

### 3.1 Material Synthesis

#### 3.1.1 Raw materials

Raw materials were procured from various manufacturers having purity more than 99%. Table 3.1 below enlists the chemicals used, their purity level and the names of manufacturers. The fine chemicals used here are mainly in the form of oxides, carbonates and phosphates.

**Table 3.1** The list of chemicals used with purity and names of manufacturer.

S. No.	Name of Chemical	Purity (%)	Manufacturer
1	Al <sub>2</sub> O <sub>3</sub>	99.5	Sigma Aldrich
2	Li <sub>2</sub> CO <sub>3</sub>	99	Sigma Aldrich
3	MgO	99	Sigma Aldrich
4	NH <sub>4</sub> H <sub>2</sub> PO <sub>4</sub>	99	Alfa Aesar
5	H <sub>3</sub> BO <sub>3</sub>	99.5	Thomas Baker
6	SiO <sub>2</sub> (Quartz)	99	Himedia
7	TiO <sub>2</sub>	99.5	Sigma Aldrich

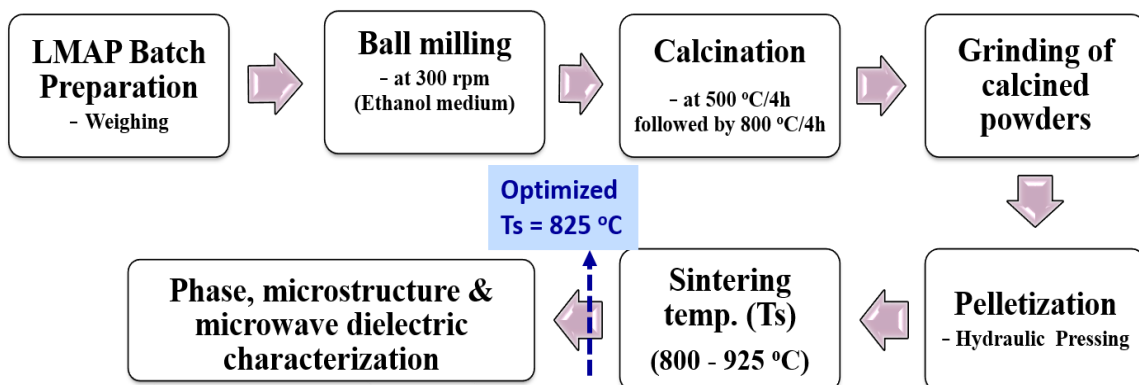
#### 3.1.2 Preparation of LMAP ceramics

Li<sub>2</sub>O–(2–3x)MgO–(x)Al<sub>2</sub>O<sub>3</sub>–P<sub>2</sub>O<sub>5</sub> (x = 0.00 – 0.08) (LMAP) ceramic was synthesized by conventional solid state route. High purity raw materials listed in Table 3.1 were used. Different LMAP batch compositions are given in Table 3.2. Stoichiometric amounts of raw materials were mixed thoroughly in ethanol medium for 8 h at 300 rpm using a Fritsch Planetary mill (Pulverisette 5/4). The ball milled batches were dried in oven and crushed using agate mortar and pestle to obtain a homogeneous fine powder. The batches were taken into the alumina crucible and were primarily calcined in air at 500 °C for 4 h

followed by second calcination at 800 °C for 4 h with intermediate grinding. Calcined powder was again ground into fine powder. The powder was pressed uni-axially to form cylindrical test pellets (diameter = 12mm, thickness = 2mm) using different dies made of stainless steel under the load of 120 kN. All pellets were sintered at different sintering temperatures ranging from 800 °C to 925 °C for 6 h with heating and cooling rate of 2 °C per minute. After optimisation of sintering temperature, rectangular pellets (23 mm × 11 mm × 6 mm) and triangular (each side length = 4 mm and height = 11 mm) pellets were also prepared and sintered at 825 °C for 6 h. Figure 3.1 shows the flow chart for solid state synthesis of LMAP ceramic composition.

**Table 3.2** Batch compositions for LMAP (x = 0.00 – 0.08) ceramics.

x	Formula
0.00	Li <sub>2</sub> O-2MgO-P <sub>2</sub> O <sub>5</sub>
0.02	Li <sub>2</sub> O-1.94MgO-0.02Al <sub>2</sub> O <sub>3</sub> -P <sub>2</sub> O <sub>5</sub>
0.04	Li <sub>2</sub> O-1.88MgO-0.04Al <sub>2</sub> O <sub>3</sub> -P <sub>2</sub> O <sub>5</sub>
0.06	Li <sub>2</sub> O-1.82MgO-0.06Al <sub>2</sub> O <sub>3</sub> -P <sub>2</sub> O <sub>5</sub>
0.08	Li <sub>2</sub> O-1.76MgO-0.08Al <sub>2</sub> O <sub>3</sub> -P <sub>2</sub> O <sub>5</sub>



**Figure 3.1** Flow chart of solid state synthesis of LMAP ceramic compositions.

### 3.1.3 Preparation of MgO–B<sub>2</sub>O<sub>3</sub>–SiO<sub>2</sub> glass-ceramics

**(a) Glass batch preparation:** Based on the glass composition (Table 3.3), weight percent (wt%) contents of each chemical were calculated from their respective mole fractions. The weighing of raw materials was done by using high precision digital balance with an accuracy of 0.0001g. Batch mixing was done using agate mortar and pestle.

