A MINOR RESEARCH PROJECT

On

Synthesis, Characterization and Microwave Applications High-*Q* Ferroelectric $Ba_xSr_{1-x}TiO_3$ Thick films

(No. F.47-767/13/(WRO))

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DECLARATION

I hereby declare and certify that, the Minor Research Project entitled **"Synthesis, Characterization and Microwave Applications High-Q Ferroelectric $Ba_xSr_{1-x}TiO_3$ Thick films"** No. F.47-767/13/(WRO) is a record of research work carried out by me during the year 2014- 2016. Further declare that the work presented in the report is original and carried out according to the plan in the proposal and guidelines of the University Grants Commission.

Sd.-

(Dr. S. A. Kanade)

Principal Investigator

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1. Introduction

It has been observed from the literature survey that Barium titanate ($BaTiO_3$) is the first practically used lead free piezoelectric ceramics and is still the basic candidates for various applications such as multilayer ceramic capacitors, actuators and sensors. However, the relatively large dielectric insertion loss, soft mode effect and limited Q value at high frequency microwave regions limits its overall usefulness.[1]

Further, barium zirconium titanate and barium strontium titanate are well studied and promising candidates among the lead free piezoelectric materials [2].

1.1.Synthesis: Various chemical routes like co-precipitation, sol-gel, sol precipitation,

hydrothermal, auto combustion etc. are known to yield nano particle products The sol-precipitation method gives fine particle size, simple compositional control and low processing temperature. It is reported by Wada et al that small grain and large volume of grain boundary make the ceramics ‘‘soft’’ and sensitive for excellent piezoelectric properties. Thus it is important to select proper method for the synthesis of nanoparticles. By considering different methods we consider sol-gel auto combustion method for synthesis of nano particle

Polymeric precursor method is an extensively used polymeric route, where a solution of ethylene glycol, citric acid and metal ions is polymerized to form a polyester type region. The metal ions can be immobilized in a rigid polyester network, and no segregation of cations was observed during thermal decomposition of metal ions [3].

1.2. Introduction to microwave integrated circuits:

Microwave circuits can be integrated by hybrid or monolithic techniques on a Varity of substrates [4]. The beginning of the modern area in microwave and milimetric wave integrated circuitry can be traced back to the early 1950’s, when Stripline [5] and microstripline [6,7] were introduced.

The microwave integrated circuit (MIC) is a miniaturized form of microwave circuit. The concept exploits the small size of the solid state device and its compatibility with the planer form of transmission line, combining the advantage of conventional integrated circuits. MIC differs from the lower frequency hybrid IC (integrated circuit) in that the

circuit interconnections are usually established by TEM mode transmission lines [8]. Every structure comprises a combination of metal and dielectric. In most cases the dielectric principally supports the metal pattern and influences the wave propagation.

1.2.1 Transmission lines for planer MIC

A transmission line is made up of a symmetrical pair of strip conductors or a single strip and ground plane, at opposite face to of sheet of dielectric material. Microstrip transmission lines are widely used in MICs

Microstripline do not require stringent tolerance and can be made at low cost. The microstripline consists of a metallic strip of width 'W' and thickness 't' and a ground plane separated by a solid dielectric of thickness 'h' as shown in Figure 1.1.

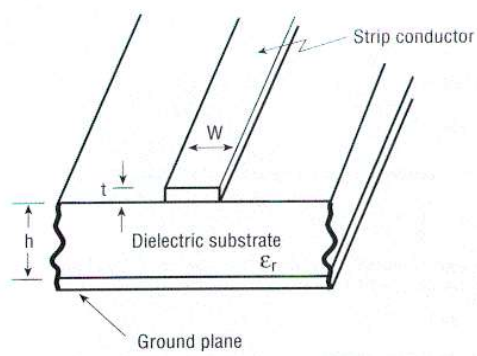

Figure 1.1: Cross sectional view of the Microstripline

Although the microstrip has very simple geometric structure, the electromagnetic field involved are actually complex. The electromagnetic boundary conditions infer the presence of longitudinal component at dielectric air interface that indicate that microstrip cannot support pure TEM (transverse electromagnetic) mode. The microstrip is characterized by its complex mode of propagation viz. superposition of LSE and LSM mode [9]. This is also called quasi TEM mode. The microstrip is a slow wave dispersive structure. Hence its propagation constant varies non-linearly with frequency [10]. The characteristics impedance is also function of frequency [11, 12]. However simple approaches to the quasi TEM mode, calculations combined with frequency dependent expressions yield satisfactory design accuracy for many applications. But at higher frequencies particularly millimeter wavelength ranges losses increase greatly, because higher order modes become a

considerable problem and fabrication tolerance becomes exceedingly difficult to achieve. The most important dimensional parameters are the microstrip width 'W', height of the substrate 'h' and relative permittivity of the substrate 'ε_r.'

