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Porosity of Metals/Alloys/Porous Solids with Design of Air Pycnometer

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Abstract – **The physical properties of materials play an important role in production, handling and its utilization. Physical properties such as density, thermal conductivity and strength depend on the pore structure of the solids. A quick and accurate method of measuring the physical property like porosity is required for more efficient management of the process with density route. Number of devices were designed to determine the density of solids but are costly and not yet accurate. In this context a simple and low cost air pycnometer is designed which utilizes ideal gas principle to determine the amount of air space within a given material and further density. It is designed with steel chambers, solenoids along with pressure sensor interfaced with AT89C51 microcontroller. The computed densities are within the specified limits with accuracy less than 10%.**

Keywords – **Pycnometer, Porosity, Density and Microcontroller, Solenoids.**

I. Introduction

Most materials are to some extent porous, indeed it is difficult to find or prepare a truly non porous solid. Control of porosity [1-2] is of great industrial importance that influences the chemical reactivity [3] and physical interaction of solids with gases and liquids. Porosity described with numerous quantities in condensed way and particle volumes are important properties of solids in numerous technical processes. Porosity of a solid is the volume of interstices measured in % of the overall apparent volume of the solid that characterizes the rate of flow of a fluid through it. Porosity existing as open [4-5] and closed [6-8] affect the apparent density [9] of a solid. Open pore structure is conductive to permeability. This is most commonly used method of measuring open porosity that is based on the increase in weight of a solid resulting from immersion in a liquid.

If, W_1 is weight of sample in air, D_1 is apparent density of sample, W_2 is the weight of sample after immersion and density of liquid as D_2 then open porosity P is

$P = (W_2 – W_1)\, D_1 \,x100 / W_1 \,x\, D_2$

Corresponding to closed porosity it is

$$P_1 = \frac{Vt - (P+S)}{Vt} \times 100$$, Where

Vt = total volume (100%), P_1 = percentage of closed porosity, P = percentage open porosity, S = percentage of solids given by

$$S = \frac{W \times 10}{D.Va}$$, where

W = weight of crushed powder, D = density of crushed powder and V_a = apparent volume before crushing.

Numerous methods [10-11] are designed in determination of porosity either with water or air for which air pycnometers are costly yet accurate. Air pycnometer is applicable to where calculation of specific weight is desired that permits direct reading of volumes.

Gas pycnometry is based on Boyle-Mariotte's law of volume-pressure relationship. This is an attractive method to determine the volume of solid particles. Two chambers are connected with a valve so the compressed air vessel can be isolated. The sample is placed in the sample vessel and the air vessel is pressurized to a set pressure. The valve is opened and the pressure is allowed to equilibrate. In a closed system with moderate pressures, as in the pycnometer, the temperature remains constant so the term nRT of the ideal gas law remains constant. Thus, if the initial pressure and volume are known, and the final pressure is measured, the final volume can be calculated using the relationship

$$P_1 V_A = P_2 V_T$$

P_1 = initial pressure in compressed air vessel (kPa),
P_2 = final pressure of equilibrated system (kPa),
V_A = volume of compressed air vessel (L), and
V_T = volume of overall system

The mass of the air initially in the full sample vessel is assumed to be negligible compared to the mass of the air in the compressed air vessel

Expanding the V_T term:

$$P_1 V_A = P_2[V_A + V_B - V_S (100 - FAS)/100)]$$

Where V_B = volume of sample vessel (L), and
V_S = volume of solid sample (L) then FAS of the sample
$FAS = 100\, [(P_1 V_A/P_2) - V_B - V_A + V_S] / V_S$

This assumes that the mass and associated pressure of the air in the headspace of the sample vessel were negligible determination of density of materials [12-13] is a measure of the mass of material within a given volume and is important in the determination of initial compost mixtures, determines how much material can be placed at a certain site of a given size further influences mechanical properties such as strength, porosity, and ease of compaction. With vital importance of density [14-15], a simple and reliable air pycnometer[16] of low cost is designed for determining the porosity of metals and alloys.

In this setup, the volume of the powder material or the porous solid is measured, and thus the density is computed. Comparison of the absolute density with that of the obtained density will yield the percentage of densification that is the degree of porosity. The accuracy is achieved upto ±5% of absolute value [17-18]. Instrument is designed using twin steel chambers of 50mm height and 50mm diameter, 3 solenoids of one way NC, Pressure sensor of 100 Psi capacity, a dedicated AT89C51 Microcontroller board to interface the experimental set-up.

