

STUDY OF THE PROPERTIES OF A PLAIN CATHODE GRIMM-TYPE DC GLOW DISCHARGE SOURCE OPERATED IN A CURRENT-CONTROLLED PULSE REGIME

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Glow discharge sources have been used for analytical purpose for several years. Recently the pulsed operation mode has gained an increasing importance. Operating the source in current-controlled mode instead of the traditional voltage-feed method may have the advantage of precise control of the source's state and it provides a convenient way to study the relationship among the emitted line intensity and the key parameters of the plasma such as the pressure of the filling gas, the voltage-drop on the source as well as the current flowing through the device. In the following the brief description of the system, the relationship among the voltage drop on the source, the pressure of the filling gas (Ar), the current of the DC pulses as well as the line intensities are presented and discussed.

Keywords: DC glow discharge, pulsed current-controlled mode, atom-spectrometric source.

Introduction

Glow discharge sources have a great importance in the atom spectrometry. Main fields of their application are the analysis of bulk materials and examination of surface layers, i. e. the depth-profile analysis. Besides that they play an important role in the mass-spectrometry an ion-sources. Beside the devices based upon the direct current glow discharge, radio-frequency operated sources have also been developed, making possible the analysis and study of non-conducting materials [1].

In case of the DC sources a substantial part of the power applied to the device will be dissipated as heat in continuous operating mode, warming up both the equipment and the sample being the cathode. To avoid this warming-up, which may also be harmful to some parts of the source either, a suitable cooling system have to be applied. Another way to avoid harmful effect is to operate the device in pulsed mode. Depending on the pulse parameters, the average power applied to the device in the later case is only a few percentages of value necessary on continuous DC mode. Several studies can be found in the literature dealing with sources operated in pulse regime [2, 3, 4].

In the above-mentioned studies the sources are powered exclusively in the so-called voltage-feed way. It means that the device is connected to the DC power supply directly, - or in order to avoid the damage in case of short-circuited condition, - through a so-called ballast resistor. As a consequence of that any change in the state of the source causes a change both on the voltage dropped on the source and that of the current flowing

through the device. If because of nay reason the impedance of the source might change , so will change again the voltage drop and the current. This might cause the the source to leave either the area of abnormal mode of operation and will enter the area of normal mode or vice versa. On the other hand it is rather difficult, - though not at all impossible, - to provide precise high-voltage pulses to feed the system in this voltage-fed mode.

So that the change of the voltage drop and the current could be avoidedat and a better control of the system could be achieved, we suggest applying a constant pulse current to the system. By using constant current pulses, the state of the system can also be controlled. To provide constant current pulses, a current generator is needed. It has a very high internal impedance, and because of that the change of the source impedance will not have any effect to the value of the current flowing through the source as long as the current generator is able to provide constant current pulses. In this way the voltage drop and the current will remain unchanged, therefore better control can be achieved.

To study the current-controlled mode we have developed a high power current-generator power supply and controller, which is able to operate in pulsed regime. The pulsed regime operation does not mean that current flows through the source only in the duty-cycle period. In the course of the development it was targeted that the source should always operate in the so-called abnormal region, or at least it should be at the border of the abnormal mode. Therefore there is a low-current period and a high-current one. The later is eventually the duty-cycle period. E.g. at a frequency value of

200 Hz and 10% duty cycle, the length of the period is 5 ms, and the duration time of the high-current lasts 0.5 ms, while the low-current one is 4.5 ms long.

The use of the pulsed regime may provide two advantages. The first one is that high power resulting in dissipating much heat and warming up the equipment is applied just for a very short time, so the average power is just a few percentages of it. This makes unnecessary to apply a cooling system, as much less heat is dissipated by the source. The other advantage is that the short-time high-current pulses the system provides make possible to study and examine thin layers.