1.2.2. Microstrip Transmission Type Resonators:

Resonators are the circuits that will pass a specified band or bands of frequencies. The various types of symmetrical and asymmetrical resonator structures can be fabricated in planer form as a part of MIC. In this work symmetrically coupled microstrip straight resonator and ring resonators has been used, the details of these are elaborated here.

1.2.3. Microstrip Ring Resonator: [13]

The microstrip ring resonator is a simple transmission line formed in a closed loop. When an integral multiple of wavelength equals the mean circumference of the ring structure, resonance (maximum transmission of the signal) occurs i.e.

$$2\pi R = n\lambda_g \text{, or } 2\pi R = \frac{nc}{f_r} \text{ for n=1, 2, 3 …} \qquad (1.1)$$

Figure 1.2.a shows the cross-sectional view and figure 1.2.b shows the planer view (geometry) of the microstrip ring resonator

1.2.4. Microstrip straight resonator:

The figure 1.4.a shows the cross-sectional view and figure 1.4.b shows the planer view (geometry) of the Stripline end coupled transmission type straight (line) resonator. The resonance occurs when length of the resonator is integral multiple of the half wavelength. The resonant frequencies of transmission type resonators are obtained as follows [14]

$$2(l + l_g) = \frac{n\lambda_g}{2} = \frac{nc}{F_r\sqrt{\varepsilon_{r,eff}}}, \quad \text{n = 1, 2, 3…} \qquad (1.2)$$

Where $\varepsilon_{r,eff}$: effective dielectric constant of the substrate.

2l: length of the resonator.

Figure 1.2.c shows shows the planer view (geometry) of the microstrip straight resonator.

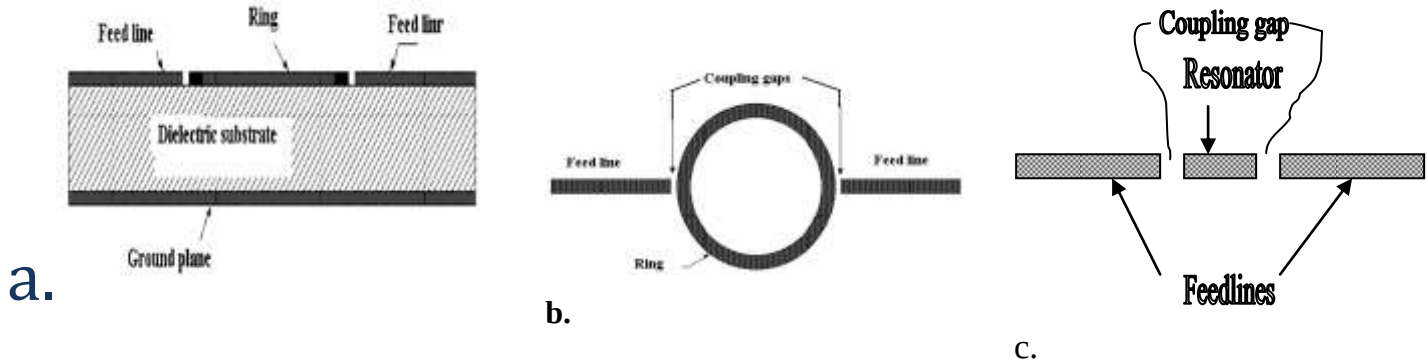

Figure 1.2: Microstrip resonator: **a.** Cross sectional view **b**. Planer view of ring resonator. C. Planer view of straight resonator

1.2.5. Fabrication of microstripline components:

There are two types of hybrid technologies involved for the fabrication of hybrid microwave circuits. The thin film technology and thick film technology. H. Sobol [15] has reported that the term thin film or thick film refers to the process used and not to the thickness of the film.

In the present work only screen printing (thick film technology) was used for microstrip circuit fabrication.

1.2.6. Microstripline Components With Overlay:

In this work since as overlay technique is used for microwave characterization, a brief review of the work done by various workers using various microstrip components is given.