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II. Materials and Methods

The present study is proposed to conduct with a view to understand and determine the density (thus porosity) of solid materials by using a low cost air pycnometer. Six samples are chosen for the present study. Choice of the samples include variety of solid materials like pure metals, alloys, and porous solids like pieces of clay pot and water filter candle.

All the samples are washed with trichloroethylene for 2 to 3 times and rinsed finally with the same liquid. Surface impurities if any present are removed in this process. Then all the samples were allowed to dry in sun light for about 2 hours. Then these were kept in an oven at 100^0C for 5 minutes to remove the moisture content completely. The samples were taken out from oven, and cooled in desiccators, which provides a dry environment through calcium chloride ($CaCl_2$) as moisture absorbent.

III. Results and Discussion

The designed air pycnometer that is highly accurate and reliable in determining density of materials is realized in number of steps.

A. Block Diagram

The block diagram of designed air pycnometer constitute
1. Experimental set-up. 2. Pressure sensor
3. Signal conditioning unit
4. Analog -to-Digital converter (ADC).
5. Microcontroller (AT89C51)
6. External memory (AT24C64) 7. Keypad
8. LCD (16 X 2 Liquid Crystal Display) and
9. MAX232 is schematically illustrated in figure 1.

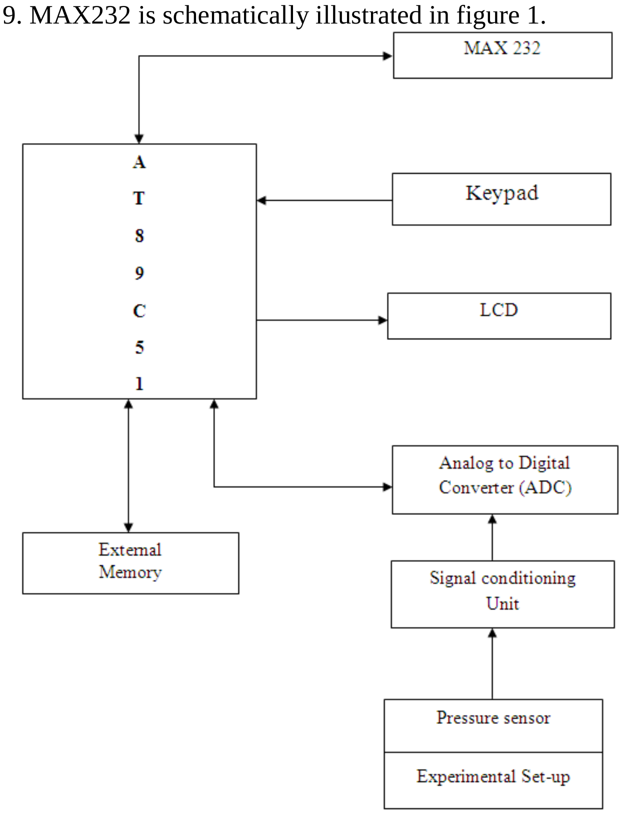

Fig.1. System Block Diagram

B. Experimental setup

The experimental setup consists of Sample chamber: 50mm diameter x 50 mm height (volume V1) Reference chamber: 50mm diameter x 50 mm height (volume V2)
Three 24V DC solenoid Valves ¼” of Aluminium
3- T’s of ¼” piping
6 - ¼” piping of NPT to ¼” BSP piping
2 - ¼” to 1/8” Elbow
Digital pressure gauge 1-1000 mbar range and vacuum pump as illustrated in figure 2.

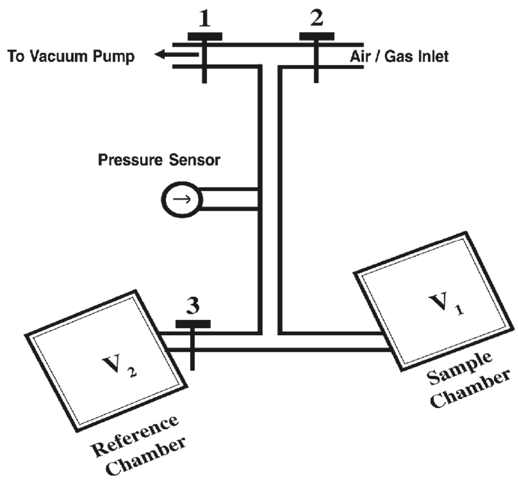

Fig.2. Experimental Set up

Pressure sensors are used for control and monitoring of applications. Pressure metrology is the technology of transfusing pressure into an electrical quantity. Normally, a diaphragm construction is used with strain gauges either bonded to, or diffused into it, acting as resistive elements. The pressure induced strain, the resistive value change. The resistors are connected as a Wheatstone bridge, the output of which is directly proportional to the pressure. Pressure sensor is a SENSYM ICT of 19C100PA4K with pressure range of 0- 100Psi.