Experimental

Main components of the system

Main parts of the system can be seen in *Figure 1*. The light emitted by the discharge is detected and measured by a spectrometer. The output signal of the detector is processed by a special computer programmed card, USB 4716 manufactured by the Advantech Co.. The electric DC voltage and current pulses necessary for the operation of the discharge is provided by the power supply and current generator unit.

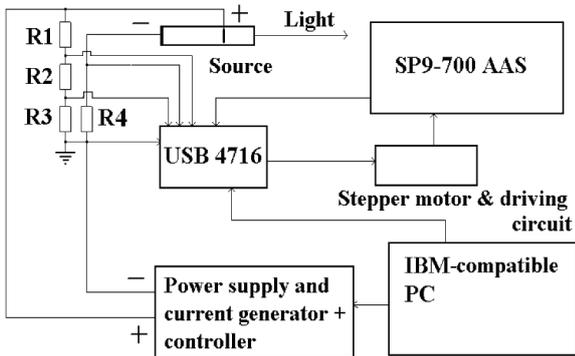


Figure 1: Main components of the system

The source

In the course of the experiments a Grimm-type source (presented in *Figure 2*) was used, it is based upon the design made by Wagatsuma and co-workers [5]. The anode is made of brass, its internal diameter is 7 mm.

The power supply and current generator

This unit was designed and built in our laboratory. It is able to provide a DC voltage as high as 1000 V. It can be operated in the frequency range of 122 Hz–5000 Hz. The duty cycle can be chosen between 32 μ s – 4.1 ms, resulting values ranging from

0.4% up to 50%, depending on the pulse period and frequency. In the experiments reported here the pulse frequency was

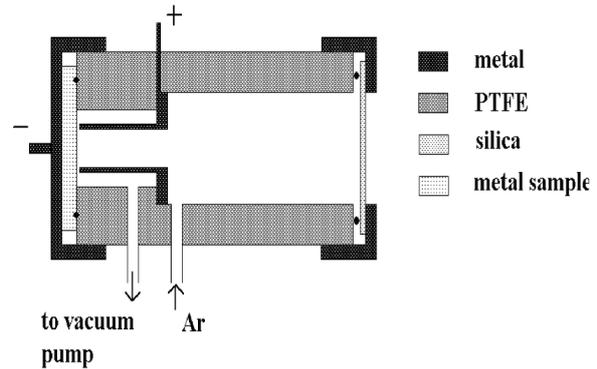


Figure 2: The source used in the experiments.

122 Hz and the duty cycle was 2.3%.

The value of the low current is 1.5 mA, while 5 preadjusted values (32, 72, 115, 135 and 160 mA) can be selected for the high current pulses. The device has its own microcontroller unit, which has a serial (RS 232C) link to the IBM compatible computer controlling the system. This build-up makes possible to choose all the important parameters (value of the frequency, duty cycle and that of the high current) using a special computer programme. The same programme is responsible to control the measuring system and store the measured data.

The spectrometer

To convert the light of the source into an emission spectrum and to convert the light intensity of the selected spectral line to an electric signal an atomic absorption spectrometer (Pye Unicam SP9-700) was used. Only the spectrometer (monochromator) and the detector (PMT) was used during the experiments. The electric signal processed by the external measuring system was taken from the loading resistor of the photomultiplier tube of the instrument. The narrowest slit width (0.2 nm) was chosen for the experimental work. So that spectras could also be recorded, the device was equipped with a wavelength driving circuitry (consisting of a stepper motor and of a control circuit).

The signal measuring system

The measuring system must be capable to measure signals both in the high-current and in the low-current period. To fulfill this requirement a multifunction module capable to provide fast analogue to digital conversion has been chosen. It is the USB 4716 multifunction module manufactured by Advantech Co Ltd. It has a 16-bit AD converter with a 16-channel

multiplexer and its minimum conversion time is 5 μ s. It can operate both in an externally triggered mode and in a so-called software triggered mode. The signal taken from the loading resistor of the photo-multiplier tube (PMT) of the spectrometer is led into the first measuring channel of the device. The electric connection between the loading resistor of the PMT and the signal input of the spectrometer own measuring electronic system was eliminated so that these circuits could not have any influence on the measurements.

