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Research paper

Design and implementation of a microcontroller based electrostatic spray pyrolysis instrument for thin film deposition

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Abstract

This research is focused on the design and implementation of a microcontroller based electrostatic spray pyrolysis instrument that can sense and regulate temperature change with an alternating current to direct current converter in the range of 220V-1KV AC to 20KV DC for thin film deposition. In spray pyrolysis technique precise management of the spray parameters helps to create good and uniform thin films with ease of reproducibility of film properties. However it is a draw back in the manually operated electrostatic spray pyrolysis instrument mostly used in developing countries of Africa which causes variations in film structure, thickness, associated film properties and difficulty of reproducibility of film properties. The need for improved locally made ESP unit is also timely due to the inadequacy/unavailability of thin film deposition apparatus in most African institutions/materials research laboratories. The circuit was simulated on Multism software and transfer to PCB, etching was done using Lead (II) oxide while the unit components where assembled by soldering of electronics components, bonding and drilling of associated components. Arduinounoatmega 328 micro controller was employed. The fabricated device operated at 1KV to 20KV dc voltage for film deposition, at a substrate temperature range of 100oC to 450oC with a steady flow rate of 0.04ml/min, define spray pattern, define precursor usage and a precise control of major deposition parameters. ZnO:Al thin film deposited with the equipment was found to have a crystalline structure of fine grains.

Keywords: *Spray Pyrolysis; Thin Film; Micro Controller; Multism.*

1. Introduction

The Electrostatic spray Deposition (ESD) also called Electrostatic spray pyrolysis (ESP) comprises of generating an aerosol by applying a high potential (5-25Kv) to a surface of a conducting liquid, which contains desired precursor.
ESD is capable of dividing a liquid into farly uniform and well distributed droplets anddimensions that can be controlled from several mi crometres down to the nanometre range [1]. It has a well-defined trajectory of spray droplets directed towards the substrate by the electric field, making it economical in precursor usage. Compared with other film fabrication methods, it offers attractive advantages of easy control of film composition, easy control of substrate temperature during deposition, high film growth rate, simple setup, lowcost and usage of chemicals and minimal waste.
The process can be carried out in ambient atmosphere, in air or other gases and at low temperature, without the need for a complex reactor and vacuum systems. ESD can produce highly pure materials with structural control at the nanometre scale. The crystallinity, texture, film thickness, and deposition rate can be controlled by adjusting voltage, flow rate, and the substrate temperature [2].
A thin film is a layer of material which ranges from few fractions of nanometre (monolayer) to several micrometres in thickness [3]. Thin films are formed by depositing material onto a clean supporting glass substrate to build up film thickness, rather than by thinning down bulk material. The act of applying a thin film to a surface is called thin film deposition and is use for depositing a thin film of material onto a substrate or onto previously deposited layers.
The enhancement in deposition efficiency and improvement in quality of the thin films can be achieved with atomization techniques such as microprocessor based spray pyrolysis system. In spray pyrolysis technique precisely managing the timings of spray and associated parameters without any error can help to create a good thin film quality [4]. In manual operations, timings, manual switching operations of ventilation, carrier gas flow and substrate movement for many numbers of cycles is been monitored and computed which is prone to many inaccuracies. Hence, such thin film deposition process become imprecise operation for a required number of spray. The research is

aim at the design and implementation of a microcontroller based ESD unit that can sense and regulate temperature/heat change with an alternating current (AC) to direct current (DC) converter in the range of 220V to 1KV AC to 20KV DC for thin film deposition with emphasis on usage of locally available materials.

2. Materials and method

Figure 1 shows the development process of the spray pyrolysis unit, while a typical block diagram of the microcontroller based electrostatic spray pyrolysis unit is shown in Figure 2.