The Analog-to-Digital convertor utilises MCP8208 device as successive approximation 12-bit analog-to-Digital (A/D) convertors with sample and hold circuit. It is programmable to provide four pseudo differential input pairs or eight single ended inputs.

The AT89C51 is a low-power high-performance CMOS 8-bit microcomputer with 4K bytes of Flash, 128 bytes of RAM, 32 I/O lines, two 16-bit timer/counters, five vector two-level interrupt architecture, a full duplex serial port, on-chip oscillator and clock circuitry. In addition, the AT89C51 is designed with static logic for operation down to zero frequency and supports two software selectable power saving modes. The idle mode stops the CPU while allowing the RAM, timer/counters, serial port and interrupt system to continue functioning. The Power-down Mode saves the RAM contents but freezes the oscillator disabling all other chip functions until the next hardware reset.

EEPROM stands for Electrically Erasable Programmable Read Only Memory to store the reference value and changed by using the serial communication I2C. It is a two wire serial communication where we can communicate with more number of devices.

Keypad is used to select the key, in which mode and to set the value of the reference value like density and mass of the samples. Momentary switches allow you to precisely time our interaction with a system. Which are of such 4 SPST black colour switches. Port 0 is connected to J2. Input J2 to JP3 means the switches are for external use. The liquid crystal display (LCD) used is 16 X 2 that displays the alphanumeric characters. It is used to display the density and the mass of the sample and the current status of the experimental procedure. Connections are from J2 port 0 to JP5 and J1 port1 is connected to LCD pins. When data is send to LCD then RS is HIGH and when commands send to LCD then RS is LOW

MAX232 is used to interface the controller to the PC. The voltage levels of the controller and PC are different using voltage level convertor it is possible. Microcontroller is CMOS technology and DB-9 convertor is the TTL technology. MAX232 is a dual driver/receiver that includes a capacitive voltage generator to supply EIA-232 voltage levels from a single 5-V supply. Each receiver converts EIA-232 inputs to 5-V TTL/CMOS levels.

These receivers have a typical threshold of 1.3 V and a typical hysteresis of 0.5 V, and can accept ±30-V inputs. Each driver converts TTL/CMOS input levels into EIA-232 levels. Density determination is performed in the following cycle as illustrated in figure 3

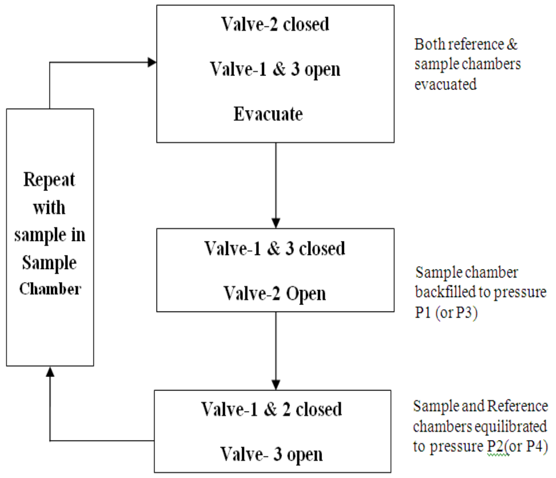

Fig.3. Measuring the Density of metals/ alloys /porous solids procedure cycle

where

P_1-Gas Pressure in the sample chamber without sample
P_2-Gas pressure after equilibration without sample
P_3-Gas pressure in the sample chamber with sample
P_4- Gas pressure after equilibration with sample
V_s –Sample Volume to be measured.
These are implemented with circuitry as shown in figure 4