When the area on the top of the microstripline is covered by some material touching the component it is called microstrip with overlay. The overlay is also called as superstrate. When the overlay is used over the open microstripline, it is called multiplayer microstrip [16]. Multilayer microstrips have been a topic of theoretical interest for many years [17-19]. The literature is available on theoretical analysis of multiplayer microstrips, by methods like, potenial theory [20], and spectral domain technique [21, 22]. Vary few references are available on the actual experimental data regarding various types of overlays on microstripline circuits. Mesa et al [23] have studied the influence of the top cover on the leakage from microstripline. Dielectric overlays are used to improve the properties of stripline components. Bulk overlays have been used to improve power handling capacity up

to kilowatts [24]. Effect of thin film overlays was investigated by Pande et al [25]. Effect of various bulk and thin film overlays on rejection filter [26], 3dB hybrid directional coupler [27], microstriplines and band pass filters [28]. Most of the literature available are from the authors laboratory and from Pune university, Pune.

1.2.7. Dielectric Properties Measurement Methods:

In literature there are many reports dealing with measuring methods of the dielectric properties of electronic materials to be employed in microwave and milimetric wave circuitry The measuring methods can be divided into two main categories [29]. The methods in the first category employ resonator in the determination of the dielectric and attenuation properties. The methods in the second category employ non-resonant structures and determination of the dielectric and attenuation properties of the materials are based in the measured propagation constant of the transmission line [30].

1.2.8. Resonance Techniques:

Resonance techniques that are widely used for the measurement of microwave dielectric properties, which can be divided into two groups:

1) The reasons are basically supported by the dielectric itself: The sample acts as dielectric resonator metal shields with different geometries are always introduced to prevent radiation loss. This type is called dielectric resonance techniques.[31]
2) The resonance is supported by metal walls of a metal cavity. The presence of the sample is the cavity causes only a perturbation as the field distribution in the metal cavity. This is called cavity perturbation technique [32].

1.2.9. Non-resonance techniques:

The various non-resonant techniques are used to study dielectric properties of materials at microwave frequencies like transmission line method, slotted waveguide method, & Time domain reflectrometry (TDR)[31]

The dielectric properties of materials can be easily and inexpensively obtained by transmission line technique but, this technique is cumbersome. The sample must be made into a slab or annular ring which exactly fits the transmission line or wave guide [32].

In shorted waveguide method the value of ε' and ε'' are derived from transmission line theory, which indicates that these properties could be determined by measuring the phase and amplitude of reflected microwave signal from a sample of material placed against

the end of short circuited transmission line.[32]. In free space transmission method, by measuring the change phase and attenuation of the transmitted wave ε' and ε'' can be calculated [33].

However all the methods expect free space transmission method, require sample preparation. The geometry and sized the sample must be exactly known. Hence all the methods are destructive.

The free space transmission method require sample size should be at least four time more then the size of radiating antenna and hence not suitable to sample of small size.

1.2.10. Planer circuit methods:

To find ε' and ε'' of the planer materials (substrate), planer circuit methods can be used. The microstrip ring resonator is used by Liu et al [34], to study $Ba_xSr_{1.x}TiO_3$ ceramics. The resonator was delineated on the ceramic which was the substrate. Other planer circuit methods have been used to study ceramics [35]. In all these ceramics has been used as substrate. The planer circuit methods are destructive in the sense that metallization has to be permanently coated on the film or sheet materials.

1.2.11. Use of Microstrip Components for Dielectric Characterization of Materials Using Overlay Technique:

There are very few reports on the dielectric characterization using overlay technique. Microstrips are being used as microwave components, and are suitable for dielectric permittivity measurement. The basic idea is perturbation of fringing field of microstrip component using a dielectric overlay[36, 37] A dielectric material overlaid on the microstrip component perturbs its fringing field due to change in effective dielectric constant (a combination of substrate dielectric constant and the material above the microstrip component) of the microstrip component, This perturbation causes change is its electrical parameters such as transmission, reflection, resonance frequency and ‘Q’ value of the component [38]

Microstrip ring resonator was used to find the variation of dielectric constant mug grains with frequency [39]. To estimate the moisture contain of wheat [40] and as bio-material moisture sensors [41-43]. These microstrip sensor can be placed in contact with one side of the test material and therefore provide non-destructive measurements of the test dielectric.