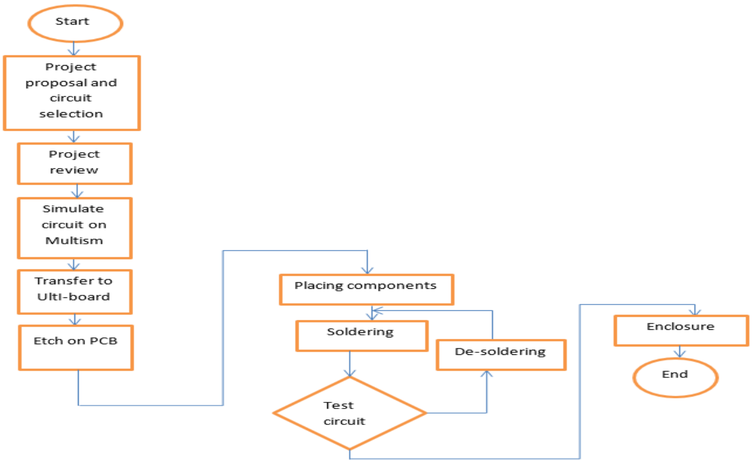

Fig. 1: Development Process of the Spray Pyrolysis Unit.

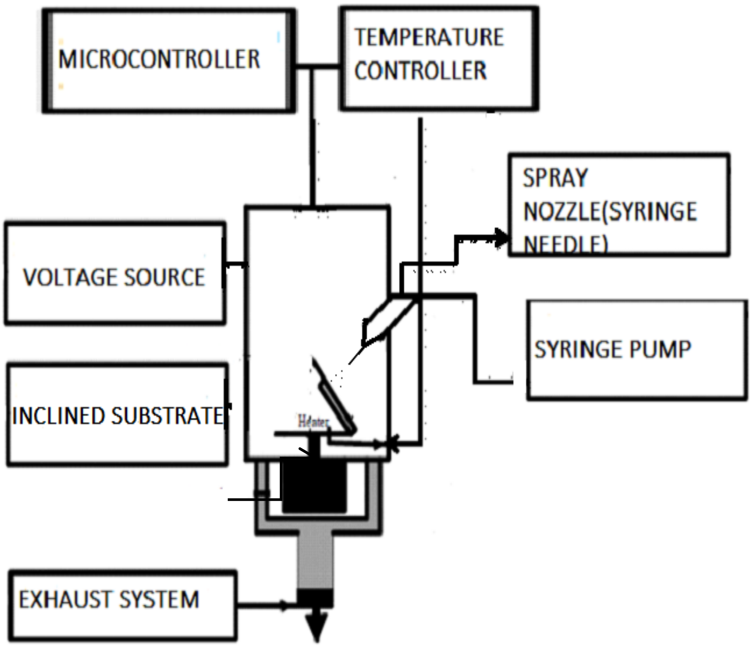

Fig. 2: Block Diagram of A Spray Pyrolysis Unit.

The microcontroller based electrostatic spray pyrolysis system consists of a voltage source, spray nozzle (needle), syringe pump, substrate heater and temperature controller, microcontroller (Arduino Uno ATMEGA328) and exhaust system. The ESD system was designed to handle voltage ranging from 1kilovolt to 20 kilovolt DC with a voltage source connected to the tip of the spray nozzle (needle) which requires a very high voltage for atomization ranging from 1kilovolt to 10kilovolt during deposition. A voltage regulator (switching voltage regulator) is built so that at any point if the system experiences low voltage it can quickly adapt to the present voltage rate without damaging the system, since high voltage is required for operation [5]. The voltage regulator is used for varying the supply voltage from 0 to 2000V. The switching regulator converts the ac input voltage to a switched dc voltage applied to a power MOSFET in the circuit. The filtered power switch output voltage is fed back to the power MOSFET that controls the power switch on and off times so that the output voltage remains constant regardless of input voltage. In operation, the regulator's filtered output voltage is fed back to the power MOSFET controller to control the duty cycle. If the filtered output tends to change, the feedback applied to the power MOSFET controller varies the duty cycle to maintain a constant output voltage. The circuit diagram of the voltage regulator is shown in figure 3.

