Novel automation of intermittent spray pyrolysis using microcontroller

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Spray pyrolysis represents a very simple and relatively cost-effective processing method (especially with regard to equipment costs). The quality and properties of the films depend largely on the process parameters. The most important parameter is the substrate surface temperature. In Intermittent spray the spray is paused after regular interval to reduce the drop down of substrate temperature in continuous spray. It is a proven fact that intermittent spray has advantages over the continuous spray pyrolysis deposition. In this work the SPTD equipment is fabricated. The novelty is the use of a simplified low cost microcontroller to automate the intermittent spray process as per the requirements. In this it simultaneously governs the precise intermittent spray, ventilation of the chamber and substrate movement. Hence, it becomes almost an automatic coating process which helps to get quality thin films. The focus of interest in this research paper is to mention the computer programme written in C language for the microcontroller. It automatically controls the spray process as per the user defined intermittent spray settings. Using this economic device thin film like TCO and TiO_2 were prepared for studying / manufacturing Dye Sensitised Solar Cell (DSSC). This paper is expected to be useful for researchers and manufacturers for making thin films by almost automatic SPTD method.

1. Introduction

In modern technology there is widespread the use of thin films for different applications spray pyrolysis had been used for several decades in the glass industry [8] and in solar cell production [9].Typical spray pyrolysis equipment consists of an atomizer, precursor solution, substrate heater, and temperature controller. The method involves spraying a solution containing precursors onto a heated substrate. Sprayed droplets or residual particles reaching the hot surface undergo pyrolytic decomposition and form a single or a polycrystalline film. In this paper an economical microcontroller is linked with it to improve the quality of the thin film by making use of user defined intermittent spray and ventilation and substrate motion. All the functions may be simultaneously done by tapping the out put from the microcontroller. Hence, once the details are set, then it's almost an automatic coating. This developed system tested to prepare thin film materials [4,6,19,21,10,13] for DSSC applications.

The quality and properties of the films depend largely on the process parameters. The most important parameter is the substrate surface temperature [1,5,14].In many studies the deposition temperature was reported indeed as the most important spray pyrolysis parameter [15]. The properties of deposited films can be varied and thus controlled by changing the deposition temperature [1,2,3,17,21].The substrate temperature has influences over the resulting optical and electrical properties of SnO-F, ZnO films [21,16,19].Hence, in some of the reported research papers it has been stated that an intermittent spray was preferred in the spray coating process for getting a good thin film material formation [19,20,21,18,9,10,11,12,13]. In the time gap between the two sucessesive sprays of a series of sprays the substrate temperature (T_s) and the air medium above the substrate are restored to its initial levels of preferred temperature.

For a taken chemical solution as the source material for spray pyrolysis the resulting thin films especially for optoelectronics applications may be improved by implementing an optimized intermittent spray. Using this developed automatic microcontroller assisted thin film coating device intermittent spray time is adjustable.

2. Innovative design using microcontroller

In SPTD precisely managing the timings of an intermittent spray (spray time and spray pause time) with out any error can help to create a good thin film and reproducibility of thickness and thin film quality. In manual operations one has to precisely monitor these timings, manual switching operations of ventilation, carrier gas flow & substrate movement for many numbers of cycles. This is to be recorded then and there for later analysis and for reporting the resulting thin film

properties. A micro controller device can easily and efficiently handle this task. Hence, such thin film coating becomes precise and hassle free operation for a required number of intermittent sprays.

3. Experiment

The microcontroller circuit necessary for the automatic intermittent spray is fabricated. The block diagram of the microcontroller circuit with the spray pyrolysis thin film coating unit is given in figure 1. The over all circuit diagram of the microcontroller (timer) circuit is given in Fig. 2. The photograph of the microcontroller circuit is given in Figure.3. The microcontroller and the spray pyrolysis apparatus photograph is given in fig. 4.
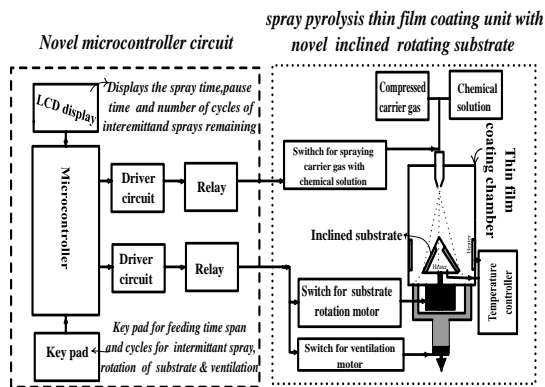

