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Technical Note

Microcontroller based Automated Freeze Thaw Instrument

by

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Abstract

We have developed an Automated Freeze-Thaw (AFT) instrument for the separation of minerals by desegregating the rock samples. Conceptually, the instrument uses natural expansion and contraction of rock samples by separating the inclusions from mineral edges. Multiple freeze-thaw cycles, by alternately dipping the sample in heating and cooling elements reduce the matrix fragments to micron-sized grains. The main objective of designing this instrument is to gently separate presolar grains from meteorite samples. The AFT automatically moves the sample from heating element (hot bath) to a cooling element (liquid nitrogen) with a certain time gap based on the time required to heat or freeze the sample. This instrument is operated over a number of cycles using a dual phase industrial grade NEMA23 stepper motor and DMS542 motor driver and the overall system is controlled by a 8-bit microcontroller (ATmega2560). The firmware was developed using Arduino IDE.

Keywords

Microcontroller — Stepper Motor — DC-DC Buck converter – Meteorites – Presolar Grains – Desegregation

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Contents

1. Introduction

Automated Freeze-Thaw (AFT) is a standard separation method widely used in all aspects of research in science. Some of these include Gas Chromatography, Decellularization of Large Tendon Specimens, Desegregation of Meteorites, etc. Being a repetitive process, it requires precise and stable automation. Microcontrollers are used to achieve this automation. For our application, we are using the AFT for desegregating extraterrestrial samples such as meteorites.

Meteorites are extraterrestrial rock fragments reaching the earth surface, mainly coming from the asteroid belt. The fine-grained matrix of primitive meteorites hosts tiny dust grains of several nanometers to a few microns in size, with highly anomalous isotopic compositions and very low abundances. These dust grains formed in the winds of massive stars and the ejecta of novae and supernovae explosions before the formation of our solar system and are hence called presolar grains. The chemical and structural compositions of presolar grains provide insights into the stellar grain formation environments and various alteration mechanisms in the ISM, protoplanetary disk and on the parent body of meteorites.

Laboratory methods have found carbon-rich presolar

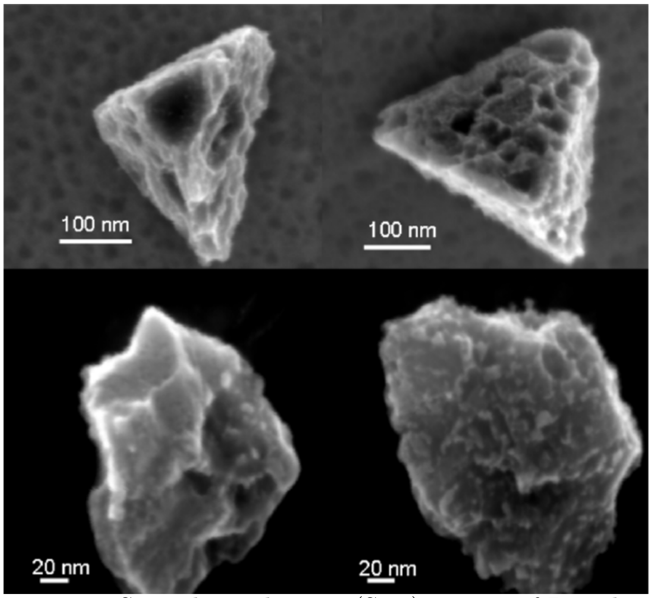

Figure 1. Secondary Electron (S.E.) images of Presolar Silicon Carbide (SiC) grains isolated from meteorites by Chemical Separation method. Irregular grain surfaces are the result of chemical etching

phases such as the silicon carbides (SiC), graphites, nitrogen-rich silicon nitrides (Si_3N_4), oxygen-rich oxides and silicates in primitive meteorites. Refractory presolar phases like SiCs and oxides are isolated from primitive meteorite matrix by dissolving solar system silicates using acid treatments, called chemical separation of presolar grains [2]. Harsh acids used in this method etch the grain surfaces as shown in figure 1, altering grains' morphology and surface chemical composition. In addition, small presolar grains might get destroyed during the chemical separation. An alternative to this destructive separation method is the Freeze-Thaw disaggregation, known as the gentle separation of presolar grains [3].