2. Experimental:

2.1. Synthesis of the Ferroelectric $Ba_xSr_{1-x}TiO_3$ powder:

Ferroelectric $Ba_xSr_{1-x}TiO_3$ powder were prepared by the Complex Polymerization Method (CPM) using barium and strontium acetates and titanium isopropoxide as starting materials

A titanium citrate solution was prepared by dissolving titanium isopropoxide in a solution of ethylene glycol. Afterwards the solution of citric was added. This solution was heated at 60°C with constant stirring for 10 min. The solution of barium and strontium was prepared by dissolving barium and strontium acetates in citric solution. The solution of barium and strontium acetates was added drop wise to the titanium citrate solution with constant stirring. The temperature was raised to up to 120-140 ^{o}C, when the solution becomes solidified into a dark brown glassy region. The decomposition of organic part was performed in muffle furnace at 200°C for one hour. The obtained powder was calcined at 500 ^{o}C for one hour.

2.2. Characterization Techniques Used.

To explore the formation temperature, crystal structure and microstructure various characterizations were carried out. The various tools used for characterizations are X-ray diffraction, FTIR.

2.2.1. Fabrication Of Ferroelectric $Ba_xSr_{1-x}TiO_3$ Thick Film.

Screen-printing is a cost effective technology to produce planer components. This section deals with the fabrication of the thick films. These thick films are fabricated completely at Department of Physics, J. S. M. College, Alibag. The fabrication procedure is explained in brief.

2.2.2. Thick film Paste formulation

The Ferroelectric thick film paste is formulated by mixing organic vehicle, functional material (ceramic powder) and oxide binder, in mortar and pestle. The paste is prepared according to the flow chart shown in Figure 2.1

The functional materials used are the ceramics listed in Table 2.1, synthesized by oxalate co-precipitation and sintering, as described in section 2.1. The inorganic oxide binder used is Bi_2O_3 and PbO with 75 wt % Bi_2O_3 and 25 wt% PbO.

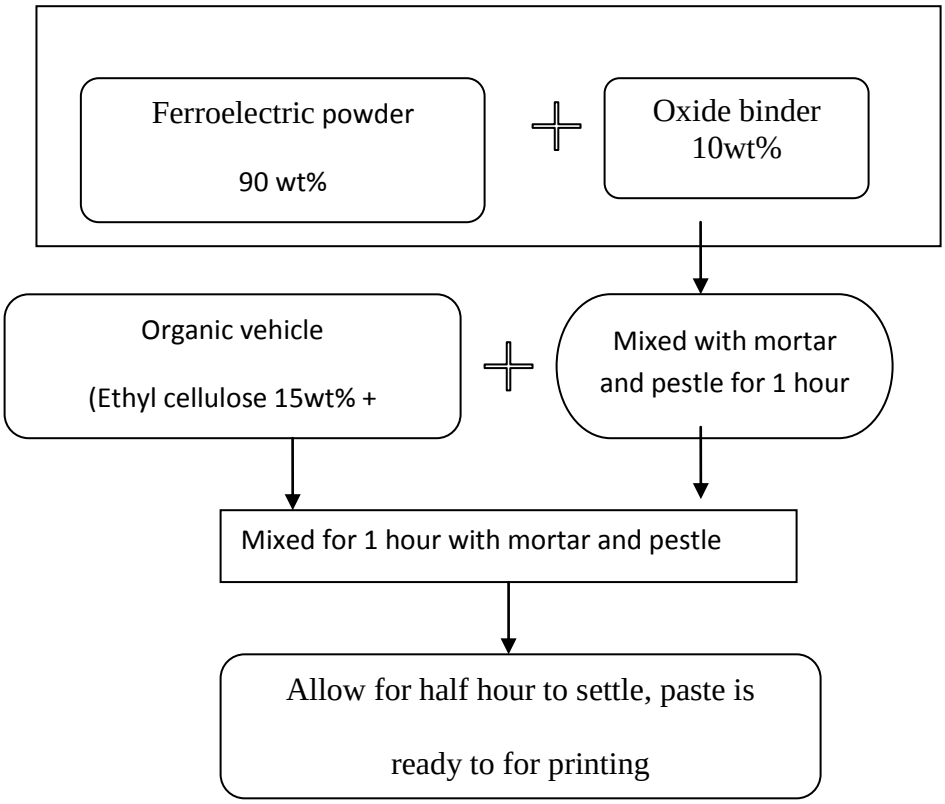

Figure 2.1. Flow chart for thick film Ferroelectric paste formulation

2.3. Design of microstrip components:

The microstrip ring resonator and straight resonator were delineated by screen-printing the silver paste on 96% alumina substrate. The thick film circuit was fired at 700°C. by conventional thick film firing cycle. The dimensions were calculated by using formula (1.1) and (1.2) respectively.