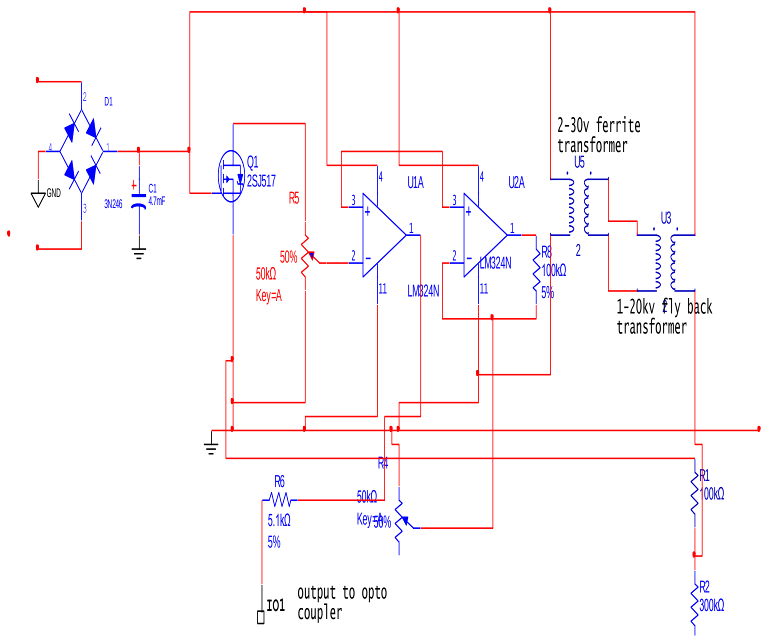

Fig. 3: Circuit Diagram of Voltage Regulator.

The spray nozzle (needle) have a liquid spray tip orifice at one end through which the contents of the syringe may be discharged and being adapted for attachment to the liquid discharge port of the syringe at the other end. The spray nozzle is made up a surgical needle and is adapted for attachment directly to the lure fitting of the syringe. It was made in a way that it sprays at the rate of nano particle form for fine atomization. The spray nozzle works with the syringe and permits it to be used as the precursor reservoir.A 29 gauge needle was used for atomisation while a 5 ml Hypodermic syringe (manufacture by Shandong Zibo Shanchuan medical instrument Ltd, Shandong China- LOT NO: 201312) was used. Figure 4 is the image of a syringe needle (spray nozzle).

Fig. 4: Spray Nozzle (Needle).

The syringe is made up of transparent plastic material for easy viewing and to avoid corrosion, it is also calibrated in order to know the reading each time any deposition is to be made. The syringe serves as the container which is used to measure the quantity or to take the level of the precursors to be dispensed or sprayed. The syringe driver or syringe pump possess an infused and withdrawing capability, it is used to gradually administer small amount of precursor to the spray nozzle for dispensation. 30V ac voltagepowers the syringe pump which also enhances steady flow rate. The syringe pump moves clockwise to infuse and moves anticlockwise to withdraw. Figure 5 shows the image of syringe pump;

Fig. 5: Syringe Pump.

Materials chosen for the design of the substrate heater were based mainly on their high vacuum properties and their machinability such that they can be manufactured with standard machine and tools. The main part of the substrate heater was made of Macor, a glass-ceramic and machinable material with excellent thermal characteristics which can be continuously used at 100℃ with a peak temperature of 450℃. The substrate heater was constructed in a form that it has a surface on which after the droplet leaves the atomizer it travels and impinges the surface of the substrate heater as it is positioned horizontally opposite the spray nozzle in the spray pyrolysis unit. A K-type thermocouple was embedded in the substrate heater to measure the amount of temperature during deposition. In order to regulate the temperature of the substrate heater, a temperature controller was developed embedded in the microcontroller. The microcontroller reads the value of the set point temperature from an adjustable rotary knob, and the substrate heater temperature from a K-type thermocouple. The difference between the set point and thermocouple signal produces an error value. Due to the digital nature of the microcontroller the error signal is converted to integer value after which it is connected to digital input of the microcontroller which generates an interrupt in the software when the logic signal changes from 0 to 1. Figure 6 is the image of the substrate heater employed.