Instead of using acids and other chemicals, this method defragments the meteorite sample by applying variation in temperature. The meteorite sample is dipped in liquid nitrogen and a hot water bath, continuously expanding and contracting the rock sample. The mineral edges within the sample act as flaws through which the ultrapure water flows. The periodic pressure produced using contraction and expansion leads to the breakage of minerals from these edges. The meteorite sample can be defragmented into very fine pieces. Different minerals within the sample can be further separated from the fine-grained meteorite matrix using heavy liquids having different densities.

The process of dipping the meteorite sample in hot bath, then in liquid nitrogen repeatedly is a labor intensive process, and may be hazardous at times. To overcome this issue we have made an 'Automated' Freeze Thaw Instrument, which can perform this repeated action of dipping the sample in hot bath and in Liquid nitrogen repeatedly and in unattended mode, with precision in timings. In this technical report, we shall discuss the construction of the Automated Freeze-Thaw (AFT) Instrument and describe various control features that can make the instrument user friendly.

2. Algorithm of Operation

AFT works on the principle of contraction and expansion of the sample matrix. Our system is designed to move the sample from a hot plate to liquid nitrogen, selected as heating and cooling elements respectively. For this purpose, two bipolar stepper motors were set up such that one of the motors control the arm to move the sample, and another is used to control the thread that moves the sample up and down towards the heating/cooling elements. This has been indicated in the schematic diagram (Figure 2).

1. Rotate the vertical motor in 3000 steps to dip Teflon vial in Liquid Nitrogen bath.
2. Pause operation to Freeze sample for a 1 minute..
3. Raise the Teflon vial from the freezing bath 3000 steps (upwards).
4. Pause the mechanism for 10 seconds to minimize swinging of the sample vial.
5. Rotate the horizontal motor to swing the arm in 100 steps to rotate it by 90 degrees towards the hot plate.
6. Pause the mechanism for 10 seconds to minimize swinging of the sample vial.
7. Rotate the vertical motor in 2000 steps to dip the Teflon vial into the hot water bath.
8. Pause operation to heat the sample for 3 minutes.
9. Raise the Teflon vial from the hot bath in 2000 steps (upwards).
10. Pause the mechanism for 10 seconds to minimize swinging of the sample vial.
11. Rotate the horizontal motor to swing the arm in 100 steps to rotate it by 90 degrees towards the Liquid Nitrogen.
12. This cycle is repeated again and again for required number of times.

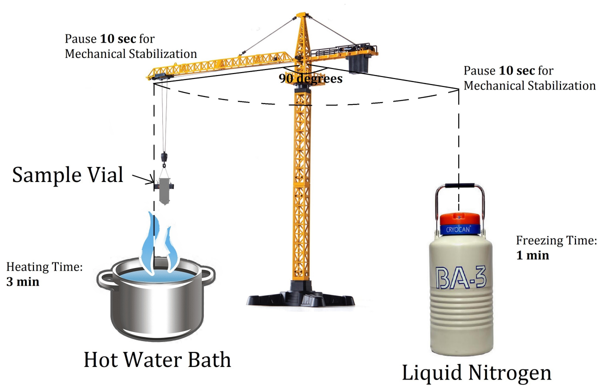

Figure 2. Schematic diagram of the algorithm of operation of the Automated Freeze-Thaw (AFT) instrument including the major elements

As we have placed the hot bath 30 cm above the level of the instrument, we have to modify the code to place sample vial exactly at the hot bath. So effectively, the sample vial is to fall 90 cm from the pulley to be dipped in the liquid nitrogen and 60cm from the pulley to be dipped in the hot bath. For the same, we have chosen steps size of 3000 and 2000 for cryogenic can and hot bath respectively. The 10 second pause is to reduce the mechanical swinging due to movement of the sample vial by the motors. The separation of 90 degrees is a trade-off between minimizing swing time of the horizontal motor and keeping a safe distance between Hot bath and Cryogenic can.