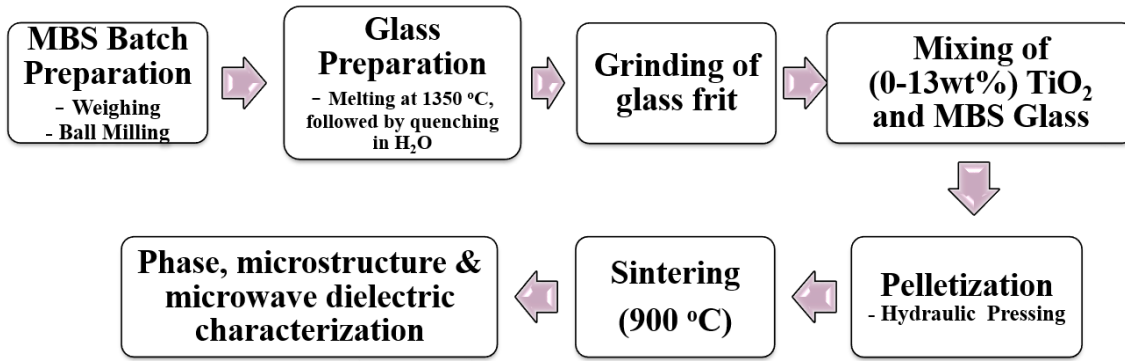
**(b) Glass melting and quenching:** After mixing batches thoroughly, they were transferred into a pure platinum crucible. The MBS glasses were melted in a raising hearth electric furnace. All the batches were transferred to the melting furnace at 1350 °C with the heating rate of 5 °C/min and kept for ~2 h to completely melt the chemical constituents followed by stirring the melt two times to attain homogeneity before its quenching in cold water. The obtained quenched glass called frit, was finely ground well for 6h and then was passed through a 200 mesh sieve.

**Table 3.3** MgO–B<sub>2</sub>O<sub>3</sub>–SiO<sub>2</sub> (MBS) Glass composition

Components	Weight %
SiO <sub>2</sub>	20
B <sub>2</sub> O <sub>3</sub>	45
MgO	35

**Table 3.4** Batch Compositions for MBS glass-ceramics.

Composition	Nomenclature Assigned
MBS Glass + 0 wt% TiO <sub>2</sub>	MBS0Ti
MBS Glass + 3 wt% TiO <sub>2</sub>	MBS3Ti
MBS Glass + 7 wt% TiO <sub>2</sub>	MBS7Ti
MBS Glass + 11 wt% TiO <sub>2</sub>	MBS11Ti
MBS Glass + 13 wt% TiO <sub>2</sub>	MBS13Ti

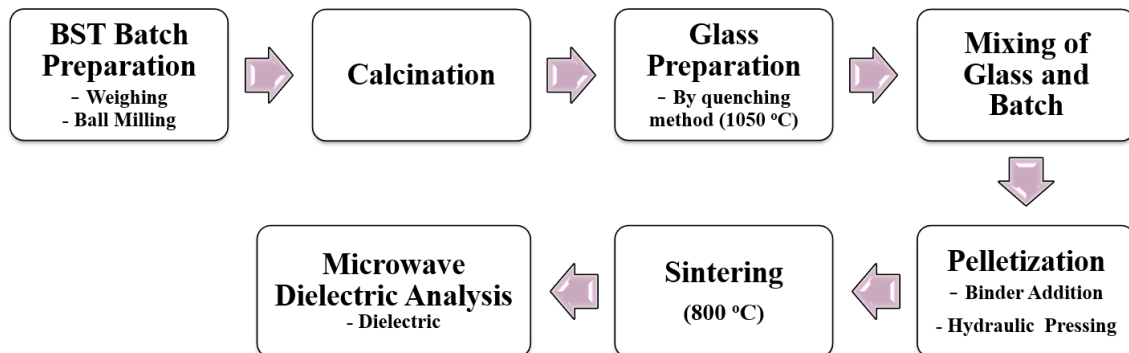


**Figure 3.2** MBS glass ceramic preparation using melt quench method.

**(c) Glass ceramic preparation:** Five batches were prepared by adding TiO<sub>2</sub> in different concentrations (0 – 13 wt%) to the obtained MBS glass powder. The composition of each batch is described in Table 3.4. The test pellets of 12 mm diameter and ~2mm thickness were prepared using hydraulic press for each batch and sintering is performed at 900 °C for 3 h (Figure 3.2).

### 3.1.4 Preparation of glass added barium strontium titanate (BSTG):

Preparation of 60PbO-5BaO-20B<sub>2</sub>O<sub>3</sub>-15SiO<sub>2</sub> glass was done by melting at 1050 °C followed by quenching. 10 wt% of this glass was added to Ba<sub>0.5</sub>Sr<sub>0.5</sub>TiO<sub>3</sub> ceramic and it is named as BSTG. The calcination temperature of this ceramic is 1000 °C. The sequential steps for BSTG preparation are shown in Figure 3.3. The sintered BSTG ceramic shows average  $\epsilon_r$  – value of 27 and tan $\delta$  of 0.121 which have been used for the design of DS–CDRA and DS–RDRA as one of the resonating segment [Tripathi et al. (2015a)].