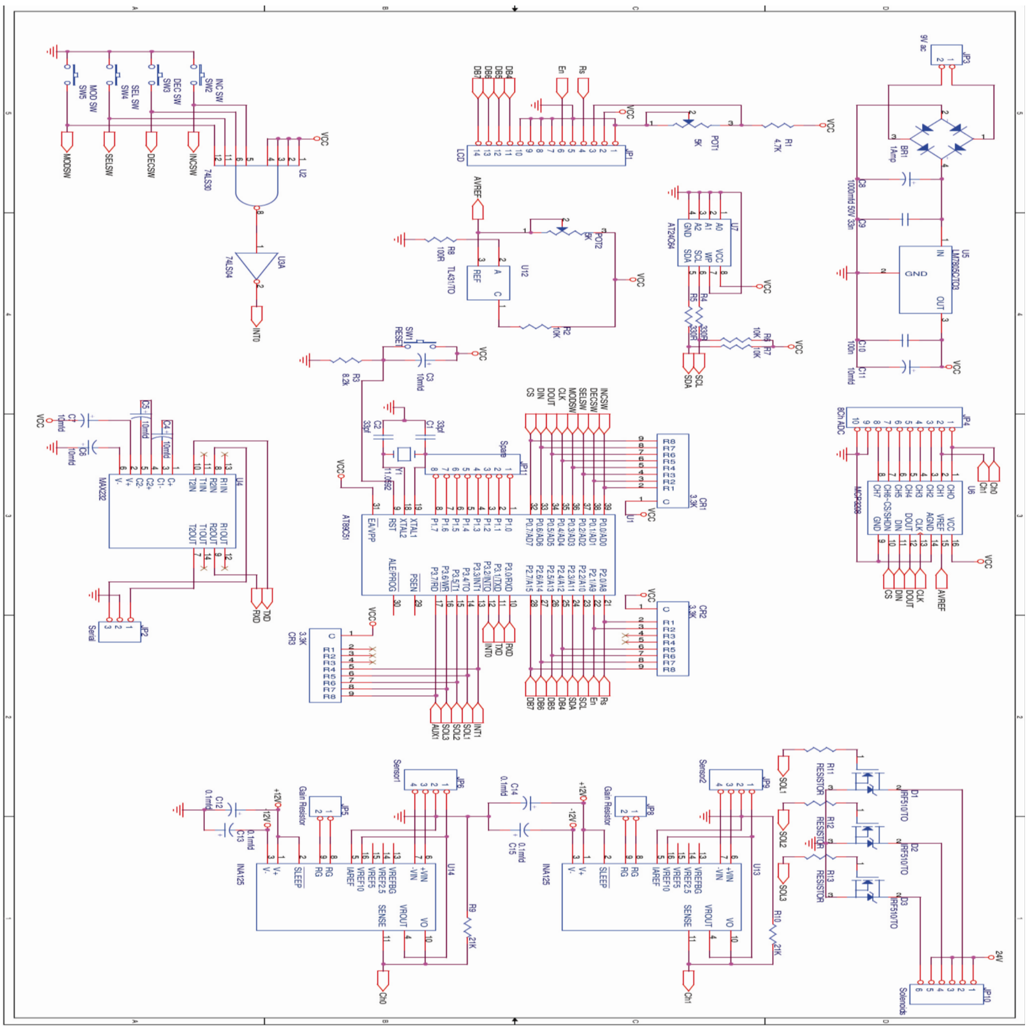

Fig.4. Implementation with circuit diagram

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Power supply section consists of the Bridge rectifier and the voltage regulator. The main purpose of the Bridge rectifier is used to convert the a.c voltage to d.c voltage. That d.c. voltage is converted into required quantity by using the voltage regulator. Positive voltage regulator 7805 is used and the output is of 5V d.c. and that voltage is converted to the V_{cc} pin of the I.C's that are operating at 5V. Another source of 12V is obtained from IC 7812 and IC 7912 by using a transformer of 15-0-15V of 1Amp.

To calculate the volume of powders/porous solids we are using the solenoid valves. The experimental set up consists of Air/Gas inlet, vacuum pump, reference chamber and sample chamber. First the valve-2 is closed, valves 1 and 3 are open then both chambers are in evacuated state. Valve 1 and 3 closed and valve 2 open then we get the pressure P1. Last one is valve -1 & 2 closed and valve 3 open then both the reference and sample chambers are equilibrated to pressure. Then the process is repeated with sample in the sample chamber. The solenoid valves are controlled with a port3 of microcontroller.

Port 3 Mapping

Port 3.7	Port 3.6	Port 3.5	Port 3.4	Port 3.3	Port 3.2	Port 3.1	Port 3.0
AUX 1	SOL 3	SOL 2	SOL 1	INT1	INT0	TXD	RXD

So whatever be the values are stored in the sample chamber are calculated by using the values of the sensor. To match the output of a sensor to A/D conversion a signal conditioning circuit is required. The sensor output is given to the INA125 which is used as the low precision instrumentation amplifier. U13, 1 & 3 pins are connected to +12V and -12V respectively. The main function of the amplifier is to amplify i.e., strengthen the weak signal and then we get the output across the sensor which is in the form of analog.

