Mar Ivanios College-Instrumentation Centre

Mar Ivanios College became the first private college affiliated to the University of Kerala to receive the status of CPE (College with Potential for Excellence) from the UGC in the year 2007 with a financial grant of Rs.60 lakhs with the scheme being extended to its next phase during 2014 receiving Rs 125 lakhs. As part of implementing the CPE Scheme, the college has set up various facilities in the campus for the benefit of UG, PG and PhD students and Teachers. The Instrumentation Centre was established in 2007 with the financial support of UGC through the CPE scheme. Initially a Perkin Elemer make FTIR spectrophotometer was installed for Infrared spectroscopy studies for Research students in Physics, Chemistry, and life science. Subsequently more numbers of research instruments were installed in the centre. It include Superconductivity measurement set up, Keithley Current –voltage characteristics set up,30 Amp current source,120Amp current source, Impedance analyzer, sensitive electronic balance, furnaces and many more. A new version of table top FTIR spectrometer was also installed in 2014.The instrumentation facilities are utilized by UG,PG and PhD students of Mar Ivanios College as well as many other faculty members and students of nearby colleges and research institutions. A logbook is kept for recording the user's details. A brief description of the major instruments housed in the Mar Ivanios College Instrumentation centre is given below.

FTIR SPECTROMETER (with diamond ATR accessory)

Perkin Elmer Spectrum two FTIR spectrometer with diamond ATR accessory (Fig 1) is a reliable spectrometer. Attenuated Total Reflection Method is used for analyzing as prepared powder sample and transmission mode for analyzing mirror polished pellets. The instrument has a standard spectral resolution of 0.5 cm^{-1} and a wavelength precision better than 0.01 cm^{-1} at 3000 cm^{-1}. It is ideal for all type of samples (powder, film and liquid).

Figure 1 : Perkin Elmer Spectrum two with diamond ATR accessory

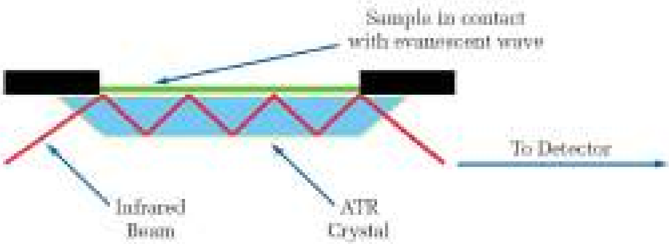


Figure 2 : Working of diamond ATR in FTIR spectrometer

An attenuated total reflection accessory measures the totally reflected infrared beam when the beam comes in contact with a sample. Internal reflection spectroscopy passes infrared radiation through an infrared-transmitting crystal of high refractive index, allowing the radiation to reflect in the crystal one or more times as shown in figure 2. The infrared radiation interacts with the sample through a series of standing waves, called evanescent waves. An evanescent wave is a penetrating electromagnetic field whose intensity quickly decays as it moves away from its source. An evanescent wave penetrates into the sample in contact with the crystal, producing a spectrum of the sample. In ATR mode no sample preparation is required as in transmittance mode as in KBr Pellet method. Most importantly, the improved spectral acquisition and reproducibility associated with this technique leads to better quality database building for more precise material verification and identification. ATR is clearly an extremely robust and reliable technique for studies involving solids and liquids.

HIGH TEMPERATURE CONVENTIONAL FURNACE

In conventional type furnaces the green pellet is indirectly heated using refractory type electrical resistance furnace, induction furnace or fossil fuel furnace. In conventional furnaces, heat energy is generated by the heating elements and transferred to the green body via radiation, conduction and convection. Figure 3 shows the high temperature furnace with molybdenum di silicide heating elements (TE- 4050, Therelek, India) Its upper limit of temperature is 1800 ^{o}C and heating rate can be varied between 1 and 20^{o}Cmin^{-1}. The temperature controller is able to maintain the temperature inside the chamber within a maximum error limits of $\pm 1^{o} C$.

Figure 3: High temperature conventional resistive furnace

MICROWAVE FURNACE

Microwaves are electromagnetic waves whose frequencies range from 300MHz to 300GHz. In general most of the cases utilizes 2.45 GHz microwaves for sintering purposes. Microwave sintering is now popular among the researchers all over the world as it has many advantages over conventional sintering. In microwave sintering the green pellet absorbs the microwave energy generated by the magnetrons and convert it into heat within. The absorbed microwave power is proportional to the magnitude of electric field distribution in the sample In microwave sintering, the densification commences at a relatively low temperature by enhancing the diffusion mechanisms. So the sintering temperature and the soaking time can be reduced considerably.