**Figure 3.3** BSTG ceramic preparation using liquid phase sintering.

## 3.2 Material characterisation

### 3.2.1 Thermal behaviour analysis

**(a) Differential thermal analysis (DTA) or Thermo-gravimetric (TG) analysis:** It is a technique in which reaction properties of the material are measured as functions of temperature where the material is subjected to an automated temperature program. It is performed to study the thermal reaction between the constituents of the composition and decomposition behavior of the material. For the case of TiO<sub>2</sub> added MBS glass, different parameters like glass transition temperature, crystallization temperature and glass melting temperature can be determined. DTA/TG analysis was performed using a Mettler Toledo (STAR system) DTA/TG machine within the temperature range 30 – 1200 °C at a heating rate of 5 °C/min under argon atmosphere.

**(b) Thermal expansion (or CTE):** A rectangular bar with dimensions 50 mm × 10 mm × 5 mm was prepared for thermal expansion measurement using pushrod dilatometer (M/s V.B. Ceramics and Consultant, Chennai, India). To perform a thermal expansion analysis, a sample is placed inside a moveable furnace. A push-rod placed directly against the sample transmits change in length to a linear variable displacement transducer (LVDT). The temperature program is normally controlled using a thermocouple located next to the heating element or sample. Rod-type sample holders of fused silica were used. The measured change in length of the sample includes both the sample holder expansion and the change in length of the sample itself. Automatic correction was made in the measurement against the expansion behaviour of reference fused silica. The coefficient of thermal expansion (CTE) is determined by comparing the change in sample length with its initial length over a temperature range. The percent thermal expansion was measured from room temperature to 700 °C at a heating rate of 10 °C/min for different samples.

### **3.2.2 Phase and microstructure**

Powder X– ray diffraction (XRD) patterns of the sintered sample were recorded using Rigaku X– ray diffraction with Cu K–alpha radiation employing a Ni filter with scan rate of 2° per minute. The XRD technique was utilized for phase identification. The peaks of the measured pattern were matched with the standard JCPDS files.

Scanning Electron Microscopy (SEM) or Energy dispersive X-ray Spectroscopy (EDS) was performed for the sintered pellets. The surface morphology and energy dispersive spectra of the sintered sample were carried out using Scanning Electron Microscope (ZEISS Instruments). Sintered pellets either after polishing on the SiC emery papers of different grades or as sintered pellets were first gold plated and then placed for SEM imaging.

For glass-ceramic samples, all the pellets were first polished thoroughly with SiC emery papers of different grades along with the diamond paste on velvet cloth. After that, these samples were chemically etched with the combined acid solution of 10 % HF and 10% HNO<sub>3</sub> for 15 seconds. After gold plating, these samples became ready for SEM characterization.

### **3.2.3 Density**

Densities of the prepared samples were measured by well-known Archimedes principle using distilled water as a buoyancy liquid. Density of a sample is measured using a weighing balance having accuracy of 0.0001g equipped with density measurement kit.

### 3.2.4 Dielectric measurements

#### (a) Microwave dielectric characterisation:

The rectangular pellets were made to the exact dimension of X– Band (22.86mm × 10.16mm × 5mm) waveguide for the measurement. High frequency dielectric measurements were carried out using Keysight Technologies make Network Analyser (Model: ENA E5071C) through waveguide method in the X– band. Before performing the measurements, calibration was performed. For the waveguide measurement, TRL (Thru-Reflect-Line) calibration was performed. The two coaxial cables (7.0 mm) were connected with the Port1 and Port 2 respectively. At the other end, both the cables were connected to the ADAPTER (Coaxial to Waveguide Transition). There are two standards for reflect and line match. Before starting the calibration, the frequency band (X– Band) for which the measurement is going to be performed is defined. The first step in TRL calibration is Thru, which means connecting both the adapters together. The second step is Reflect in which the em signal coming from Port 1 is reflected back by using the reflect standard (Figure 3.4). Similarly, signal is reflected from port 2. Last step is the Line match, in which the standard for line match (also called short) is connected in between the two adapters. After the calibration was completed, the sample was kept inside the waveguide (sample holder) and measurement was performed. The measurement is based on transmission/reflection (NRW technique) in which the reflection ( $S_{11}$  and  $S_{22}$ ) and transmission ( $S_{12}$  and  $S_{21}$ ) parameters were measured. The values of  $S_{11}$ ,  $S_{12}$ ,  $S_{21}$ ,  $S_{22}$  were used to obtain the values of dielectric constant and tan delta of the sample. The signal coming from port 1 and reflected back to port 1 provides the value of  $S_{11}$ , the signal coming from port 1 and transmitted to port 2 gives  $S_{21}$ . Similarly, signal coming from port 2 and reflected back to port 2 provides  $S_{22}$ , and the signal coming from port 2 and transmitted to port 1 gives  $S_{12}$  (Figure 3.5).