Analog signal cannot be given directly to the micro controller. So the ADC 3208 used for level matching, which is a 12 –bit and consists of 8-channels. The CS and CLK signal should be in the high state when ever the signal is received on the DIN pin and the output Dout is connected to PO.5 ADC is calibrated as the ratio of full range voltage to number of bits multiplied by number of states of ADC. i.e. $\left(\frac{5.06}{4096}\right)\times 12 = 0.147$ Therefore 0.147V is subtracted from analog output.

Port 0 Mapping

Port 0.7	Port 0.6	Port 0.5	Port 0.4	Port 0.3	Port 0.2	Port 0.1	Port 0.0
CS	DIN	DOUT	CLK	MODSW	SELSW	DECSW	INCSW

Port0.0 to port0.3 of microcontroller are connected for key pads. The circuit consists of 4 switches INC, DEC, SEL and MOD which are connected to PO.0, PO.1, PO.2 and PO.3 which is used to selected the mode and set the reference values. Port0.0 to increment (AD0), port0.1 for decrementing (AD1), port0.2 for selecting (AD2) and port0.3 for mode(AD3) of operation.

Port0.4 to port0.7 of microcontroller are connected for ADC for the following purpose respectively. Port0.4 for clock (AD4), port0.5 for DOUT (AD5), port0.6 for DIN(AD6) and port0.7 for CS(AD7).

Whenever the values are set these are stored in the EEPROM and that data is send through SCL and SDA which are connected to P2.2 and P2.3. The serial clock signal should be in the high state whenever the data is transferred.

After that the values are displayed on the LCD. The LCD is used in the 4-bit mode to reduce the connection and these values are send to the experimental to the PC through the MAX232.

Port 2 Mapping

Port 2.7	Port 2.6	Port 2.5	Port 2.4	Port 2.3	Port 2.2	Port 2.1	Port 2.0
DB7	DB6	DB5	DB4	SDA	SCL	En	Rs

Before using the LCD, it is initialized and configured. This is accomplished by sending a number of initialization instructions to the LCD. The first instruction we send must tell the LCD whether we'll be communicating with it with an 8- bit or 4-bit data bus. We also select a 5x8 dot character font. These two options are selected by sending the command 38h to the LCD as a command. The KS line must be low if we are sending a command to the LCD.