Since our aim was to defragment the meteorite sample into micron size particles, the sample must experience the freezing and heating in such a way that contraction and expansion works efficiently to defragment the meteorite sample. Following the experimental procedure by Clarke 2018[10], we initially selected the heating time and freezing time of 2 minutes each, with the heating temperature of 65 ºC and the freezing temperature of liquid nitrogen. But after several cycles, we observed that the heating time and temperature were not sufficient to completely melt the frozen sample. Therefore, after several trials we increased the heating time to 3 minutes, simultaneously we also increased the heating temperature to 80 ºC. Later we decreased freezing time to 1 minute as it was sufficient to appropriately freeze the sample. With these parameters, we could efficiently heat and freeze the rock sample. Also, we did at least 400 freeze-thaw cycles to be sure that no big aggregates of the rock samples were remaining.

With the algorithm of operation quite clear, it is the implementation that needs focus. There are various aspects of implementation, which we shall discuss in great detail. They include:

- Mechanical Design and Structure
- Electronics Control and Power Supply
- Additional Features

3. Mechanical Design and Structure

The objective of the AFT is to have repetitive cycles of heating and cooling. For achieving this objective there are two methods/designs:

Heat exchange pipes: In this design the sample is to remain stationary, while the Liquid Nitrogen and Hot water bath are alternatively flown through pipes to freeze/Thaw the sample.

Crane Motors: In this design, the liquid nitrogen and hot water bath are to remain stationary, while the Sample vial would be moving and immersing itself in the hot bath and cryogenic can. The movement is typical to that of a crane used at construction sites.

Both the designs have their advantages and disadvantages.The Heat exchange pipes design ensures the safety of the sample, whereas the Heat exchange pipes are not an effective in transferring heat. Besides that, for flowing liquid nitrogen, special cryogenic pipes are required, which can withstand the freezing Liquid Nitrogen. Also specialized pumps are required with with the rating of liquid nitrogen.

On the other side, The Crane motors design, keeps both the extreme temperatures stationary and removes the requirement of specialized pipes and pumps for flowing liquid nitrogen. To withstand the extreme temperatures of Freeze and Thaw, the sample vial is made up of Teflon and the thread connected to the vial is made up of Nylon. With the use of bipolar stepper motors, the required precise movement of the sample vial can be achieved. The only disadvantage of using the Crane motor mechanism is minute possibility of damaging the sample due to mechanical movement and vibration.

On considering advantages and disadvantages these 2 designs we found the Crane motor design more suitable for our application and hence we chose to implement the same.

3.1 Mechanical Structure

A heavy base plate is used to hold the structure, which supports the central beam. The height of the central beam is 96 centimeters. On top of the beam, the motor rotating the arm of the crane is placed. It is named as the 'Horizontal' Motor. The crane's arm holds the Nylon string, which holds the sample vial on the free end. The sample is pulled up and down via the string with the help of another stepper motor, which is the 'Vertical'

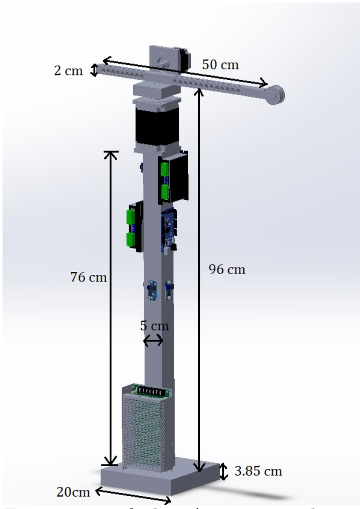

Figure 3. Drawing of the Automated Freeze-Thaw mechanism using the CAD software Solidworks

Motor. The vertical and horizontal motors control all the movements of the crane structure.
The whole structure is analyzed using the CAD Software Solidworks. The Solidworks drawing is shown in Figure 3.
The hot water bath and the cryogenic can filled with liquid nitrogen are placed directly under the crane arm, where the sample vial moves up and down. All the Electronic components are attached to the central beam according to the ease of connections. The whole set-up is covered from sideways, hiding the wiring.