Keysight Network Analyser(Model:E5071C)

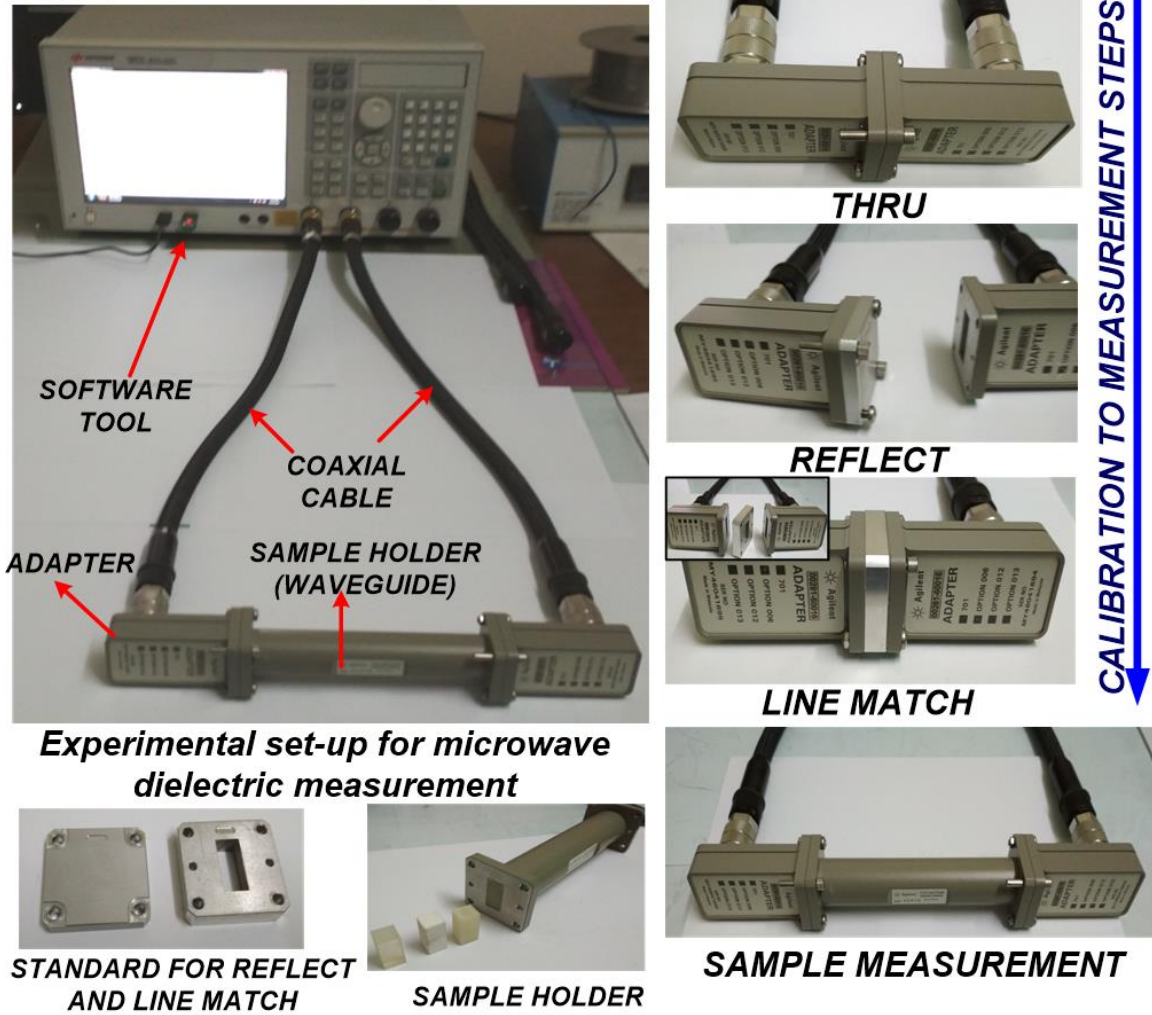


Figure 3.4 Microwave dielectric measurement of the sample using Network Analyser.

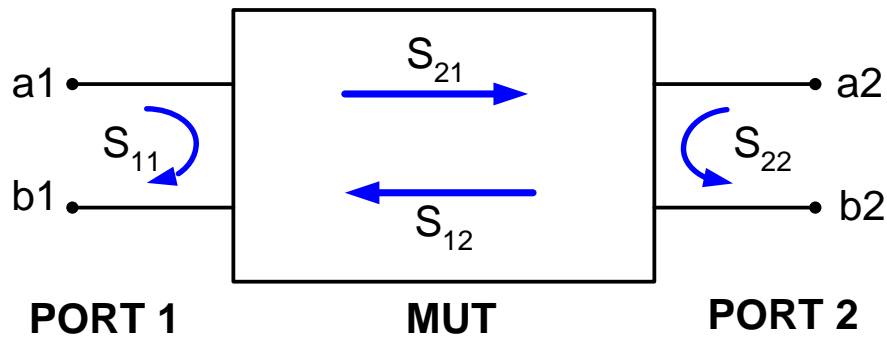


Figure 3.5 Showing reflection and transmission coefficients in 2 port measurements.



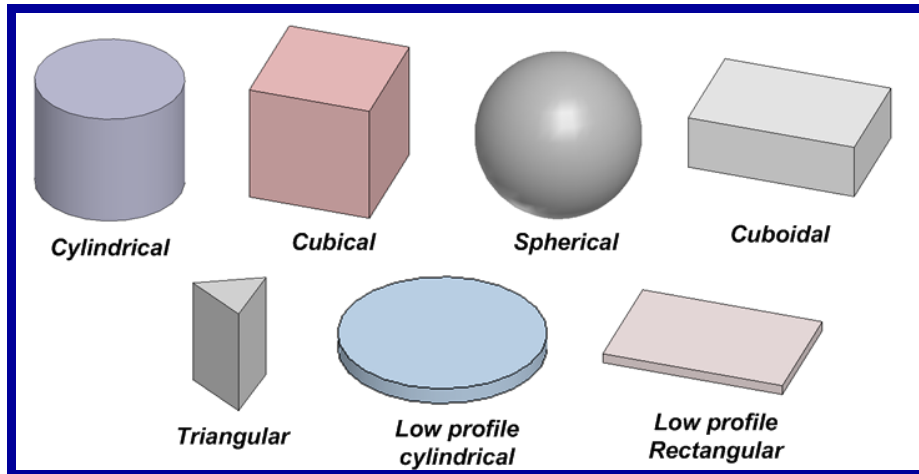
### **(b) Low frequency dielectric characterisation:**