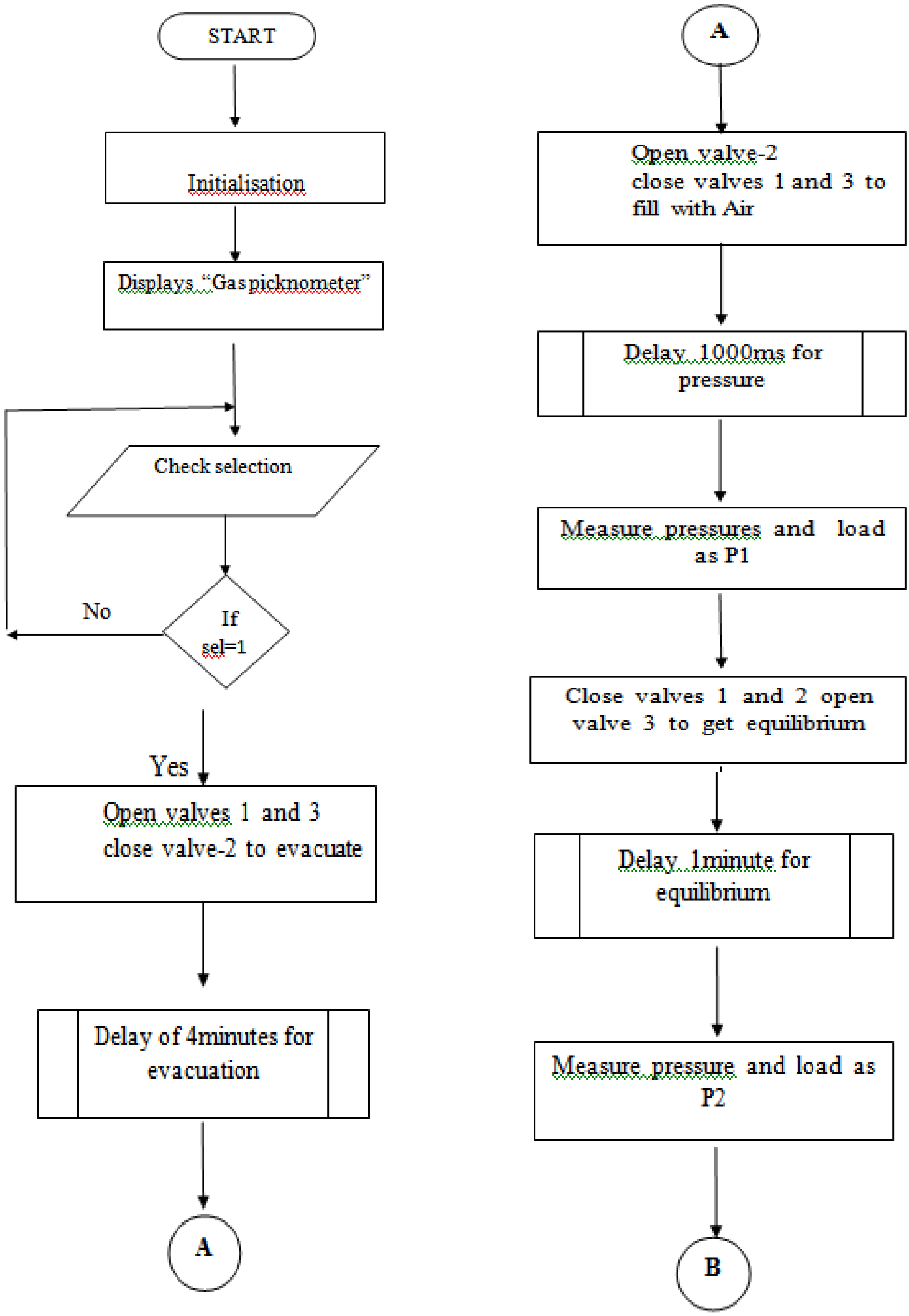
C. Software development

With an advent of microcontroller, it has become quite easy to add intelligence and computing power to any instrument, machine, process or system. The levels of intelligence and computational power that can be added to a microcontroller based system depends on the memory capacity, software support and the interfacing arrangements actually provided.

The design of a microcontroller based system has to solve two problems:

(1) Interfacing problems associated with interfacing hardware and
(2) The software problem associated with the application requirements.

It is also very essential to develop user friendly S/W for any application. The development of interactive S/W simplifies the operator's job very much and also enables error-free operation of the system as represented in flow chart figure 5.


START
Initialisation
Displays "Gas picknometer"
Check selection
If sel=1
No
Yes
Open valves 1 and 3 close valve-2 to evacuate
Delay of 4minutes for evacuation
A
A
Open valve-2 close valves 1 and 3 to fill with Air
Delay 1000ms for pressure
Measure pressures and load as P1
Close valves 1 and 2 open valve 3 to get equilibrium
Delay 1minute for equilibrium
Measure pressure and load as P2
B

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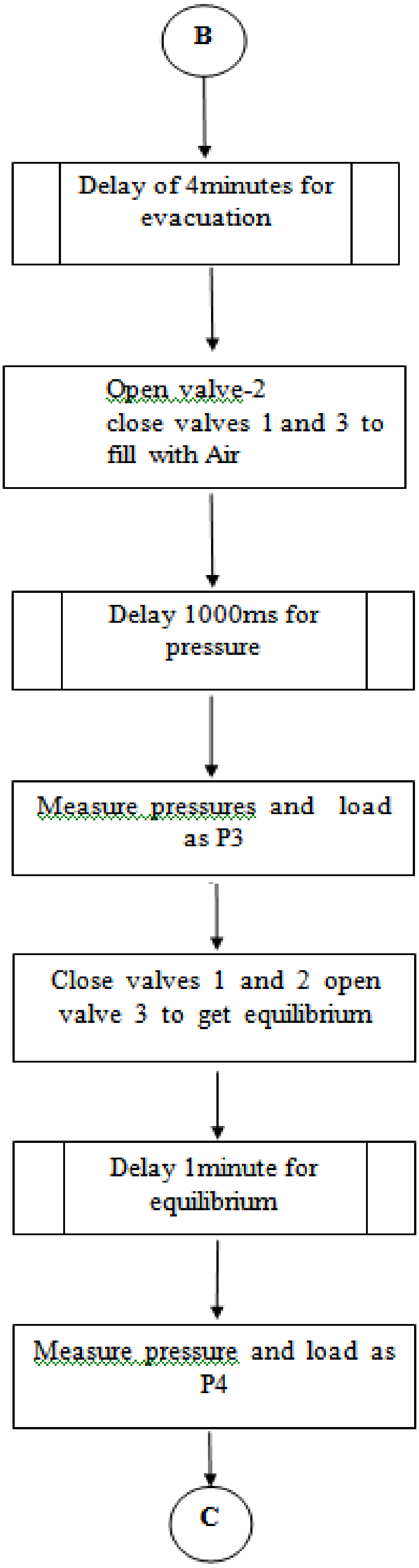