Figure 4: High temperature susceptor assisted microwave furnace

Figure 4 a microwave furnace with a pair of 2.45 GHz magnetrons with power 1.1kW each and silicon carbide susceptor (VBCC/MF/86, VB Ceramics Consultants, India) .Its upper limit of temperature is 1600^{o}C. The heating rate optimised for the present work is 40 ^{o}Cmin^{-1} and the soaking duration is 20 minutes. In this furnace a high quality pyrometer is used to sense the sample temperature. Considering the variations in the emissivity of ceramic samples the maximum error possible in the sample chamber as sensed by the pyrometer is

$\pm 10^{o}C$ .The temperature controller is able to maintain the temperature inside the chamber within a maximum error limits of $\pm 1^{o}C$.

MICROWAVE HYBRID FURNACE

Resistive coupled microwave sintering is developed to overcome the pit falls of the susceptor assisted microwave sintering. It is observed that in susceptor assisted microwave sintering, one has no control over the conventional sintering part. Because the silicon carbide is absorbing the microwave energy and eventually transferring the heat to the pellet in a conventional manner. At high temperature the sample is able to couple the microwave and results in a volumetric heating to achieve high density. In resistive coupled microwave sintering method the green body gets microwave energy directly from a pair of 2.5GHz magnetrons with power 1.1 kW each and the heating power rate can be controlled effectively. In addition to this the pellet is getting heat from a pair of Molybdenum disilicide heating elements as in conventional furnace which is also controlled according to the processing requirements. Uniformly distributed fast heating of the entire green body by effectively coupling the microwave and resistive power results in high quality infrared transparent ceramics with reduced grain size and minimum porosity which can be tailored to fabricate high quality infrared transparent windows cost effectively for demanding missions. In resistive microwave hybrid heating a reduction in sintering temperature, soaking duration etc are observed.

Figure 5 shows a resistive-microwave hybrid furnace VBCC/HMF/71,VB Ceramics Consultants, India with a pair of 2.5 GHz magnetrons with power 1.1kW each, a pair of molybdenum disilicide heating elements. Its upper limit of temperature is 1600 ^{o}C. The heating rate optimised for the present work is 40^{o}Cmin^{-1} and the soaking duration is 20 minutes. The proportion of coupling of the microwave power and resistive power is optimised for each sample and are explained in the forthcoming chapters. Considering the variations in the emissivity of ceramic samples the maximum error possible in the sample chamber as sensed by the pyrometer is $\pm 10^{o}C$ .The temperature controller is able to maintain the temperature inside the chamber within a maximum error limits of $\pm 1^{o}C$.

Figure 5: High temperature resistive coupled microwave furnace

HIOKI 3532-50HiTESTER

Figure 6 shows Hioki 3532-50 HiTester used to study the dielectric response of a sample at low frequencies.

Fig.6. Hioki 3532-50 HiTester

Hioki 3532-50 LCR HiTester features variable measurement frequencies over broad ranges. It can provide 50Hz-5MHz, for measurements with ±0.08% basic accuracy. It is used for laboratory applications such as for evaluating operating characteristics and with its 5ms fastest response, versatile interface options and comparator functions, and is also ideal for a broad range of production line applications.