2.3.1. The characterization of the microstrip components

The characterization of the microstrip components designed was done by measuring transmission and reflection of the microwaves in the frequency range 8 to 18 GHz (X and Ku-bands). The measurements were carried out using Vector Network Analyzer model Agilent Technologies N5230A. The microstrip component was mounted in a resilient MIC (microwave integrated circuit) test fixture. The connection to the coaxial probe of the vector network is achieved using SMA (semi-miniaturized adaptors).

For overlay studies the pellet of the composition of $Ba_{1-x}Sr_xTiO_3$:$0 \leq x \leq 1$ was kept as in touch overlay on Ag thick film microstrip resonator and S_{21}measurements were made. The effective dielectric constant of the microstrip component is calculated by using Owen's formula [19]

$$\varepsilon_{eff1} = \frac{\varepsilon_r + 1}{2}\left[\left(1 + \frac{29.98}{Z_0}\left(\frac{2}{\varepsilon_r + 1}\right)^{0.5} * \left(\frac{\varepsilon_r - 1}{\varepsilon_r + 1}\right)\left(Ln\left(\frac{\pi}{2}\right) + \frac{1}{\varepsilon_r} Ln\left(\frac{4}{\pi}\right)\right)\right)^2\right] \quad (2.1)$$

Where ε_r = 9.6, the dielectric constant of the substrate.

Zo = 50 ohm

The effective dielectric constant of the multilayer structure (microstripline overlaid with the dielectric) was calculated from the resonance frequency with and without overlay. By using equation(2.2)

$$\frac{\varepsilon_{eff1}}{\varepsilon_{eff2}} = \frac{F_2^2}{F_1^2} \quad (2.2)$$

Where ε_{eff1} and F_1 are the effective dielectric constant and resonance frequencies without overlay and ε_{eff2} and F_2 are the corresponding parameters with the overlay. [20]

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3. Results and discussions

The results of the structural and electrical characterizations carried out on the pellet or powders are presented in this chapter along with the discussions.

3.1: Structural analysis:

3.1.1. X-ray diffraction

The X-ray diffractograms of the polycrystalline $Ba_xSr_{1-x}TiO_3$:$0 \leq x \leq 1$powder samples were obtained by powder diffraction technique. The diffractograms for the $Ba_xSr_{1-x}TiO_3$: $0 \leq x \leq 1$ are shown in figures 3.1. The diffractograms are well defined with (44) as strong reflection peak, with a perovskite structure (JCPDS Card No. 34-0411). The broadening of peaks indicates the formation of nanosized particles.

