AUTOMATED ELECTROCHEMICAL ETCHING PROTOTYPE FOR COLD FIELD EMISSION CATHODES

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Abstract: This article deals with a setup for automated cold cathode etching, its difficulties and benefits. As the method is well know there are no articles which describes this procedure in detail at least for tungsten wires from which cathodes are made. To provide stable and repeatable process of cathodes manufacturing, the experimental setup was proposed with full hardware control with aim to assure etching circuit disconnection in order of hundreds of nanoseconds.

Keywords: electrochemical etching, field emission cold cathode, tungsten, automation

1 INTRODUCTION

Cold field emission cathodes are widely used in electron microscopy where the small and stable source of electrons is required. The cold field emission cathodes differ from thermionic or Schottky cathodes by presence of strong electric field which modifies shape and height of the potential barrier and allows electrons to secede from the metal surface. Cathodes usually works at high levels of vacuum with relative low potential applied (approximately 200 V) and do not require additional heating like Schottky or thermionic cathodes [1].

Above mentioned cathode can be obatined either from tungsten wire or any other transition metals such as Niobium, Tantalum, Hafnium or Lanthanum. Electrochemical etching is a cathode manufacturing process in which are involved electrolyte (for tugsten usually KOH or NaOH aqueous solution at specified concentration), an anode and a cathode [2]. Tungsten wire acts as an anode and as a negative electrode a chemically resistant stainless steel wire is commonly used [3]. After applying ething potential between cathode and an anode, anode metal is dissolved as cation in the electrolyte and at the same time an equal amount of cation is deposited on the surface of cathode [4]. The rate of dissolution and deposition depends on the applied elecrochemical potential and concentration of the electrolyte solution and also the process of electrochemical reaction is inflouenced by a complex of other electrochemical effects on anode and cathode (e.g. non-uniform concentration of cations in solution, effect of impurities both in electrolyte and on the surfaces of electrodes, local inhomogenities of electric field etc.), but dissolution and deposition are still dominant processes [5]. The main goal of electrochemical etching of cathode is to a obtain sharp metal tip, which a source a stable emission of electron beam with low fluctuations in an electron microscope system. When the tungsten wire dissolves to an electrolyte, it reaches the point where the bottom part of wire drops off to the solution (from which the name of the method is derived) resulting in rapid voltage drop which is indicated by a steep decrease of current flow through the electrolyte. From this reason, there is a crucial requirement for control system of etching circuit to rapidly disconnect etching potential. If not, the wire tip continues to be etched, leading to decrease of cathode sharpness [6].

The proposed circuit for cathode etching is consisted from a DC power source, Microcontroller Unit (MCU) Arduino Mega 2560, a Printed Circuit Board (PCB) with control electronics and a stepper motor. The MCU sets the parameters of PCB in most of the cases via Serial Peripheral Interface (SPI), such as Digital to Analog Converter (DAC) output voltage and its range, algorithmization of relays switching and selection between AC or DC output. The MCU was chosen because of its well known programming interface, reliability and cost effectivity. The PCB is used as a MCU shield and was designed to control the etching procedure fully via hardware, which eliminates the previous weak point of the process control by Matlab and the GPIB interface - the slow reaction to the drop of the part of the etched wire with subsequently delayed etching potential disconnection. This solution allows the system to simultaneous measurement and amplification of etching voltage and current, motor movement, detection of the etched wire drop, followed with automatic disconnection of the circuit. Because the PCB requires a stabilized power supply with various voltage ranges (+5, +12, +15, -5, -15 V), the power supply with multiple outputs was also designed and manufactured. As the last and the most challenging part of the whole circuit is the motor because as any motor it generates a significant noise background of the circuit due to the Pulse Width Modulation (PWM) which can not be overlooked. This feature is also remarkable during inactivity of the motor because of the effect of the standstill current.

Due to complexity of the circuit this article will be focused on the control PCB, which is the main part of the circuit.

