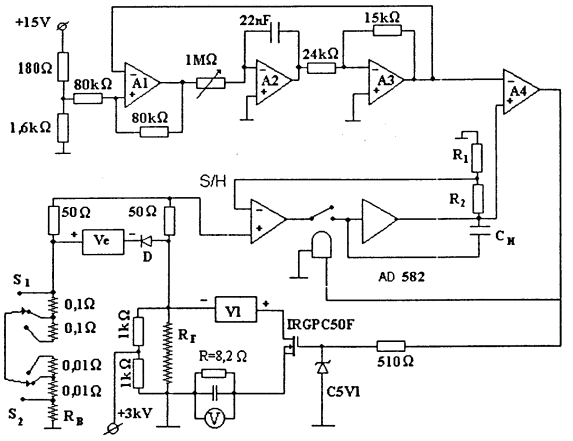

Fig. 2. Schematic diagram of filament temperature stabilizer. A_1–A_4 are operational amplifiers comprised in LM321, C_H = 1 μF, R_1 = 4 kΩ, R_2 = 800 kΩ, V_e is a dc power supply (5 V, 5 A), V_1 is a dc power supply (10 V, 5 A). R_B is a piece of manganin wire with resistance of R_F at room temperature.

3. Circuit details

Most of the circuit details are shown in Fig. 2. The amplifiers A_1 to A_3 comprises a typical generator of a triangular waveform (cf. [8]). The amplifier A_4 is used as a comparator, while AD582 as a noninverting sample-and-hold amplifier. Its amplification, $1 + R_1/R_2$, may be varied from 100 to 200 000. The variable resistor R_V comprises a pair of decade switches with resistors made of manganin wire ϕ0.5 mm. With the resistances shown in Fig. 2 the filament temperature can be set from room temperature to maximum possible (considering melting point) in 100 nearly equal steps (i.e. about 20 K/step).

The filament in our thermoemission ion source has two pairs of leads—one pair for V_1 power supply whilst the other for the bridge connection and high voltage supply to the filament. Those leads are connected to the four filament ports in the ion source flange.

A power metal oxide semiconductor field effect transistor (MOSFET) type IRGPC50F was used as the switching transistor. The gate this transistor is protected by is a diode C5V1. In selection of the power supplies, V_e and V_1, particular care to voltage stability was given in the case of V_e. We applied commercially available switching regulators (L4970) which are small in size and inexpensive.

4. Results

Potassium was loaded on to a rhenium ribbon filament, 0.04 mm × 0.8 mm × 15 mm, as 25% KCl solution in 10% phosphoric acid diluted in distilled water. After air drying and degassing under vacuum, the ribbon was preheated in 800 K. Ion emission was measured at a filament temperature of 1100 K. Comparison of ^{39}K ion currents obtained by use of a MI-1305 mass spectrometer with solid-state ion source is shown in Fig. 3.

As it may be seen from Fig. 3, the stabilization of filament temperature results in lower variance of obtained ion currents, in comparison to filament voltage stabilization. The variance ratio is V_U/V_R = 5.4, where V_U is the variance of the ion current obtained with voltage stabilization, and V_R is the variance of the ion current obtained with temperature stabilization.

The filament length is crucial in obtaining stable ion currents. A temperature plateau exists in case of long filaments. In such a case, the fluxes of ions are emitted from a relatively large surface with fluctuations due to changes in surface coverage at the plateau region. Steady conditions for ionic emission were obtained only for relatively short filaments, for which no temperature plateau exists. Details on defining short and long filaments may be found in [9].

5. Discussion

The detailed solution of the complete differential equation describing the temperature distribution along the metal filament heated in vacuum may be found elsewhere, e.g. [10] or [11], the latter additionally includes the electron cooling effect. However, the variance of the filament temperature may be estimated from a simplified power balance equation, assuming that all the power delivered to the cylindrical filament