So that not only the emitted signal of the source, but the current flowing through on that and the voltage drop could be measured, there is a resistor network in the system. R3 is a resistor of 15 Ohm. It is for measuring of the lamp current. Resistors R1 and R2 (20,000,000 and 120,000 Ohms respectively) form a voltage divider, making possible the measurement of the voltage drop on the source.

The USB 4716 has digital inputs and outputs, therefore it is able to control the stepping motor of the wavelength drive. The unit is controlled by a programme running on an IBM compatible computer.

Other important details of the experiments

In the course of the experiments high purity argon (99,996%) was used as filling gas. A pressure regulator and a vacuum pump made possible to adjust the required value for the pressure of the filling gas. In all the experiments expect for the demonstration of sputtering ability a brass (copper – zinc alloy) probe was applied as the cathode of the source.

Results

As the device operates in pulsed mode, all the signals are eventually pulses. *Figure 3* shows the relative signals of emitted light, discharge current and that of the voltage-drop on the source as the function of time. The different signals are shifted in time so that they could be better observed.

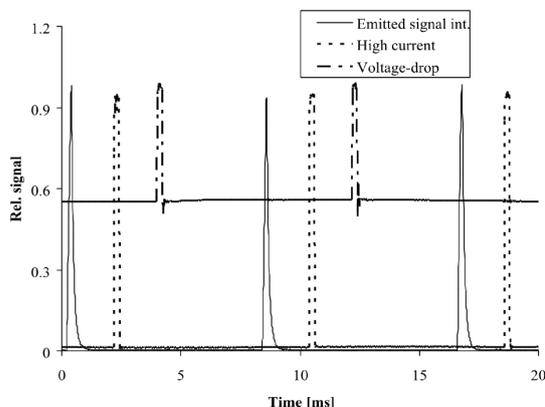


Figure 3: Signals of the emitted light, current and voltage-drop pulses as the function of time

Stability of the current generator

From the view-point of the operation it is important to know how much the changes in the pressure of the filling gas affect the value of the high current, in other words how stable the current generator is. As it can be seen in *Figure 4*, the value of the high current is eventually not affected in the pressure range of 300–950 Pa, but under 300 Pa it is no longer constant, i.e. under the above-written range the current generator no longer works.

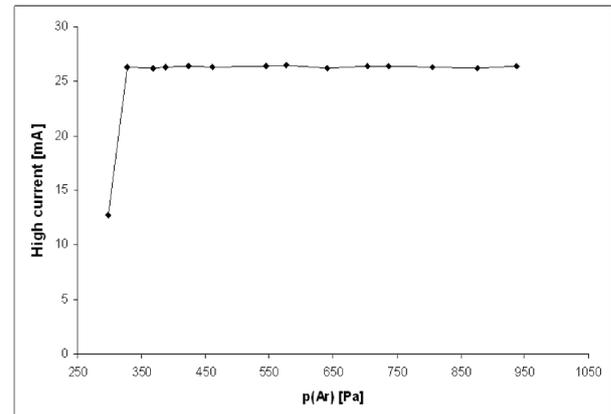


Figure 4: The high-current pulses as the function of the filling gas pressure

The relation among the voltage-drop, the current and that of the filling gas pressure

As the device operates in the abnormal working range, it can be expected that the voltage-drop on the source will increase with increasing current. The relation measured is presented in *Figure 5*, where the voltage-drop on the source can be seen as the function of the current at three argon pressure values. It can be stated that according to the expectations the voltage-drop is greater at higher current, but the increasement is higher at lower argon pressure.

The relation of the voltage-drop to the filling gas pressure measured at 72 mA high current is shown in *Figure 6*. It can be observed that there is only a moderate increasement in the voltage-drop with decreasing argon pressure in the range of 400–900 Pa, while the voltage-drop increases sharply below that range.