2 EXPERIMENTAL RESULTS

The PCB controls whole etching process and is responsible for applying voltage, right immersion of the wire into the solution and disconnection of the circuit when the etching process is done. Since the PCB the source of etching signal, the DAC converter is utilized. The output of DAC is amplified by the power amplifier in order to obtain required potential between the anode and the cathode. In Fig. 1 the DAC_1 connection diagram as a power source is plotted. The PCB is intentionally designed to govern both oxidation and reduction electrochemical prosesses of further research on cathode manufacturing, i.e. not only etching of the filament, but also for electrochemical deposition of thin layers on the cathode surface with the purpose of the cathode lifecycle enlongation, the increase of its emissivity as a protection from the environmental damage. Beside this, electrodeposition of thin layers on the surface can seriously modify the electronical properties of the cathode, such as the work function of its surface. The desired function of the output is driven by relays K1 or K2. If the relay K2 is switched on, the DAC output will be inverted by the Operational Amplifier (OPA) 4131P.

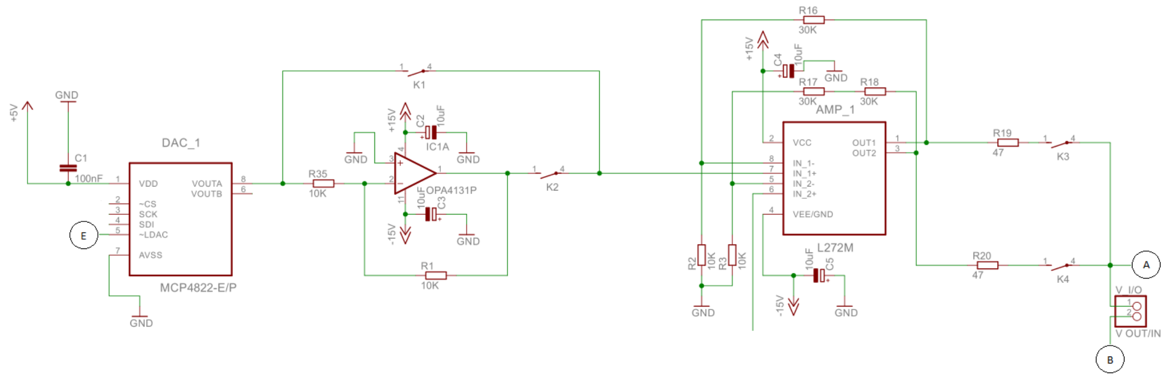

Figure 1: Schematic diagram - DAC power output with inverter (switchable polarity) and amplifier (4 times magnification).

The power amplifier AMP_1 L272M is used to amplify the output of the DAC_1 with the magnification of four. The reason of the signal magnification is the fact that the DAC internal voltage reference is 2.048V, which is the maximum applicable etching voltage without amplification. Even though the 2.048 V is already to perform cathode etching, one of the requirements on the PCB is to provide higher etching voltage, which allows vaster etching parameters variability, such as control of etching rate by higher etching voltage etc. The second input of AMP_1 is used for generation of harmonic signal (generator not shown in the schema) for cleaning the wire before etching. In this case, the magnification is seven, because the amplitude of harmonic signal generator has its maximum at 1 V. The choice between etching/deposition or cleaning modes of the PCB is realized by appropriate combination of switch of relays K3 and K4. The ouputs of AMP_1 are connected to Analog to Digital Converter (ADC), which is part of the MCU (see Fig. 2) and to the tungsten filament.

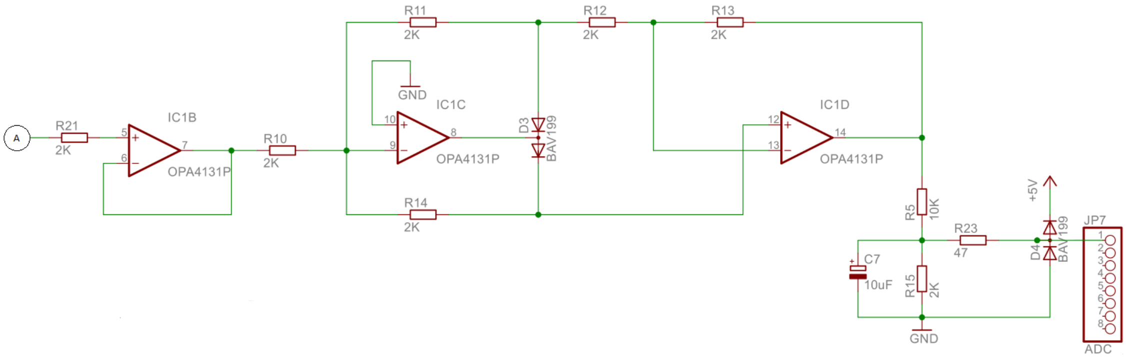

Figure 2: Schematic diagram - full-wave rectifier near ADC converter with voltage divider.