The dielectric measurements up to 1 MHz were performed by Alpha-A High Performance Frequency Analyser (Novo Control Technologies). This is based on parallel plate capacitor method. The measurement of dielectric constant can be done in wide temperature range i.e. in the range 30 – 400 °C. The temperature coefficient of dielectric constant can be predicted by observing the variation in dielectric constant with temperature. The cylindrical pellets of ~10 mm diameter and thickness of < 2mm were prepared. The sintered sample was coated with the silver paste on both surfaces. After silver paste was applied, it was dried in oven for an hour and kept in the furnace for maturing at ~500 °C. Now, this sample which behaves like a parallel plate capacitor was kept in the temperature controlled furnace attached with the Frequency Analyser for the dielectric measurement in the specified frequency range.

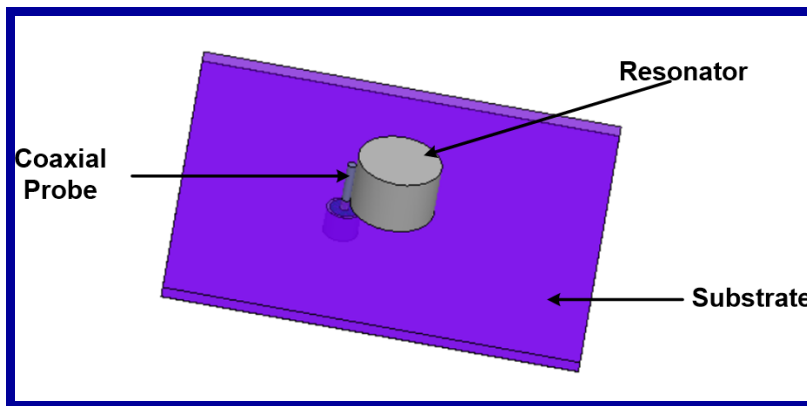
## **3.3 DRA design and simulation**

### **3.3.1. Basics of DRA design**

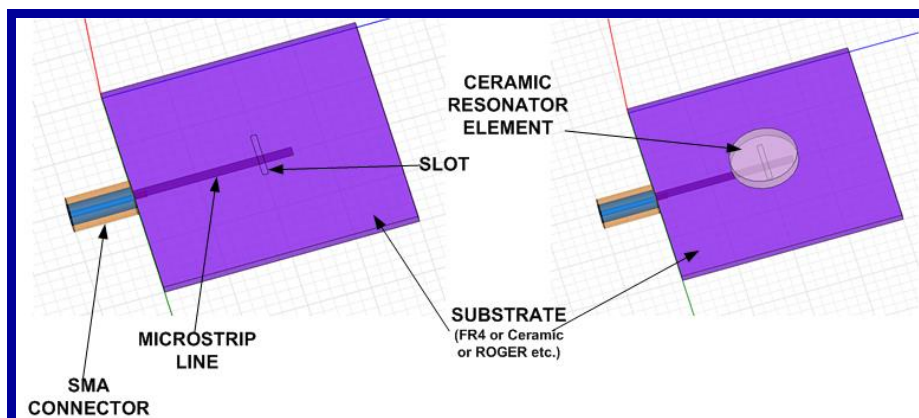
Antenna design components includes substrate (FR4 / Copper / Ceramic), ground plane, resonator element (Ceramic) and feeding structure. Antenna resonator has different standard geometries (Figure 3.6) such as cylindrical, rectangular, triangular and spherical made of different ceramic materials of wide range of dielectric constants (5 – 50). Two different excitation schemes are used in the present investigations i.e. Probe feed, Micro-strip line fed aperture coupling (Figures 3.7 and 3.8).