Fig. 6: Substrate Heater.

The microcontroller used is called the Arduino uno atmega328.It is an open-source, small, single-board computer with analog and digital inputs and outputs pins. The digital input and output pins are 14 of which 6 can be used as pulse width modulation (PWM) output, 6 analog input, a 16 MHz crystal oscillator, a USB connection, a power jack, an ICSP header, and a reset button. The programming language is a simple version of C. Code was developed on a personal window computer and downloaded to the board through a USB channel. The maximum length and width of the Uno PCB are 2.7 and 2.1 inches respectively, with the USB connector and power jack extending beyond the former dimension. Three screw holes allow the board to be attached to a surface or case. Microcontroller needs 5V operating voltage, 2V input voltage and the input voltage is 6V to 20V.The microcontroller takes its power supply from the voltage source.

C programming language was chosen because it combines the element of high-level language with functionalism of assembly language. C language also allows the manipulation of bits, bytes and addresses. C codes are portable and they can possibly adapt software written for one type of computer to another. The special feature of C is that it allows the direct manipulation of bite, byte, word and pointers. A text editor was employed to enter the source into the disk file. As noted earlier, the text editor also functions as error correcting in the program. The text editor employed is the MATLAB text editor.The small device C "compiler" comes with a lot of modules which carry out the task of compilation, linking binary to hexadecimal conversion once the compilation command is issued to an object file, a hexadecimal and a binary file. VISUAL BASIC was used to interpret, compile and translate the program to the microcontroller (ARDUINO UNO ATMEGA328) for execution. Visual basic also enhances the graphic user interface (GUI).The image of a Micro controller (Arduino Uno Atmega328) is shown in Figure 7.

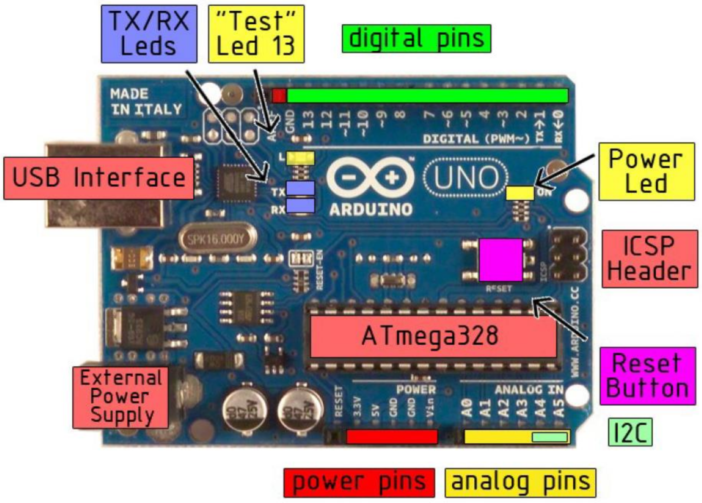

Fig. 7: Microcontroller(Arduino Uno Atmega328).

The microcontroller help in controlling the temperature of the substrate heater, it measures the flow rate of the precursor during deposition, it also manage the spray time and pause time during deposition.

The exhaust system as shown in Figure 8, is a part of spray pyrolysis unit that serves as vent. It is developed to remove the harmful gas produced inside the chamber of the spray pyrolysis unit during the process of deposition. The pipe is built and connected from the inner chamber of the spray pyrolysis unit to the outside environment of the machine. Because of the harmful nature of possible gas, a tail like pipe to the exhaust system to take it far from the immediate environment of the machine was employed.

Fig. 8: Exhaust System in Spray Pyrolysis Unit.