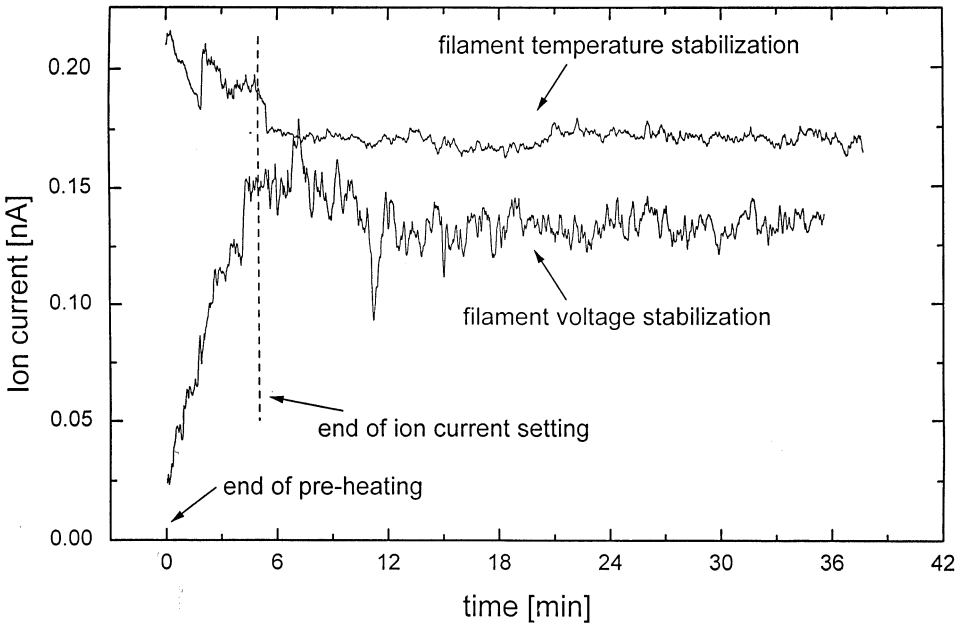

Fig. 3. Comparison of ion ^{39}K currents obtained for filament temperature and filament voltage stabilizations.

element of length dx and resistance R is lost by radiation:

$$P = D\epsilon\sigma T^4\, dx \tag{3}$$

where D is the filament circumference, ε is the total emissivity, σ is the Stefan–Boltzmann constant, and T is the filament temperature. Power $P = I^2 R = U^2/R$ is delivered by the heating current I. Temperature of a filament element dx may now be found from Eq. (3) as a function of either the resistance R, power P, voltage U, or current I, and differentiated to find the temperature variation, dT. The temperature variation due to respective instabilities dR, dP, dU, or dI are

$$dT(R) = \frac{1}{4} CR^{-3/4} I^{1/2} dR \tag{4}$$

$$dT(P) = \frac{1}{4} CP^{-3/4}\, dP \tag{5}$$

$$dT(U) = \frac{1}{2} CR^{-1/4} U^{-1/2}\, dU \tag{6}$$

$$dT(I) = \frac{1}{2} CR^{1/4} I^{-1/2}\, dI \tag{7}$$

where $C = (D\epsilon\sigma dx)^{-1/4}$. The respective variances may be calculated as squares of the above mentioned temperature variations, e.g. $V_R = [dT(R)]^2$. From simple calculations we obtain

$$V_P/V_R = 1 \tag{8}$$

$$V_U/V_R = 4 \tag{9}$$

$$V_I/V_R = 4 \tag{10}$$

From above ratios it may be clearly seen that stabilization of the filament resistance results in 4 times lower temperature variance than stabilization of either filament voltage or current. The theoretical factor 4 is comparable to experimental result equal to 5.4, mentioned in Sec. 4.

Stabilization of filament power is rarely used because of the complication of the electronic circuit that has to stabilize two variables, U and I, simultaneously. The stabilization of the filament current actually yields worse results than stabilization of the filament voltage ($V_I/V_R > 4$), because from the voltage–current characteristics it is known that even in the case of short filaments $dI/dU < 0.5$ [A/V]. This means that the filament itself tends to

stabilize its current, which makes temperature stabilization by use of stable heating currents very difficult, if not impossible. Nevertheless, stabilization of the heating current or voltage is still used in solid-state mass spectrometers.

6. Conclusion

The electronic circuit presented here allows for good temperature stabilization of a filament, by controlling the filament resistance. It was shown that filament resistance stabilization, being much simpler than the filament power stabilization, results in a significantly lower variance of the ion current than in the case of filament voltage or current stabilization.

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