The etching voltage is measured via ADC. Because of the generation of harmonic, positive and negative DC output it has to contain a full-wave rectifier that converts the signal to a positive and prevents the MCU from damage caused by overvoltage as shown in Fig. 2. The rectifier also contains voltage follower composed from OPA to eliminate loading effects. The rectifier is terminated with a voltage divider 1:6 as the input voltage range of ADC is 0–5 V. The pair of diodes D4 strengthens the input protection that the voltage cannot be higher than +5 V and lower then 0 V.

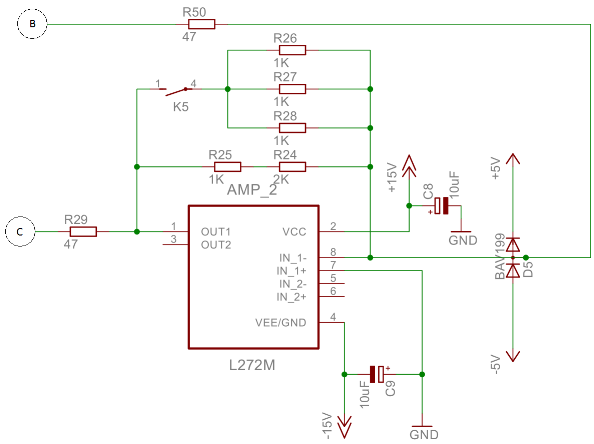

Figure 3: Schematic diagram - current to voltage converter with selectable range.

The cathode is connected to the inverted input of AMP_2 as shown in Fig. 3. This OPA is used as a current to voltage converter with the ability to measure current which flows in the circuit. The range of the amplifier in normal state is 0–10 mA. If the relay K5 is switched on, the range is increased to the value up to 100 mA. For the apllied potential (approximately 5 V), the highest measured current was

20 mA. Such a high current value is caused by biggest etching area of the cathode during initial stage of etching. Thus, the range should satisfy the conditions for even higher etching voltage. The output of the amplifier is devided into two branches where the first one is connected via same configuration as in Fig. 2 to another ADC of the MCU and the second one is connected directly to circuit disconnection logic.

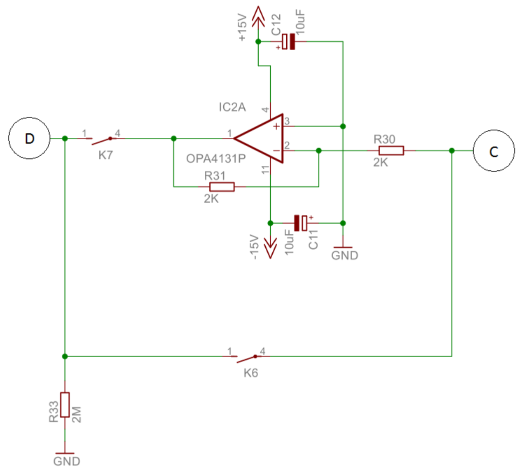

Figure 4: Schematic diagram - inverter near circuit disconnection logic.

If the polarity of output voltage of AMP_2 is negative, the invertor has to be connected into circuit in order to assure the correct function of circuit disconnection logic. This is carried out by switching the relay K7 as shown in Fig. 4. In other cases, i.e. if the output voltage of AMP_2 is either DC positive or harmonic, the inverter must not be used.