The enclosure of the machine was made of hard plastic material called Perspex (Poly methyl methacrylate)which was bent to our desired shape with the aid of line heater (hard plastic bender). The system was built in such a way that the front view is transparent for easy viewing and access to the inner chamber during any deposition and the rest body was coated with aluminium paint with other components made in automated form to function. The measurement of the enclosure of the spray pyrolysis unit is shown in the Figures 9 and 10, while Figure 11 is the image of the completed assembled microcontroller based electrostatic spray pyrolysis unit.

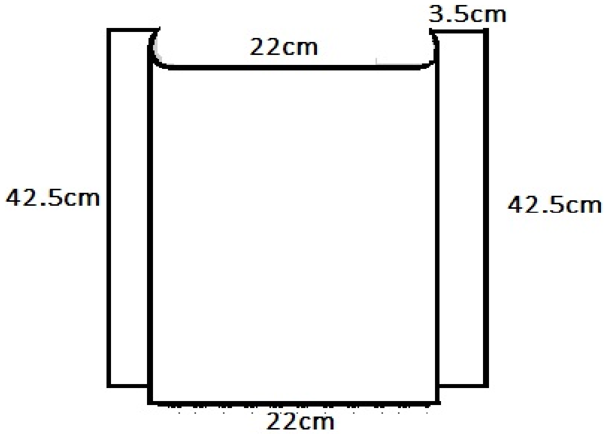

Fig. 9: Enclosure of the Spray Pyrolysis Unit.

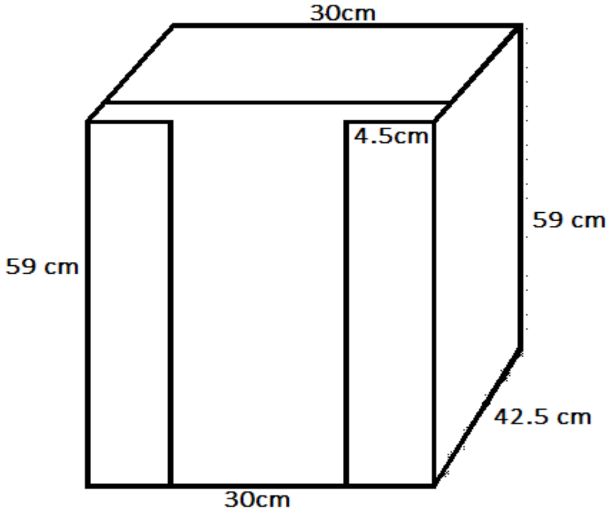

Fig. 10: The Door of the Enclosure.

Fig. 11: Microcontroller Based Electrostatic Spray Pyrolysis Unit

3. Result and discussion

Physical assessment was first carried out to ensure proper assembly of components of the ESP unit. The result of the Multism simulation of the design voltage regulator circuit is shown in Figure 12 which was transferred to ultiboard and printed on A4 paper as shown in figure 13 in a 3D format. The circuit was first designed and simulated on NI Multism 11.0 (electronic workbench) to ensure its operation and afterwards transferred to a Ultiboard where it was printed out on an A4 paper. The simulation was launched on the double sided PCB (printed circuit board) and etched using lead (II) oxide (PbO_2) followed by components assembly.