Fig. 1. Block diagram of the newly developed spray pyrolysis set up

Since, this paper is focusing the details of microcontroller programme for the automatic intermittent spray and its interface with SPTD the significance of intermittent spray and its experimental part are not given in detail (These will be published elsewhere). The optimized spray of on, off durations is fed through the keyboard and checked on the LCD display. The spray process can be paused indefinitely using the pause switch in the keyboard. Later the paused experiment can be resumed by using the start key. Using the apparatus SnO-F, TiO_2 thin films were prepared.

A mathematical model was developed [15] for the prediction of thin film thickness deposited using spray pyrolysis and it is used here to optimize the fabricated SPTD apparatus.

The programme written for the microcontroller is given below.

```
#include <REGX52.H>
#include <lcd_smcl.h>
#include <i2c_23.h>
```

```
sbit col1=P1^0;
sbit col2=P1^1;
sbit col3=P1^2;
sbit col4=P1^3;
sbit row1=P1^4;
sbit row2=P1^5;
sbit row3=P1^6;
sbit row4=P1^7;
sbit rel1=P3^0;
sbit rel2=P3^1;

void time_set();
void time();
void save(unsigned char);
void timer_init();
void stp();
void hex(unsigned char);
void pause();
void data_write(unsigned char,unsigned int);
unsigned int get(unsigned char);

unsigned int
cu,n,p[5],ontime,offtime,cycle,count,time1,time2,time3,va
r;
unsigned char a,b,c;

void main()
{
        lcd_init();
        lsb();
        timer_init();
        var=0x50;
        i2c_write(var,50);

        while(1)
        {
home:
                rel1=rel2=1;
                command(0x80);
                lcd_condis(" Microcontroller ",16);
                command(0xc0);
                lcd_condis("    Timer      ",16);

                row1=0;row2=row3=row4=1;
                if(col1==0)time_set();


                row4=0;row2=row3=row1=1;
                if(col3==0)
                {
                        command(0x80);
                        lcd_condis("  Processing
",16);
                        command(0xc0);
                        lcd_condis("              ",16);
                        del();del();
                        command(0x80);

                        lcd_condis("   Cycles:  ",16);
                        command(0xc0);
```

```
                    lcd_condis("ON:    OFF:
",16);
                    time1=get(0x10);
                    time2=get(0x20);
                    time3=get(0x30);

        command(0xc3);hex_dec1(time1);

        command(0xcc);hex_dec1(time2);

        command(0x8c);hex_dec1(time3);

                    while(time3--)
                    {
//                  rel1=rel2=0;
                            command(0x8c);
                            hex_dec1(time3);
                            time1=get(0x10);
                            TR0=1;
                            while(time1)
                            {
                            c=1;
                            command(0xc3);
                            hex_dec1(time1);
                            rel1=rel2=0;

        row4=0;row2=row3=row1=1;

        if(col4==0){rel1=rel2=1;goto home;}

        row1=0;row2=row3=row4=1;

        if(col2==0){pause();if(var)goto home;}
                            }
                            command(0xc3);
                            hex_dec1(time1);
                            time2=get(0x20);
                            while(time2)
                            {
                                    c=2;

        command(0xcc);

        hex_dec1(time2);
                                    rel1=rel2=1;

        row4=0;row2=row3=row1=1;
        if(col4==0){rel1=rel2=1;goto home;}
        row1=0;row2=row3=row4=1;
        if(col2==0){pause();if(var)goto home;}
                                    }

        command(0xcc);

        hex_dec1(time2);
                            command(0x8c);
                            hex_dec1(time3);
        row4=0;row2=row3=row1=1;
```

```