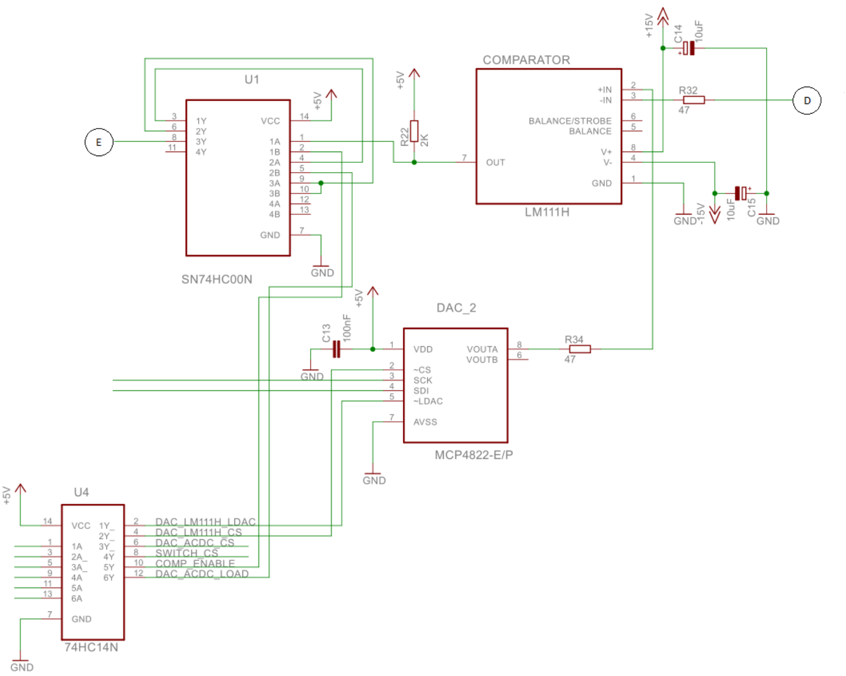

Figure 5: Schematic diagram - circuit disconnection logic.

In Fig. 5 is shown the main logic for circuit disconnection. It includes comparator, DAC_2 for setting the reference voltage, NAND gate (U1) and inverter gate Schmitt trigger (U4). When the voltage on the comparator inverted input is lower then the reference one, comparator grounds the output and the low (logical 0) signal occurs on 1A input of U1. If output Y5 of U4 is set to high (logical 1) it activates latch registers of DAC_1 which was previously set to 0 V and shuts down the circuit.

3 CONCLUSION

This article showed the prototype of PCB used to cotrol fully automated electrochemical etching process of cold field emission cathode manufacturing. Due to the complexity of the whole etching system, the article contains only description of the main parts of the PCB circuit, which is already functional for application. Compared with the previous system, the reaction times of the system are 6 fold faster (hundreds of ns). One of the issues not described in this paper is the motor and its control. Motor carries out wire immersion and emmerge of a wire. When the motor is stopped on standstill current it generates pulses back to the circuit approximately with frequency of 40 kHz. This can not be ommited because the amplifiers also amplify the noise which may lead to instability of the circuit especially the comparator which is very susceptible to oscillations and may cause premature circuit disconnection.

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REFERENCES

[1] L. W. SWANSON and G. A. SCHWIND. Review of ZrO/W Schottky cathode. *Handbook of charged particle optics*. 2nd ed, edited by Jon Orloff. Boca Raton: CRC Press, 2009, xi, 665 p. [cit. 2016-02-20]. ISBN 978-1-4200-4554-3.

[2] M. RICKART and M. BAUER: An improved tip etch procedure for reproducible sharp STM tips. AG Magnetismus, Universität Kaiserslautern, Annual report (1998), p. 53-55[cit. 2016-02-20].

[3] A. I. OLIVA, A. ROMERO G., J. L. PENA, E. ANGUIANO and M. AGUILAR. Electrochemical preparation of tungsten tips for a scanning tunneling microscope. *Review of Scientific Instruments* [online]. 1996, vol. 67, issue 5, p. 1917- [cit. 2015-02-02]. DOI: 10.140631.1146996. Available from URL:<`http://scitation.aip.org/content/aip/journal/rsi/67/5/10.14063/1.1146996`>.

[4] J. LINDSAY. A pulse/pulse reverse electrolytic approach to electropolishing and through-mask electroetching. Products Finishing. *Gardner Publications Inc.* [online]. HighBeam Research. 2011 [2016-02-20]. Available from URL:<`https://www.highbeam.com`>.

[5] O. M. BEHR and M. R. BEHR. Environmentally safe Etching. *Chem Tech* [online]. 1991, vol. 21, issue 4, p. 210-215 [cit. 2016-02-20]. Available from URL:<`http://www.electroetch.com/chemtech.htm`>.

[6] A. J. MELMED, J. The art and science and other aspects of making sharp tips. *Fifth internationl conference on scanning tunneling microscopy/spectroscopy*. 1991, vol. 9, issue 2, p. 601-608 [cit. 2016-02-20].