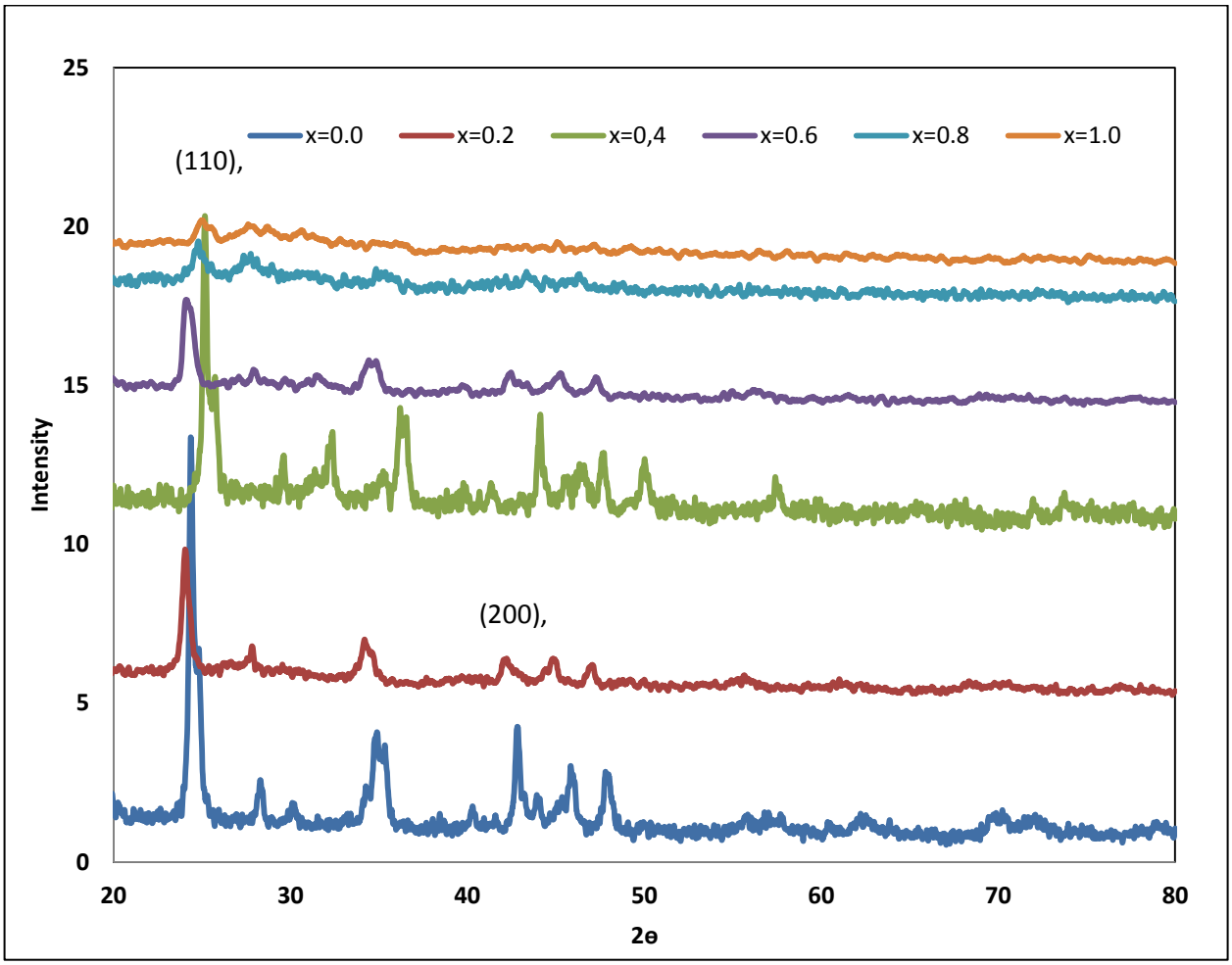

Figure 3.1. XRD pattern of the $Ba_xSr_{1-x}TiO_3$:$0 \leq x \leq 1$.powder for x=0.2 and x=0.4

3.1.2: FTIR Analysis:

The FTIR (Fourier transform infrared) spectra of the $Ba_xSr_{1-x}TiO_3$ powder samples obtained by Complex Polymerization Method (CPM) and sintering at 500°C, having composition $Ba_xSr_{1-x}TiO_3$:0≤x≤1, are recorded for the conformation of the crystal structure formed. Figures 3.2. shows the recorded FTIR spectra.. The bands in the region of (1500 cm−1) were attributed to the formation of Ba-O-Ti bond [45]..