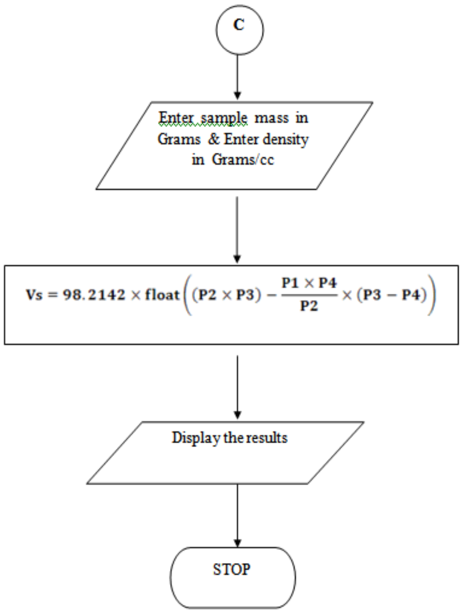

Fig.5. Flow Chart of software

The system software is developed using RIDE (Resonance Integrated Development Environment). Which is a cross complier and is divided into four stages of two different cases. The Hex code is dumped written into microcontroller by having Tx to pin 2, 3rd pin to Rx and the 7th pin to GND of MAX232.

A. Case 1: (With out Sample)

Stage 1

Once the power is on, the LCD gets initialized then the functions of EEPROM, ADC and Interrupt are processed. Then all the 3 solenoid valves are opened then the value is 0X00. Display of “Gas Picknometer” then “PRESS SEL TO START” appears.

Stage 2

Select 1 by using a switch to send 0X50 to port p3. Thus the valves 1 & 3 opened and the valve 2 closed to evacuate both the reference and sample chambers. This gets continued for 4 minutes and the pressure value will be simultaneously observed on the LCD.

Stage 3

Now by sending 0X20 to port p3, the valves 1 & 3 closed and the valve 2 opened to fill with air by an air compressor for that the delay of 1000ms maintained. Thus the pressure p1 in sample chamber is noted.

Stage 4

Now by sending 0X40 to port p3, the valves 1 & 2 closed and the valve 3 opened to get the equilibrium position of both the chambers with in a minute and the corresponding pressure noted is p2. The display of “PRESS SEL AFTER SAMPLED” appears to further proceed with the sample to put in a sample chamber.

B. Case 2: (With Sample)

Stage 1

Once the pre weighed sample is taken in sample chamber and select the switch. Then all the 3 solenoid valves are opened.

Stage 2

Select 1 by using a switch to send 0X50 to port p3. Thus the valves 1 & 3 opened and the valve 2 closed to evacuate both the reference and sample chambers. This gets continued for 4 minutes and the pressure value will be simultaneously observed on the LCD.

Stage 3

Now by sending 0X20 to port p3, the valves 1 & 3 closed and the valve 2 opened to fill with air by an air compressor for that the delay of 1000ms maintained. Thus the pressure p3 in sample chamber is noted.

Stage 4

Now by sending 0X40 to port p3, the valves 1 & 2 closed and the valve 3 opened to get the equilibrium position of both the chambers within a minute and the corresponding pressure noted is p4.

Once the display of “Enter Sample Mass in grams”, enter the mass of the corresponding mass of a sample (which is already a known value) with a switch. Similarly the display of “EnterDensity in grams/cc” to enter the density. Finally all the following calculated parameters will be displayed as “Density”, “Differential density” and “Factor of densitizations”.that are depicted in table 1.

Table 1: Parameters Obtained

S.No	Name of the Sample	Pressure Values in Psig (P1, P2, P3, P4)	Sample Mass in gms	Absolute Density of Sample in gms/cc (D_s)	Sample Volume (V_s) cc	Sample Density (D_p) gms/cc	Difference of Densities (D_s - D_p)	Factor of Densiti zation in %
1	Aluminium Pieces	29,15,29,14	29.61	2.71	12.658	2.34	0.37	86.346
2	Aluminium metal block	28,15,28,14	35.30	2.8	13.095	2.695	0.105	96.25
3	Copper pieces	28,15,29,14	150.52	8.93	18.769	8.02	0.91	89.80
4	Brass	26,14,28,14	63.97	8.5	14.030	4.56	3.94	53.647
5	Stainless steel (304)	25,15,28,13	190	7.93	24.444	7.8	0.13	98.36
6	Water filter candle	26,14,27,13	7.623	0.3965	20.043	0.38	0.016	95.83
7	Clay pot pieces	27,14,27,12	24.91	1.362	25.25	0.986	0.373	72.52

The following formulae are used for the calculations.