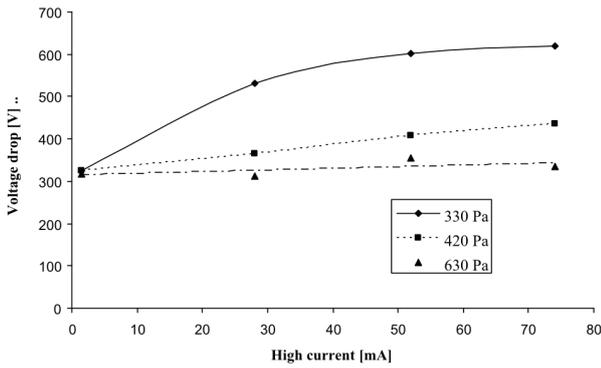


Figure 5: The voltage-drop as the function of the current at 3 different filling gas pressure.

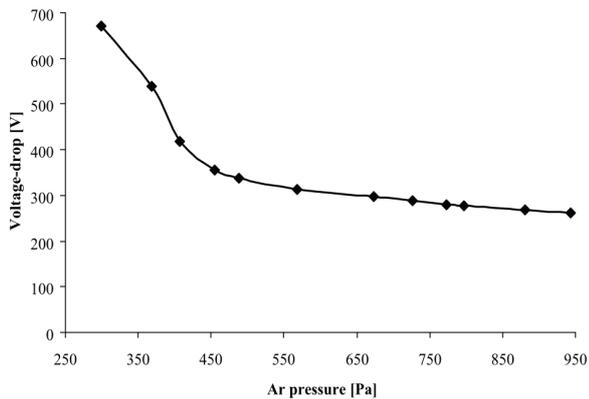


Figure 6: The voltage-drop as the function of the filling gas pressure at 72 mA.

The light intensity emitted by the cathode material

From the viewpoint of the possible practical analytical application the emission by the elements building up the cathode sample has the greatest importance. This emission, i. e. the intensity of the

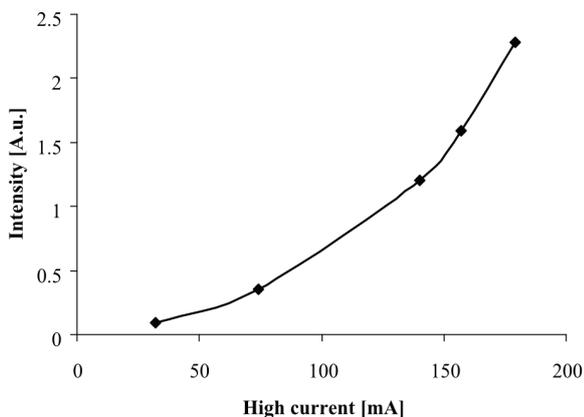


Figure 7: The emitted light intensity measured on the 324.8 nm Cu I. line as the function of the current at 350 Pa.

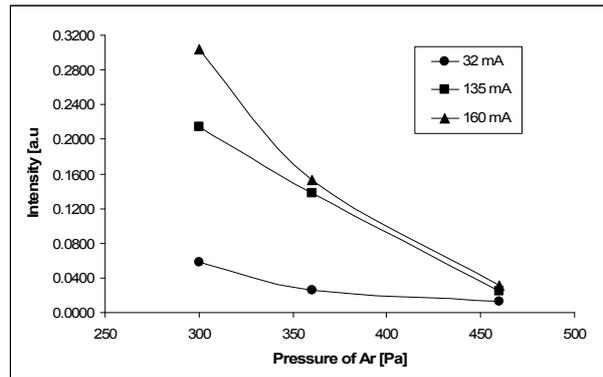


Figure 8: The emitted light intensity measured on the 324.8 nm Cu I. line as the function of the pressure of the filling gas.

emitted light is also the function of the current and the pressure of the filling gas. In the course of the experiments the emission on the strongest atomic line of Cu (324.8 nm) was measured. The results are presented in Figure 7 and Figure 8. Looking at these figures it can be stated that the emitted line intensity sharply increases with increasing pulse current and decreases with increasing filling gas pressure.