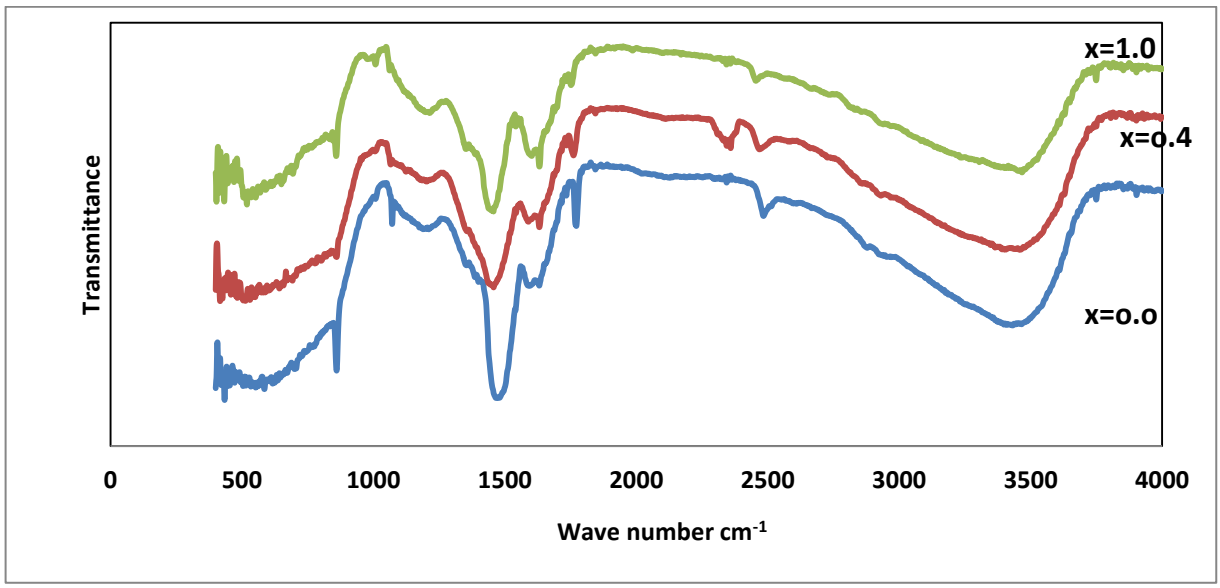

Figure 3.2. FTIR spectra of $Ba_xSr_{1-x}TiO_3$:0≤x≤1

3.2. Dielectric Measurements:

The dielectric constant was measured by parallel plate capacitor method. The obtained dielectric constant $Ba_xSr_{1-x}TiO_3$:0≤x≤1, at various frequencies are plotted in the following figure 3. 3

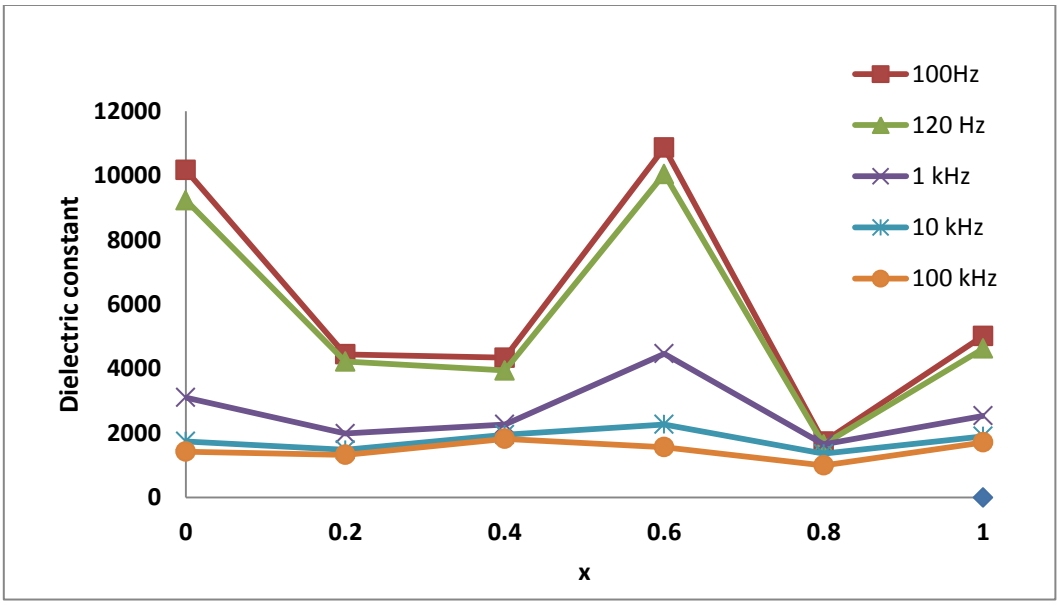

Figure 3.3.:variation of dielectric constant with composition of the $Ba_xSr_{1-x}TiO_3$:$0 \leq x \leq 1$, at various frequencies

3.3. microwave characterisation of $Ba_xSr_{1-x}TiO_3$:$0 \leq x \leq 1$

3.3.1. Overlay Studies on Microstrip Ring resonator

The variation in the transmittance (S_{21}) and reflectance with frequency of the designed microstrip ring resonator is shown in figure 3.4. The resonance (Maximum S_{21}) is at 15.14 GHz. The response of the resonator due to the $Ba_xSr_{1-x}TiO_3$ overlay is shown in figure 3.5. From the figures it is seen that, due to the overlay the resonance frequency shifts towards lower frequency side with substantial increase in the peak transmittance. The resonator shows broadband characteristics due to thick film overlay