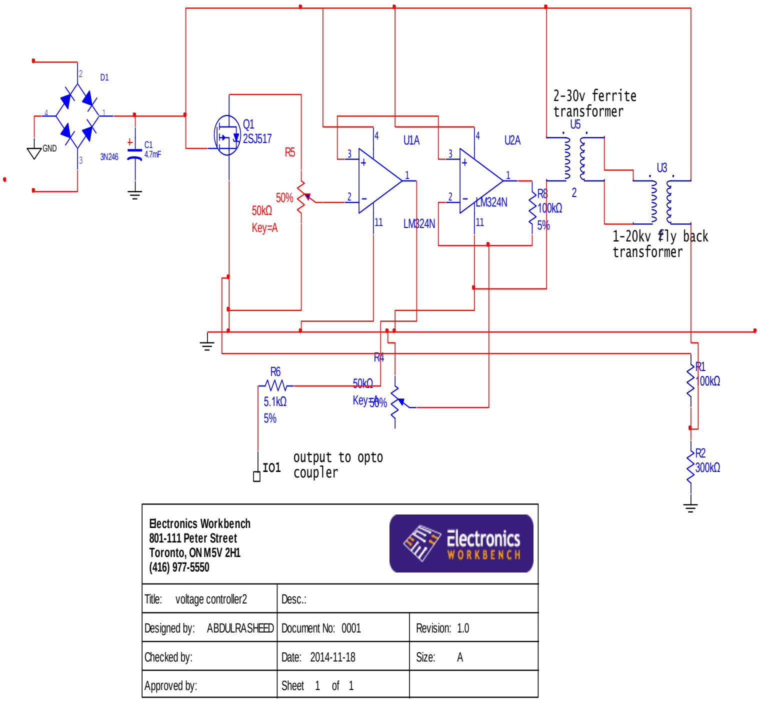

Fig. 1: Circuit Design of Voltage Regulator.

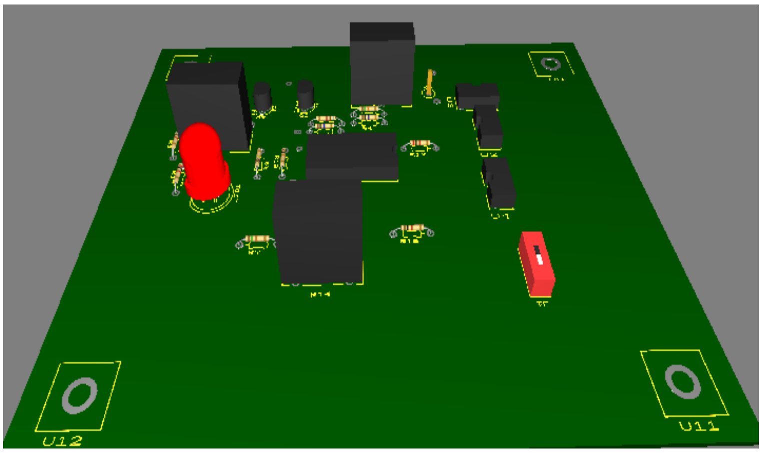

Fig. 13:3D Design of Voltage Regulator Circuit on Ultiboard.

The voltage regulator converts the 220V input ac to dc by a bridge rectifier and filtered by a ceramic capacitor which goes to power the MOSFET to switch it on. The ferrite transformer steps down the voltage to 2V-30V dc which is now step up to 1KV-20KV dc by a fly-back transformer to atomize the tip of the spray nozzle. 5V dc gotten from the 20KV dc by voltage divider was fed back to the power MOSFET which controls it ON and OFF (duty cycle). The microcontroller also operates at 5V dc.The substrate heater afterwards gets it supply from the microcontroller. The syringe pump operates via aasynchronous motor which has a transformer that takes the 220V ac voltage from voltage source to steps it down to 30V which powers the syringe pump for a steady flow rate. When the ESP unit is powered ON, the machine, initialize the temperature using the temperature knob. With the precursor loaded on the syringe, the substrate on the substrate heater and temperature adjusted to the desired value, a high voltage source atomizes the syringe tip to create the aerosol that impinges the substrate on the substrate heater to thermally decompose the precursor leading which initiates a chemical reaction to form a thin film deposited on the substrate in a process called pyrolysis.