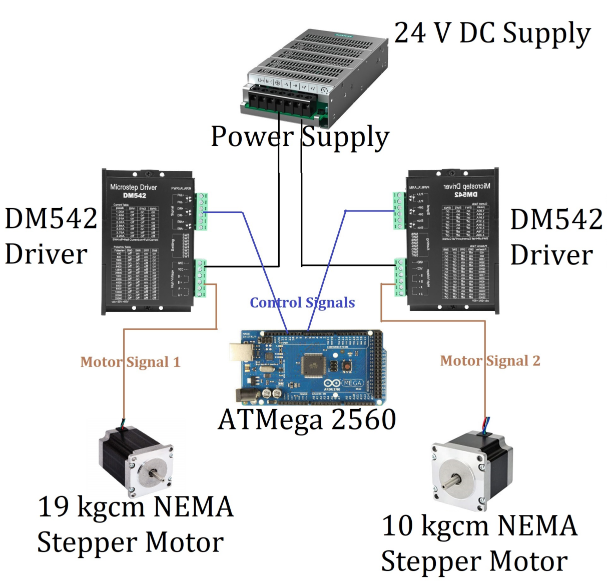

Figure 4. Schematic diagram representing the electronic controls of motors, including the three main components: power supply, motor drivers and the microcontroller

4. Electronic Controls and Power Supply

4.1 Electronic Controls

4.1.1 Stepper Motors

Motors are the heart of our design. Amongst DC, Stepper and Servo motors, we have chosen stepper motors since high precision and more torque is needed for our experiment.Through the Mechanical structure and Solidworks, we were able to determine the ratings of the motor required. The moment of inertia of the vertical motor and the arm structure was approximately 2.5 $kgcm^2$, and the sample vial was approximately 1.6 $kgcm^2$. For the same, we chose industrial-grade NEMA stepper motors 19 kg-cm and 10 kg-cm, respectively, considering the safety factors in the drive mechanism.
The NEMA23 DC Stepper Motor is a heavy-duty industrial grade bipolar motor, with 4 wires controlling its motion. The 4 wires correspond to 2 sets of electromagnet coils, which magnetize based on the input from the 4 pins of motor driver. The magnetized coils align the permanent magnet rotor to a fixed position. A dual H-bridge drive controls the 4 wires of the stepper motor. The stepper drivers DM542 and Rhino Motor Drivers have embedded MOSFETs, which act as switches for the dual H-bridge.

4.1.2 Stepper Motor Drivers

As mentioned earlier, the Stepper motor driver uses a dual H-bridge configuration to translate the instructions of the microcontroller to current magnetizing the electromagnet. The connections of the stepper motor driver with the microcontroller module and power supply are shown in figure 4.
The Stepper motor drivers also come with an in-built current regulator circuit, which can be manually set. The settings are manipulated using dip switches located between the screw shield pins. With the help of the Switch configuration of the Driver, we have to set the maximum current load to 2.8 Amps, which is the maximum rated phase current as per the datasheet of the motor. The Drivers also come with a heat sink to dissipate the heat when operating in a long continuous fashion.

4.1.3 Microcontroller

The microcontroller used is an ATmega2560 chip. It is preferred over other microcontrollers due to its long term stability and adequate digital and I2C pins required for controlling 2 motor through their drivers and displaying current status cycle number of the AFT instrument through a LCD Display. Further, it will require more pins to accommodate Temperature sensors and numeric keypads in the future upgrades.
The microcontroller is used to control the stepper motor through driver by inputting the Enable, Direction and Pulse control information. Enable pin shares the operational condition of Motor whether it should run or

not. Direction pin shares the direction of rotation of the motor, whether clockwise or anti-clockwise. The Pulse pin shares the frequency of motor rotation. The Pulse frequency is translated to Revolution Per Minute (RPM) with the following formula:

$$RPM = \frac{step\ angle}{360} X pulse\ freq X 60$$

We chose a time period of 1.8 milliseconds for our requirement, which translates to an RPM of 83.33. These parameters can be further fine-tuned using the microcontroller. The schematic of motor control is shown in Figure 4.