                    if(col4==0){rel1=rel2=1;goto home;}

                    row1=0;row2=row3=row4=1;

                    if(col2==0){pause();if(var)goto home;}
                            }
                        }
                }
        }
void timer_init()
{
        TMOD=0x01;
        TH0=TL0=0x00;
        EA=1;
        ET0=1;
        TR0=1;
}

void timer0(void ) interrupt 1
{
        count++;
        if(count==15)
        {
                count=0;
                if(c==1 && time1)time1--;
                if(c==2 && time2)time2--;
        }
}
void time_set()
{
        TR0=0;
        command(0x01);
        command(0x80);
        lcd_condis("   ON Time:0000",16);
        time();
        ontime=(p[0]*1000+p[1]*100+p[2]*10+p[3]);
//      command(0xc0);hex_dec(ontime); del();
        data_write(0x10,ontime);
//      command(0xc0);hex_dec(ontime);
//      i2c_write(0x10,ontime);del();

        command(0x80);
        lcd_condis("   OFF Time:0000",16);
        time();
        offtime=(p[0]*1000+p[1]*100+p[2]*10+p[3]);
//      command(0xc0);hex_dec(offtime); del();
        data_write(0x20,offtime);
//      command(0xc0);hex_dec(offtime);
//      i2c_write(0x20,offtime);
        del();

        command(0x80);
        lcd_condis("   No cycle:0000",16);
        time();
        cycle=(p[0]*1000+p[1]*100+p[2]*10+p[3]);
//      command(0xc0);hex_dec(cycle); del();
        data_write(0x30,cycle);
//      command(0xc0);hex_dec(cycle);
```

```
//      i2c_write(0x30,cycle);
        del();
        command(0x80);
        lcd_condis(" Microcontroller ",16);
        command(0xc0);
        lcd_condis("    Timer      ",16);
}

void data_write(unsigned char locat,unsigned int value)
{

        unsigned char a,b;
        a=value/255;
        b=value%255;
//      command(0xc0);hex_dec(a);hex_dec(b);
        lsb();
        i2c_write(locat,a);locat=locat+1; delay(500);
        i2c_write(locat,b); delay(500);
}

unsigned int get(unsigned char loc)
{
        unsigned char aa,bb;
        unsigned int cc;
        lsb();
        aa=i2c_read(loc); delay(500);
        bb=i2c_read(loc+1); delay(500);
        cc= (aa*255)+bb;
        return(cc);
}
void time()
{
        row1=0;row2=row3=row4=1;
        while(!col4);
        delay(60000);

        cu=0x8c;n=0;command(0x0e);
        row1=0;row2=row3=row4=1;
        while(col4)
        {
                command(0x0e);command(cu);
                row2=0;row1=row3=row4=1;
                        if(col1==0) save(0);
                        if(col2==0) save(1);
                        if(col3==0) save(2);
                        if(col4==0) save(3);
                row3=0;row1=row2=row4=1;
                        if(col1==0) save(4);
                        if(col2==0) save(5);
                        if(col3==0) save(6);
                        if(col4==0) save(7);
                row4=0;row1=row1=row3=1;
                        if(col1==0) save(8);
                        if(col2==0) save(9);

                        row1=0;row2=row3=row4=1;
        }
}
void save(unsigned char s)
{
```

```
        delay(65000);
        command(cu);
        write(s+0x30);
        p[n]=s;
        n++;cu++;
        if(cu>0x8f){cu=0x8c;n=0;}

}

void pause()
{
rel1=rel2=1;
        TR0=0;
        data_write(0x40,time1);
        data_write(0x50,time2);
        data_write(0x60,time3);

        command(0x01);
        command(0x80);
        lcd_condis("     Pause      ",16);

        row4=var=0;row2=row3=row1=1;
        while(col3 && col4);
        if(col3==0)
        {
                time1=get(0x40);
                time2=get(0x50);
                time3=get(0x60);
                command(0x80);
                lcd_condis("    Cycles:    ",16);
                command(0xc0);
                lcd_condis("ON:    OFF:   ",16);


                command(0xc3);hex_dec1(time1);
                command(0xcc);hex_dec1(time2);
                command(0x8c);hex_dec1(time3);
                TR0=1;
        }

        if(col4==0)          var=1;
}
```