**Figure 3.6** Standard geometries of DRAs



**Figure 3.7** Coaxial probe feed

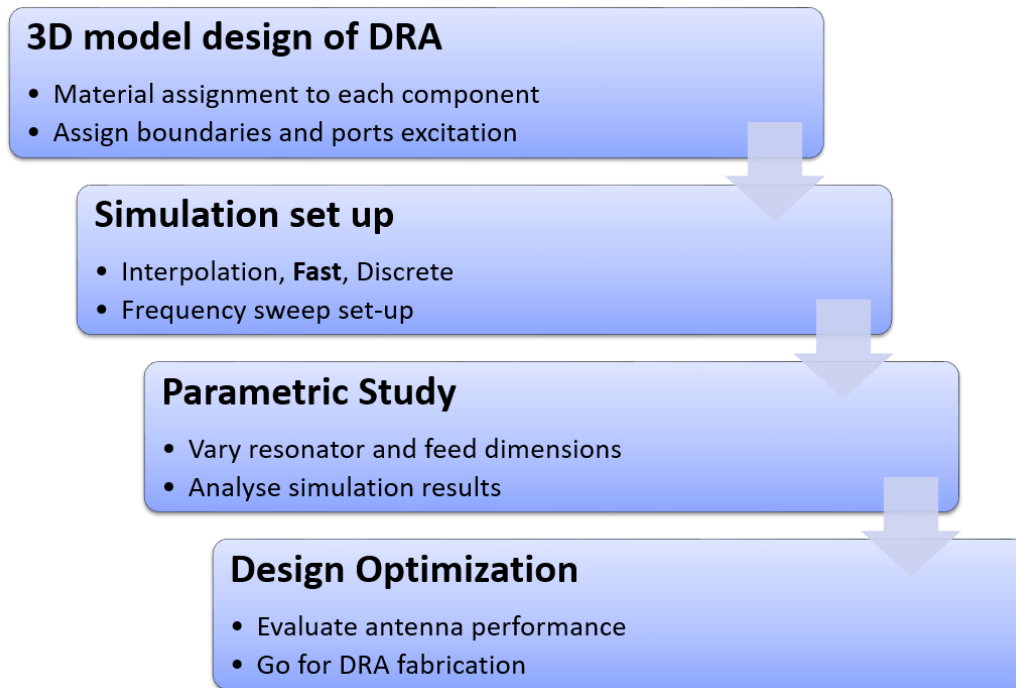


**Figure 3.8** Aperture coupling

### 3.3.2. DRA design steps in HFSS

The procedure for the design of DRA in High Frequency Structure simulator (HFSS) software is shown in Figure 3.9. The design steps followed in HFSS are as follows:

- i. Design of DRA using variables  $x$ ,  $y$ ,  $z$  for all of its structural dimensions (Assign values to the selected variables by considering antenna design equations).
- ii. Proper material assignment to each antenna component.
- iii. Proper excitation of the port.
- iv. Draw air box and assign radiation boundary.
- v. Define frequency sweep setup.
- vi. Use of three iterative techniques: Interpolation, fast and discrete.
- vii. Opt for fast iteration for the parametric study. Vary different antenna parameters: probe height, material assigned (dielectric constant and loss tangent), aperture dimensions, and antenna resonator dimensions, and observe the antenna characteristics.
- viii. After optimization, go for discrete iteration for final simulation results.
- ix. Discrete iteration takes more time, but gives accurate results. So, it can be verified for short range of operating frequencies only.
- x. Extract different antenna characteristics, viz. reflection coefficient – frequency characteristic, far field radiation pattern, 3D gain and near field distribution.
- xi. Analyse the antenna performance. If antenna performance is good, go for antenna fabrication for the optimized design.

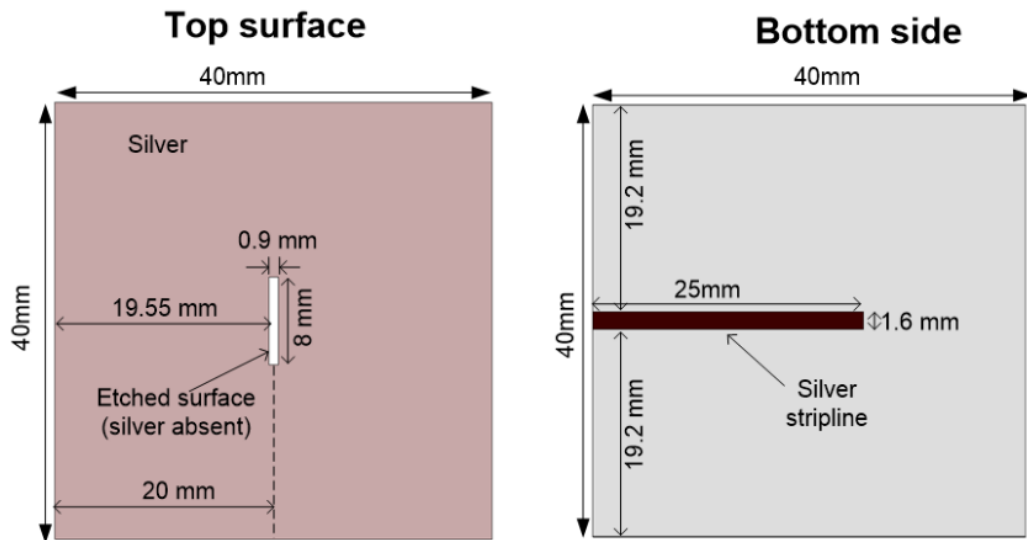


**Figure 3.9** Design procedure in HFSS

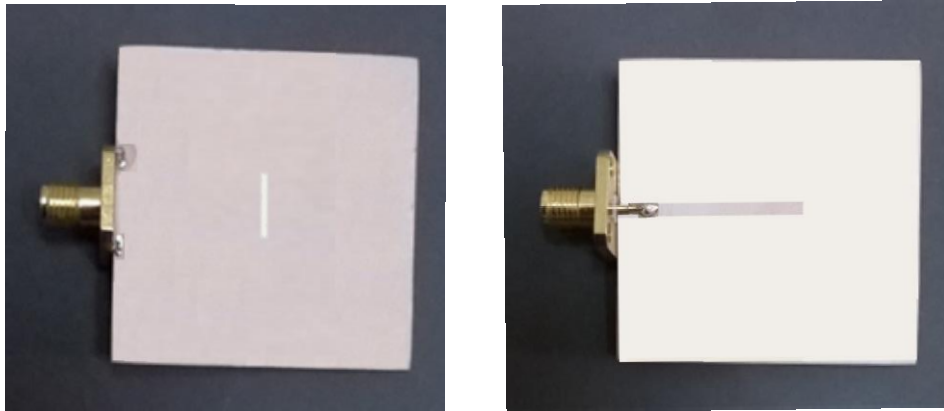
## 3.4 Antenna fabrication

### 3.4.1 Requirement

- i. Substrate (Ceramic): For the design of DS-CDRA, LMAP ceramic was used for the substrate. Silver coating on ceramic substrate was done for the ground plane (except the slot) and the microstrip line. After applying silver paste, it was kept in oven for 1 h at 100 °C for proper drying. Finally, silver coating is matured at 500 °C for 10 mins after keeping it in electric furnace with a heating and cooling rate of 1 °C/min (Figure 3.10).