The sputtering ability

The atoms of the bulk cathode material can get to the plasma by the mean of sputtering, so this process has a key-importance in the proper operation of the source. Beside that it makes possible the depths profile analysis. In order to get information on the sputtering efficiency the sputtering speed has to be determined, which will be performed in the future. In these experiments only the demonstration of the sputtering ability is presented.

As this source is operated in pulse mode, high power is applied to the source just a few percentages of the pulse duration, therefore rather low sputtering speed can be expected if it is averaged for the whole period. On the other hand it might advantageous in case of thin layers.

To present the sputtering ability a steel plate, which had a $2,0 \pm 0,2 \mu\text{m}$ thick zinc coating on its surface was taken as cathode sample and the zinc emission was measured on the 213.85 nm Zn I line at 350 Pa argon pressure as the function of time. The value of the high current was 72 mA, the pulse frequency was 122 Hz and the duty cycle was 2.3%. The measured data are presented in Figure 9. It can be observed that after 3000 s time interval, no zinc emission can be measured.

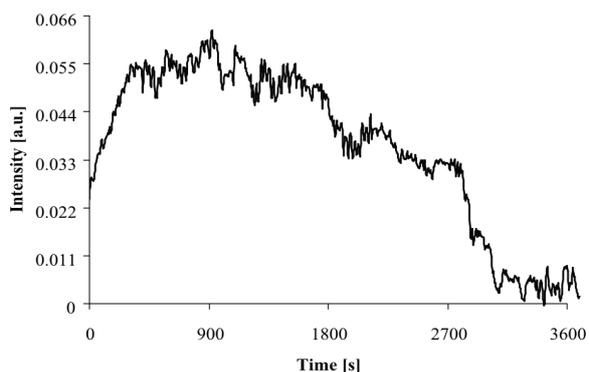


Figure 9: The line intensity measure on the 213.85 nm Zn I. line as the function of time.

Discussion of the results

In the experiments a DC glow discharge source was studied, which was operated in pulse regime, at constant high and low current values. It could be seen that the current generator can be used in a wide range of Ar pressure and the constant current makes possible an easier control of the device. From the viewpoint of the operation of the device the voltage-drop has a key importance, namely as long as it does not exceed the capabilities of the current generator, the constant current mode works. When the current generator runs out of the voltage, it stops working, therefore care must always be taken of the voltage-drop, and careful examination is needed to reveal the relation among the high current, the voltage-drop and that of the filling gas pressure. In *Figure 6* it can be seen that at 72 mA the source can be used in current regulated mode at the range of 300–950 Pa. Below 300 Pa the voltage-drop sharply increases with decreasing pressure, so it probably will not work at pressure values lower than 260–270 Pa.

In *Figure 5* the relationship between the voltage-drop and that of the current is shown. It must be noticed that the first point in the figure is the voltage-drop at the low current value. This also suggests that the device operates in the abnormal operating region within the whole current range.

The emission data measured on the 324.8 nm Cu I line are presented in *Figure 7* and *Figure 8*. It is worth mentioning that the emitted light intensity sharply increases with increasing current, it seems to be an exponential function rather than a linear one. The reason for that sharp increase may be the better sputtering efficiency. On the other hand, the emitted light intensity decreases with increasing filling gas pressure. In *Figure 9* the sputtering ability is demonstrated. It took about 3000 s to eliminate the 2 μm thick Zn layer. If the duty cycle (2.3%) is taken into account, this would mean a sputtering speed of 1.74 μm /minute at continuous DC current. It must be mentioned that the duty cycle the speed depends also on the current and that of the argon pressure beside the duty cycle of the pulses.

It also could be seen that the behaviour of the source is similar to the ones operated in voltage-feed mode,

except for the fact that its state can always be characterised precisely because of the constant-current mode.

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