The equipment was used to depositZinc oxide doped Aluminium (ZnO:Al) thin film using 0.2 M Zinc Acetate (99.99%, BDH) solution which was prepared by dissolving a solute quantity of 1.756 g of Zinc Acetate in 40 ml of solvent (Ethanol: water (80:20 v/v)) with 0.2M aluminium chloride (99.50%, BDH) solution which was prepared by dissolving a solute quantity of 1.0666 g of Aluminium chloride in 40 ml of solvent (Ethanol: water (80:20 v/v)) at 3at % dopant concentration. The two solutions were mixed and stirred for 30 minutes using a magnetic stirrer at room temperature, after which the resulting solution was filtered through a 0.22 µm syringe filter and then deposited on a soda lime glass substrate. A syringe pump(5 ml- Hypodermic syringe, manufactured by Shandong Zibo Shanchuan medical instrument Co. Ltd, Shandong China- LOT NO: 201312)was used to feed the precursor solution through a silicon tubing connected to a small stainless steel needle (0.184 mm and 0.3366 mm, inner and outer diameter respectively) for atomization. D.C voltage supply (5-20 kv) was applied between the needle tip and the hot plate to get a stable cone-jet mode by a D.C power supply. The distance between the nozzle and the substrate was kept constant at 10 cm for all depositions at a constant substrate temperature of 400 ℃, spray volume of 0.2 mL and spray rate of 0.04 ml/min.A Profilometer (VEECO DEKTAK 150) was used to carry out measurement of the thickness of the deposited film while a Phenoworld, Pro X model of scanning electron microscopy (SEM) was used to observe the morphology of the ZnO thin film at 5000x magnification. The thickness of the deposited film was found to be 0.18 µm while the morphology as shown in figure14 is fully covered with fine and large grains which leads to better surface morphology and crystallographic structure.

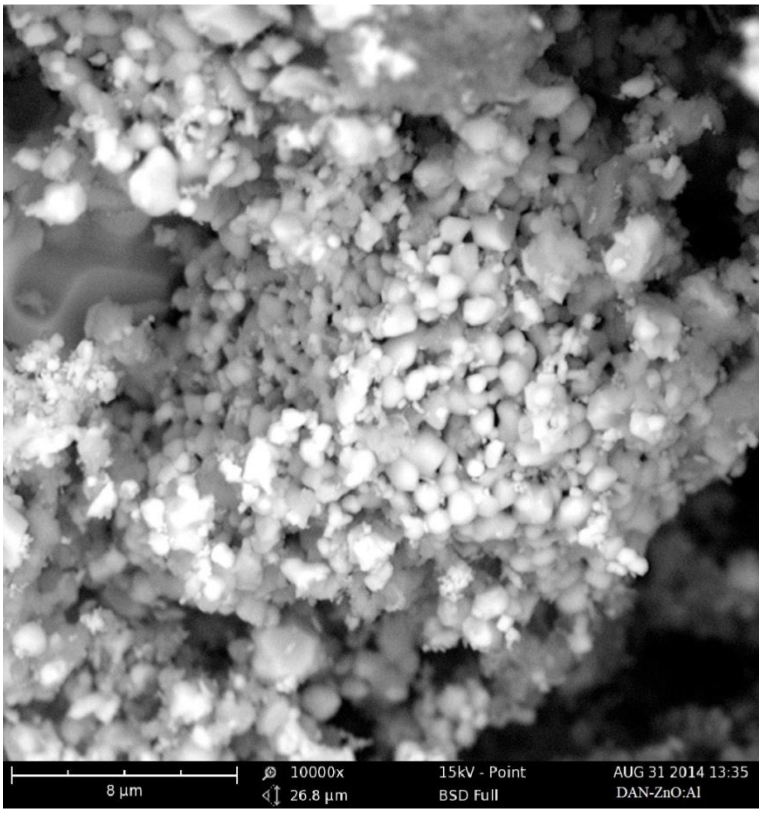

Fig. 14: Zno:Al Thin Film.

4. Conclusion

The fabricated electrostatic spray pyrolysis machine operated maximally with an operation voltage of 1KV to 20KV dc voltage, substrate/deposition temperature range of 100ºC to 450ºC and a steady flow rate of 0.04ml/min. The components were made in automated form with the aid of standard micro controller (arduinouno atmega328) to enhance efficiency of the spray pyrolysis system and to make it user friendly.ZnO:Al thin film deposited with the equipment was found to have a crystalline structure of fine grains.

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Conflict of interest

The authors declare that they have no conflict of interest

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