(a)



(b)

**Figure 3.10** View of the substrate of DS-CDRA with slot on the top side and the microstrip line on the bottom side. (a) Dimension of slot and microstrip lines (b) Image of fabricated prototype.

- ii. Substrate (Copper plate): For the design of TDRA, copper plate was used as the substrate. Drilling was done at proper position on the copper plate for probe feed. Hole diameter ( $D$ ) should be slightly greater than inner probe diameter of the SMA connector. [Precaution:  $D <$  Diameter of middle dielectric of the SMA connector]

- iii. Resonator elements (Ceramic / Teflon): Prepare the antenna resonator elements of exact or very close to the optimized dimensions.
- iv. RADIALL make 3.5 mm SMA connector, Araldite adhesive, soldering kit and digital multimeter.

### **3.4.2 Fabrication steps**

- i. Test the plated ceramic substrate using multimeter (Precaution: Ground and microstrip line must not get short-circuited).
- ii. Assemble all the antenna components and fix them at proper positions on the substrate with the help of adhesive. [Precaution: Mark the positions carefully before fixation of resonators]
- iii. Fix the SMA connector using soldering kit, and again check the connections of the SMA connector with the multimeter.
- iv. Go for measurement of antenna characteristics.

## **3.5 Antenna measurement**

### **3.5.1 Antenna measurement site**

Antenna measurement site is called as antenna measurement range which is of two types i.e. outdoor range and indoor range. On the basis of principle of measurement, there are three different types of ranges: (a) Reflection range (b) Free space range and (c) Compact Range.

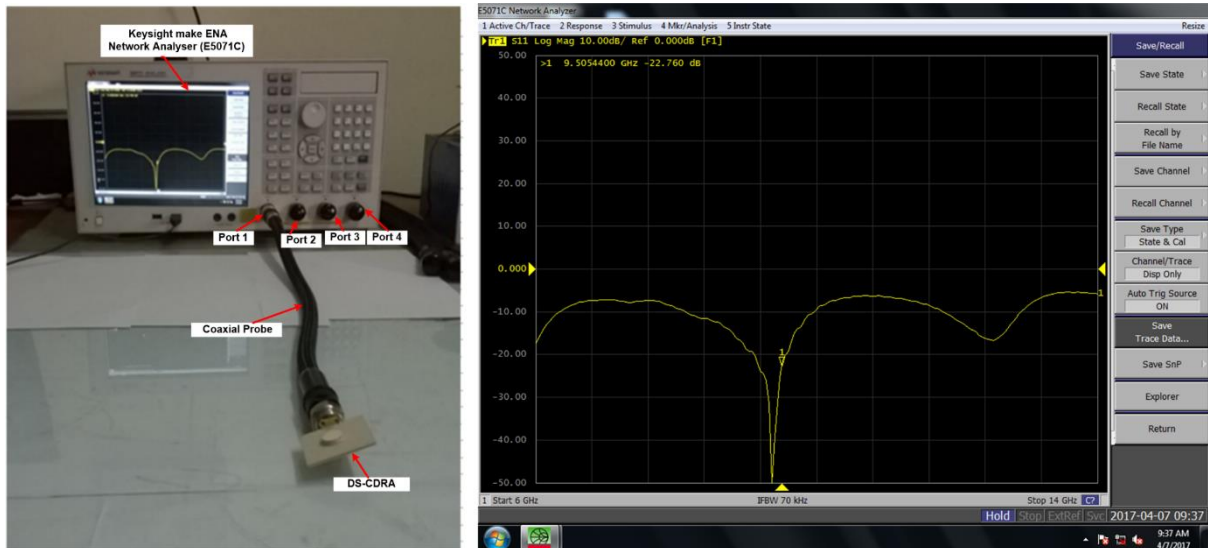
**(a) Reflection range:** It is designed in such a way that the direct and reflected (usually from ground) waves interfere constructively and form uniform wavefront (both in magnitude and phase) in the region of antenna under test (AUT). The ground reflection which occurs is due to soil or rock etc. (of some permittivity or conductivity). At the site

of AUT, constructive interference occurs and maximum gain is achieved. This region is called **Quiet Zone**.