4.2 Firmware and Programming

The Firmware used was Arduino IDE version 1.8.13. The programming logic used in the firmware, is to define and control, pulse, direction and enable pins to control the motion of the motor. As stated earlier we have fixed the time period of pulse as 1800 microseconds, with a 50% duty cycle for all the motor movements. 50% duty cycle generates sufficient torque to lift the sample and rotate the crane arm. A flowchart of the programming logic is shown below.

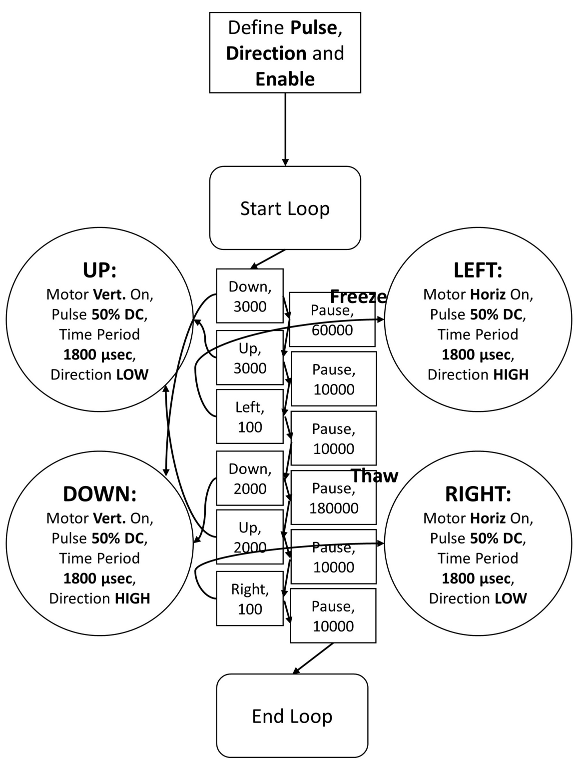

Figure 5. Programming Logic for Motor control

4.3 Power Supply Distribution

4.3.1 Industrial DC Power Supply

For continuous operation, a standard power supply is a simpler and better option than using batteries. The DC Industrial Power Supply converts the readily available 230V A.C. signal to our required D.C. voltage. Considering the fact that the phase current of 2.8 Amps that our motor consumes, it is best to have a power supply that can manage the current as well. Overlooking these factors, we have chosen an Industrial Power Supply of 24V D.C. Output and a maximum current rating of 6.25 Amps.

4.3.2 DC-DC Buck Converter

The Industrial Power supply steps down and converts 230V A.C. to 24V D.C.; however, the Motors require 18V D.C., and the microcontroller requires 12V D.C. To resolve this issue, we used a DC-DC tune-able buck converter. The DC-DC Buck Converter uses the concept of switching the input voltage on-off rapidly to reduce the effective voltage to our desired voltage.

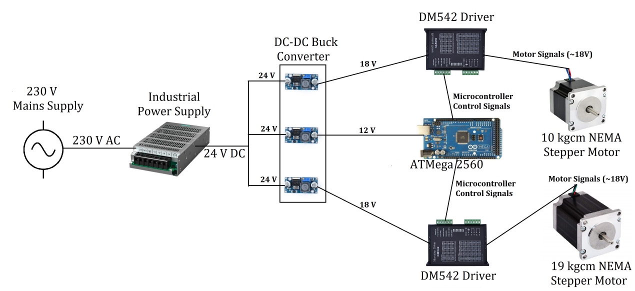

Figure 6. The schematic diagram showing the power distribution for the Automated Freeze-Thaw instrument

For our operation, we have used an LM2596 DC-DC Buck converter. This buck converter has a switching frequency of 150 kHz, with a maximum permissible current of 3 Amps. The Voltage conversions range from 4-40 V from the Input side to 1.25-35V on the output side. The output voltage is regulated by changing the load resistance (potentiometer) attached to the LM2596 module. The Power distribution layout as discussed above is shown in Figure 6.