Sample volume (Vs) =V_1 (($P_2P_3 – P_1P_4$) / $P_2(P_3 – P_4)$)

Where V_1 = 3.14 x 2.5 x 2.5 x 5 = 98.2142 cc.

Volume of two chambers is converted from mm into cms.

Since the chambers size is 50mm height and 50mm diameter

Therefore, Radius r = $\frac{50}{2}$ = 25 mm = 2.5 cm

Similarly Height h = 50 mm = 5cm

Therefore Volume of the chamber (V_1) = x r^2 x h = □ x 2.5 x2.5x5= 98.2142 cc.

Sample Density (D_p) = (Mass of the sample / volume of the sample) gms/cc

Difference of Densities = Standard Density of Sample (D_s) - Sample Density (D_p)

Factor of Densitisation In % = (Sample Density(D_p) / Standard Density of Sample (D_s))

Features of the obtained parameters reveal that

Sample 1: Is small aluminum pieces of irregular shapes Since the large surface area and irregular shapes of the cut pieces, the theoretical density and the actual density differ to a large extent.

Sample 2: Aluminum metal block: since the block is cut from a cast aluminum ingot certain porosity present in the cast samples is the result of the deviation for 100% density. Generally aluminium alloy of high strength variety has 2.8 density.

Sample 3: copper pieces are obtained by cutting copper wire into 2-5 mm pieces. Here again due to high surface are of the cut pieces of irregular lengths the density can never reach their theoretical density.

Sample 4 : Brass metal turnings pulverized in a mortar is taken. The material is almost in powder form (coarse) the density is low.

Sample 5: Stainless steel block: The actual density and experimental value differ by about 1.64% due to the experimental errors only. To avoid such errors a highly accurate weighing balance and accurate pressure measuring transducers which are highly expensive are to be employed.

Sample 6: Water filter candle pieces: The density of the material is approximately 95% with about 5% porosity that filters dust particles from water.

Sample 7. Clay pot pieces have much higher porosity about 25%..These are illustrated in figure 6

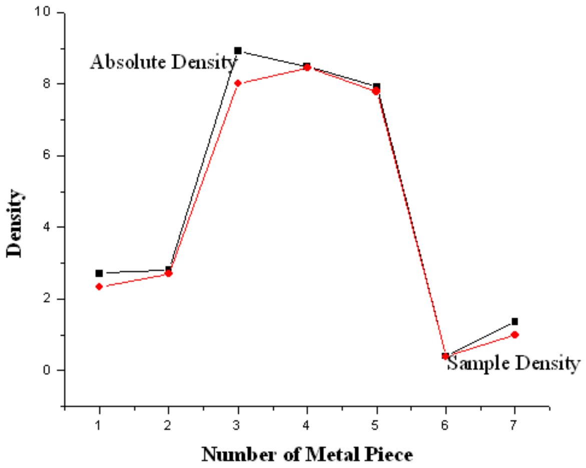

Fig.6. Illustration of densities

IV. Conclusion

In many applications a certain amount of porosity is designed in material processing and fabrication. The pore structure is employed to impregnate a certain material or lube component to avoid friction, to provide diffusion of gaseous component, or to filter out any unwanted material into a process stream. Consolidation of materials is required in some applications. In every such application the extent of porosity is to be determined within certain range. In many cases a rough (approximate) estimation will suffice. The low cost gas pycnometer described above will certainly find greater acceptance due to simplicity of design and low investment.

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Author's Profile

Dr. Ravi Shankar Kumar.Ch

is associated with frontier areas of research in Materials science and published 14 papers in national and International journals as corresponding author and further with 30 papers published in conferences of national and International repute. The author is reviewer of peer reviewed journals and member of various academic and professional bodies and peer teams.

B. Balaji Bhanu

M.Sc., M.Phil., M.Tech. an experienced person of 21 years in teaching for graduates and post graduates with rich experience in the design of electronic circuits for physical applications. To his credit he had presented papers at both National and International conferences along with papers in peer reviewed journals. Presently pursuing for doctoral degree in electronics and computer engineering.

B.V. S. Goud

is Professor in Department of Electronics and Instrumentation guiding number of students for higher degrees. His experience and quality of research leads to develop interest in the field of design of electronic circuits and technology. He had published number of papers in national and International journals further with invited talks and guest lecturers in various conferences and seminars of repute.