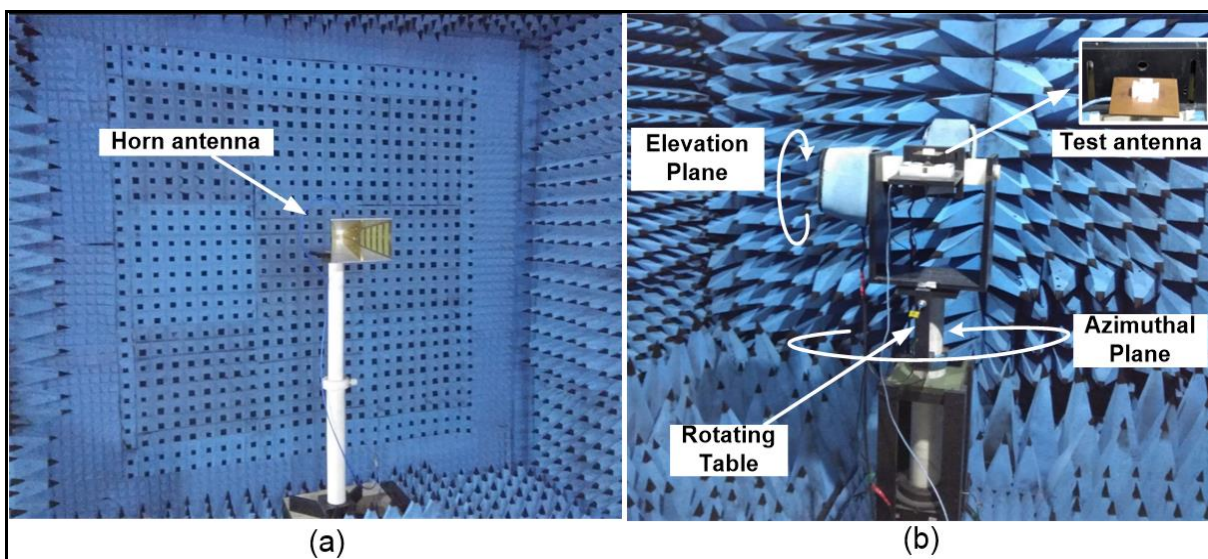
**(b) Free space range:** It includes outdoor and indoor ranges. The outdoor ranges are carefully built to minimize reflections from buildings and other objects. It is realized as elevated ranges and slant ranges. Source should have narrow beam and very low side lobes. Line of sight should be clear. Indoor range corresponds to anechoic chamber, in which reflections from the walls, floors and ceilings are suppressed using the microwave absorber linings, which are of pyramidal shape and wedge or V shape.

For pyramidal absorber, bottom side of the pyramidal foam is conducting sheet with intrinsic impedance,  $\eta = 0$  and at the tip of the absorber the intrinsic impedance equals to free space impedance of  $377 \Omega$ . The graphite 'C' is sprayed to improve the impedance matching. The pyramidal absorber corresponds to four quarter wave transformers with its height equal to the wavelength at the lowest frequency of operation. In this case, the maximum absorption takes place on normal incidence which provides nearly 50 dB attenuation while on oblique incidence, the attenuation goes down by about 20-25 dB. In case of wedge shaped absorber, maximum absorption occurs when incident signal is along the ridge impedance changes from ( $377$  to  $0 \Omega$ ). The wedge shape is better than the pyramidal shape of absorber.

The tapered configuration/structure of anechoic chamber is more preferred than the cuboidal structure. This is so because the two signals interferes constructively after reflection from the tapered walls of the anechoic chamber to give the maximum amplitude and phase (uniform wavefront) at the antenna test site (Quiet zone).



**Figure 3.11** Experimental set-up for input characteristic measurement of dual segment cylindrical DRA.



**Figure 3.12** Experimental set-up for radiation characteristic measurement of the 4–element composite triangular DRA.

The limitation of anechoic chamber is that it does not meet the required distance for the far field measurements for large antennas or scatterers. The two approaches to overcome this limitation of anechoic chamber are (a) **Compact antenna test ranges (CATRs)** and (b) **Near-to-far field transformation**. In CATRs, nearly uniform wave is produced in a



short distance using a system of reflectors (serrated edge and rolled edge reflectors or a large paraboloidal reflector). In the later case, near field measurement is performed and then near field distribution is converted to far field pattern by using Fourier transformation.

In the present investigation, the antenna measurement was performed in the free space range (Indoor) i.e. inside the anechoic chamber with cuboidal structure and the pyramidal absorber lining.

### **3.5.2 Antenna measurement steps**

Primary measurement to ensure whether AUT is getting excited or not, is done by one port measurement using Keysight make Network Analyser (Model: E5071C) (Figure 3.11). If the reflection coefficient - frequency characteristic is obtained below -10 dB in the desirable frequency range (or as per simulation results), then go for second step measurement for radiation characteristics in anechoic chamber. Else, some fabrication error is expected.

An anechoic chamber (an-echoic meaning "non-reflective, non-echoing") is a room designed to completely absorb reflections of electromagnetic waves. It is made of pyramidal foam material (fireproof polyurethane foam loaded with conductive carbon black). Its dimensions are set specific to the wavelength of operation (as per the lowest frequency). The double ridged broadband horn antenna (800 MHz – 18 GHz) with N-female connector is used as reference horn antenna for the radiation characteristic measurement. Reference horn antenna is a transmitting antenna, which is fed by 0 dBm power (1mW) and AUT is the receiving antenna. The test equipment connected with the measurement system is the Network Analyser (Agilent Technologies, Model: N5224A). The AUT is kept on a rotating table, at a distance of around 5.5 m for signal reception

coming from the horn antenna. The AUT can be rotated in both the azimuthal (horizontal plane) and the elevation (vertical) planes. Thus, the radiation patterns are recorded in both the planes using the automated measurement system as shown in Figure 3.12.