4.4 Additional Features

4.4.1 Real-time Clock (RTC)

For having an independent clock, we have placed a real-time clock. In the future upgrades of the AFT instrument housekeeping parameters will be recorded. For the same an independent clock is required to note the timings of the parameter variations. The DS3231, a high-precision real-time clock module, has an accuracy of 2ppm, which amounts to an error of about 1 minute per year when operated in the °C range. Besides communicating the time, it also has an inbuilt temperature sensor, which accuracy of ±3°C. The DS3231 RTC is interfaced with the microcontroller via an I2C connection. The RTC runs on a single 3V CR2032 Lithium coin cell battery.

4.4.2 LCD Display

To view the operation status of the AFT, we have interfaced with a 16 x 2 LCD unit. The LCD unit can display 2 lines of 16 characters each. The first line shows the Date and Time of the operation, and the second line shows the Room Temperature and Cycle number of the AFT operation. A view of the LCD is shown in Figure 7.

We know that to connect LCD Display directly with the microcontroller will utilize 11 digital pins, 2 GND pins and a 5V pin. To overcome this issue of excessive pin usage for display and to provide over-voltage regulation for the LCD, we use an LCD I2C backpack with our LCD. This I2C Backpack uses a PCF8574 Remote 8 bit I/O Expander. It translates the data received from the I2C Bus into Parallel data needed for the LCD Display.

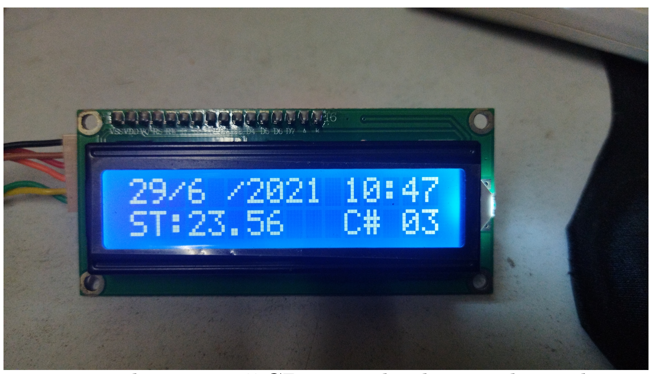

Figure 7. The 16 x 2 LCD unit, displaying the real-time parameters during the working of the Automated Freeze-Thaw instrument

5. Assembly and Operation

5.1 Development Procedures

On assembling the Mechanical and Electronic parts, the overall assembly looks like the one shown in Figure 9. The Electronic components were attached to the central beam sideways. A layer of electrical insulation was also introduced between the Components and the central beam. The wiring and assembling of connections were done.

The Horizontal Motor is screwed on 4 sides on top of the central beam. The shaft of the central beam holds the total weight of the crane arm and the Vertical motor. The Vertical motor holds the Nylon string's casing in its shaft. The whole arm structure is perforated with 15 mm holes to reduce the weight of the arm.

To indicate whether the AFT receives 230V A.C. from the power supply, we have placed an industrial grade high voltage red LED parallel to the input supply. This arrangement would help in troubleshooting, if any.

The whole of the central beam and all the components are enclosed in an aluminum casing. The casing considers the additional LCD and Power LED features placed at its desired location on the outer side of the casing. We also made an extra room for having a programming port on the external casing to enable changes to the primary code whenever required.

The hot bath and cryocan are aligned under the arm's pulley, with a sweep angle of 90 degrees.