4. Results and discussion

The thin film formed by this method of microcontroller aided spry pyrolysis deposition technique is tested for its performance. For a prescribed intermittent spray on time, off time and for a specified number of cycles the device functioned correctly as per the instructed data. The microcontroller developed is helpful by simultaneously monitoring the intermittent spray, ventilation and for substrate motion control. Using these economic device thin films like TCO (Sno-F) and TiO_2 are prepared for studying / manufacturing Dye Sensitised Solar Cell (DSSC).The standard peaks indicate the polycrystalline nature confirming presence of the elements in nano crystal size range (TiO_2:~30nm; Sno-F: ~80nm).Using them a DSSC is prepared using

pomegranate dye. The efficiency of it is 0.49%.The thin films are homogeneous and reproducible. The thickness of thin film prepared is also variable in accordance with
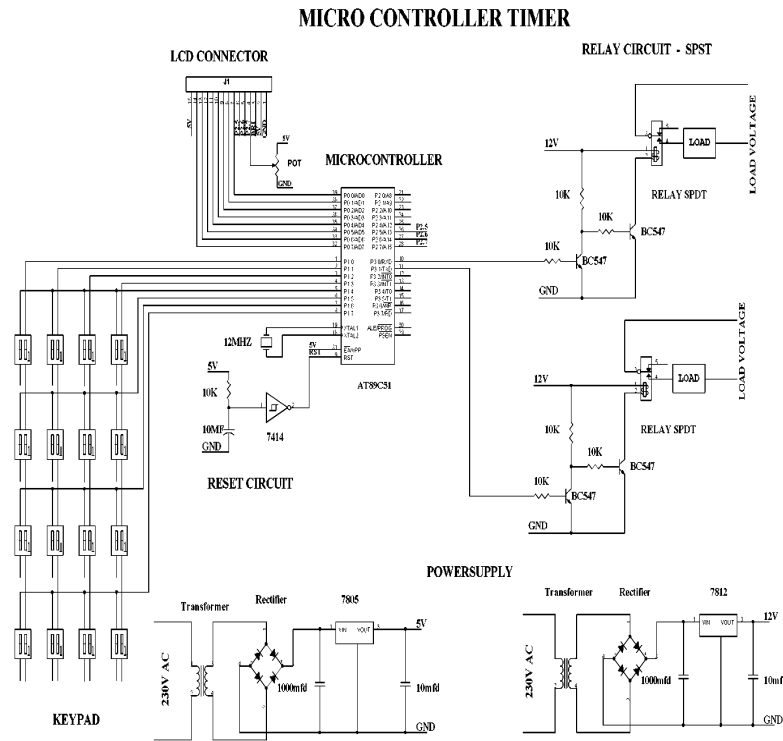
DSSC application [15].

MICRO CONTROLLER TIMER

Fig. 2.The overall circuit diagram of the microcontroller circuit.
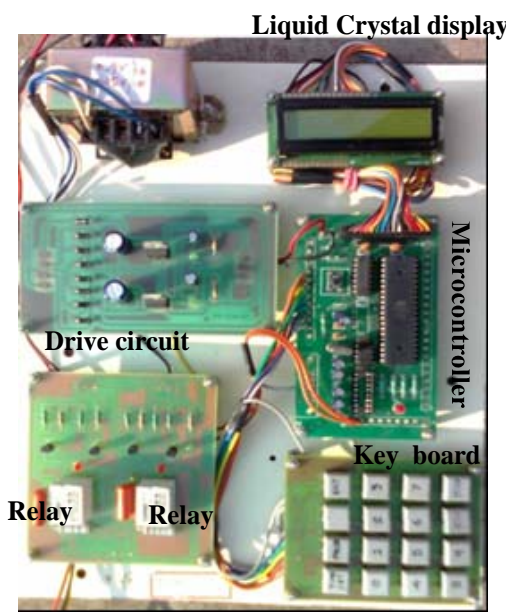

Fig.3. Photograph of the microcontroller circuit.

Fig. 4. Photograph of the microcontroller circuit with spray pyrolysis set up.

5. Conclusion

In this developed novel SPTD the whole intermittent spray processes were automatic and hence a good quality thin film coating is achieved every time. Using it thin films like SnO-F and Nano TiO_2 are prepared for studying / manufacturing Dye Sensitised Solar Cell (DSSC).Other thin film preparations can be attempted using this equipment.

This microcontroller may be modified accordingly for automating spin & dip coating methods of thin film preparations.

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