IR Spectrometer

Microwave Furnace Tube Furnace

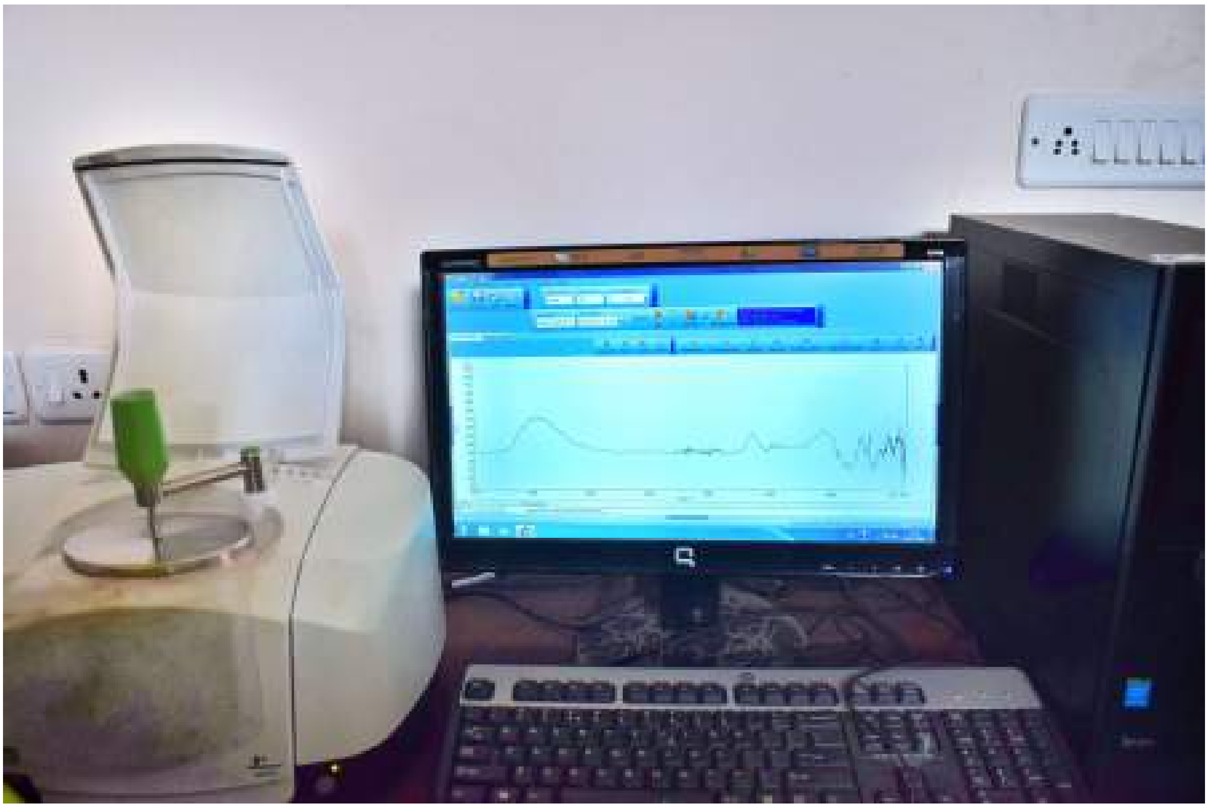
Impedance Analyser

Z-Scan

Muffle Furnace

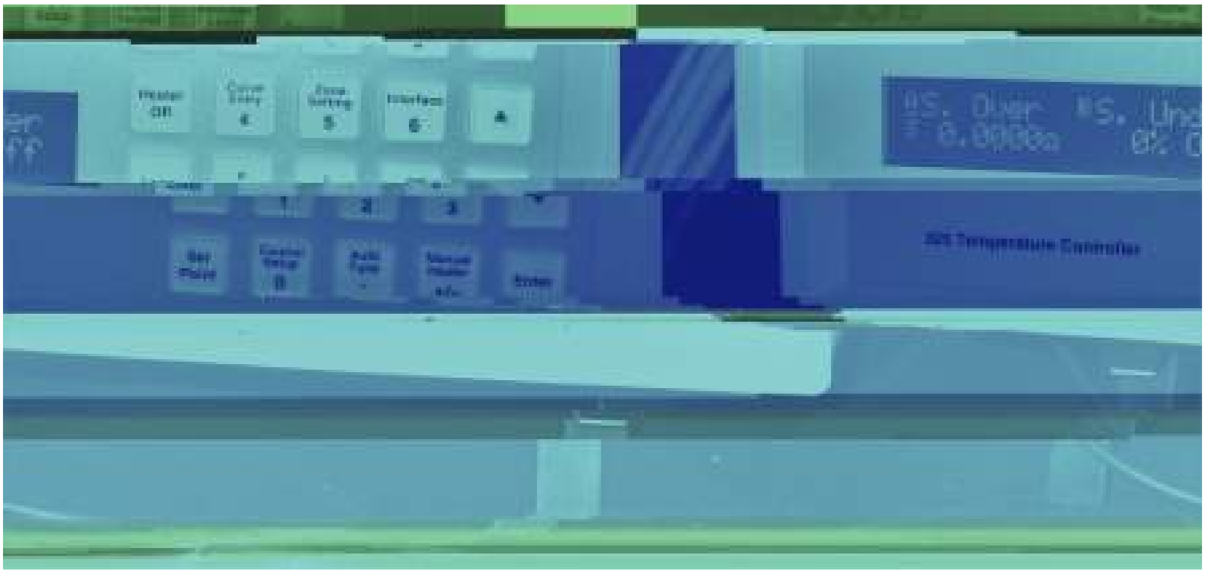
UV Spectrometer

Infrared Spectrometer



IR Spectrograph

Microwave Hybrid Furnace



Temperature Controller

Nanovoltmeter

Perkin Elmet IR Spectrometer

Superconductivity Measurements