5.2 Current Operational Status

Once we assembled the mechanical and electronic parts of the instrument and programmed the microcontroller, the AFT started operating successfully. However, the AFT had to undergo several rounds of fine-tuning and program debugging to match the electronic and mechanical requirements.

5.3 A Case Study Using the AFT Instrument

We utilized the AFT instrument to disaggregate two meteorite samples, Allende (carbonaceous chondrite-CV3) and Dhajala (ordinary chondrite- H3.8). Approximately 300 mg of a meteorite sample was taken in two different teflon vials, and 400 Freeze-Thaw cycles were done for each sample vial. Hence, 1600 AFT cycles were completed for two meteorite samples within a month of operation of the AFT instrument. The specifications for one Freeze-Thaw cycle is given below:

Cooling element: Liquid Nitrogen kept in a cryogenic can, Interaction time with the sample: 1 minute.

Heating element: Hot water bath kept at 80 ºC, for 3 minutes

Currently, the instrument is set such that one Freeze-Thaw cycle takes 5 minutes 3 seconds. The AFT cycles followed by density separation using a heavy liquid such as sodium poly- tungstate enabled us to separate graphite grains from the meteorite matrix. For example, Figure 8(B) shows a graphite grain separated from the Dhajala meteorite matrix using our AFT instrument at PRL.

5.4 AFT Instrument Datasheet

The Summary of the instrument is tabulated overleaf:

S. No.	Parameter	Value
1	Input Supply	230V Ac, 6A (max)
2	Mechanical Accuracy (Horizontal Motor)	0.9 degrees ±5%
3	Mechanical Accuracy (Vertical Motor)	0.3 millimeter ±5%
4	Timing Resolution	1 microsecond
5	Expected Lifetime	10 years

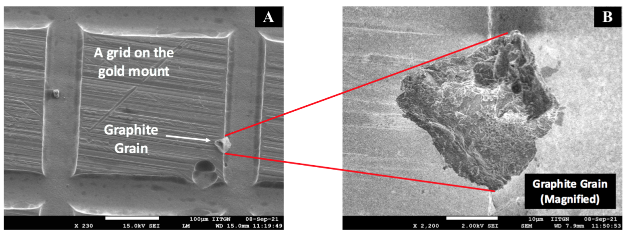

Figure 8. Graphite grain separated from Dhajala meteorite using AFT instrument. (A) Secondary Electron (S.E.) image of a graphite grain on the gold mount; (B) A magnified S.E. image of the graphite grain. (SEM analysis done at CIF, IIT Gandhinagar)

Figure 9. The Automated Freeze-Thaw instrument, after configured mechanically and electronically

6. Future improvements

There are more features we intend to add to the AFT Instrument. Firstly, it is important to know the accurate temperature the sample is experiencing in the Freeze and Thaw environment. We plan to add a versatile temperature sensor that would display the sample temperature when the sample is in the Freeze and Thaw state. This will help us to exactly know the required interaction time for freezing and thawing of the sample, eventually reducing the overall time for the complete defragmentation of the sample, without excessively heating or freezing it.
Liquid nitrogen is consumed in the sample freezing process. Eventually, on operating the AFT in continuous mode, the liquid Nitrogen may be get completely consumed. Hence the Cryogenic can will be required to refill. For the same, we plan to add a pause button externally. This external switch will pause the operation of the AFT indefinitely until pressed again. This will provide ample time for the user to refill the Cryogenic can.
In addition, we have also planned to include a numbered keypad attached to the front side of the AFT instrument such that it will allow the user to set the number of cycles without making a change in the program code.
Besides external user control, we have also planned to make a software to control the AFT instrument with the help of a computer. We would be using MATLAB to achieve the same.

Acknowledgments

We humbly acknowledge the contribution from PRL workshop, Mr Hitesh Vaghela and Mr Pramod Suthar in making the customized External casing of the AFT, which also has room for additional features.

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पीआरएल के
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समविष्ट हैं
पृथ्वी एवं
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