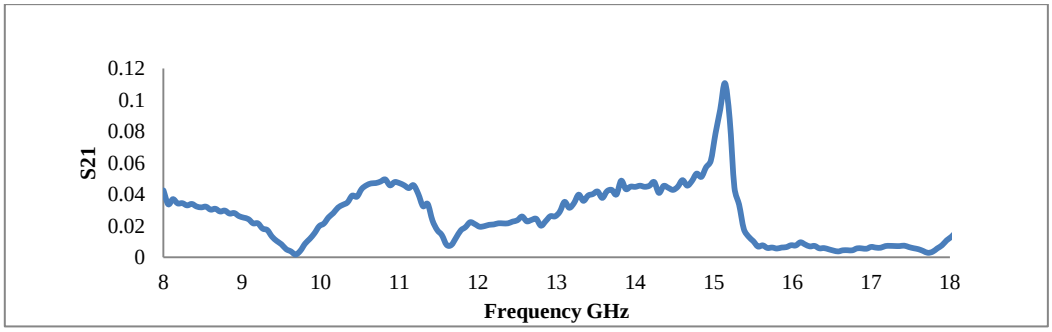

Figure 3.4: Variation of transmission coefficient (S_{21}) of microstrip Ring resonator as a function of frequency, without overlay

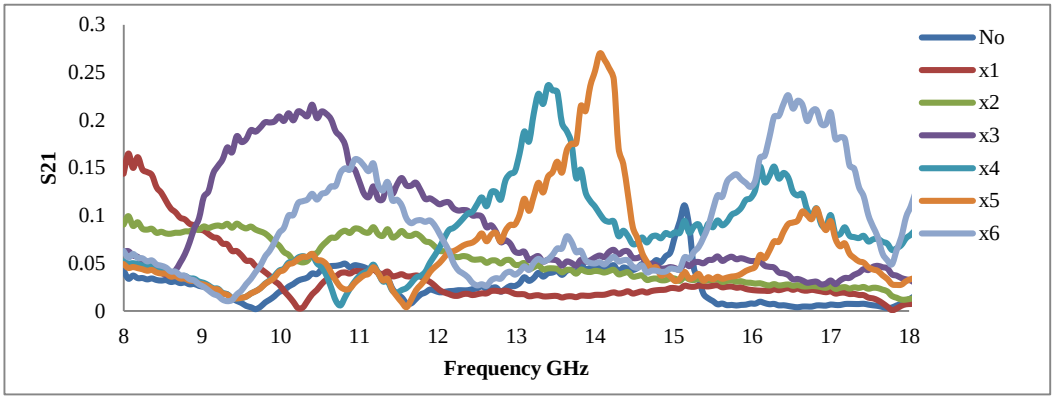

Figure 3.5: Variation of transmission coefficient (S_{21}) of microstrip Ring resonator as a function of frequency, with overlay of $Ba_xSr_{1-x}TiO_3$

The effective dielectric constant of the multilayer structure was calculated using formula (1). The plot of composition dependent effective dielectric constant of the multilayer structure for the $Ba_xSr_{1-x}TiO_3$ is shown in figure 3.6. From the figure it is seen that dielectric properties are composition dependent

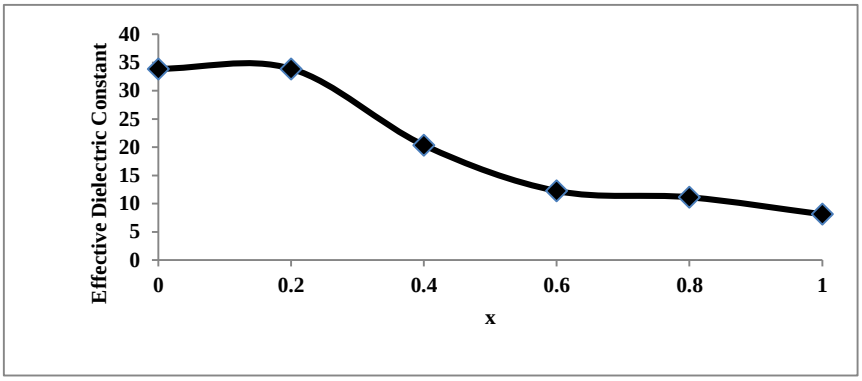

Figure.3.6: Variation of effective dielectric constant with composition of $Ba_xSr_{1-x}TiO_3$, of overlay.

3.3.2. Overlay Studies on Microstrip straight resonator:

The variation in the transmittance (S_{21}) with frequency of the designed microstrip straight resonator is shown in figure 3.7. The resonance (Maximum S_{21}) is at 16.58 GHz. The response of the resonator due to the $Ba_xSr_{1-x}TiO_3$ overlay is shown in the same. From the figure it is seen that, due to the overlay the resonance frequency shifts towards lower frequency side with substantial increase in the peak transmittance.

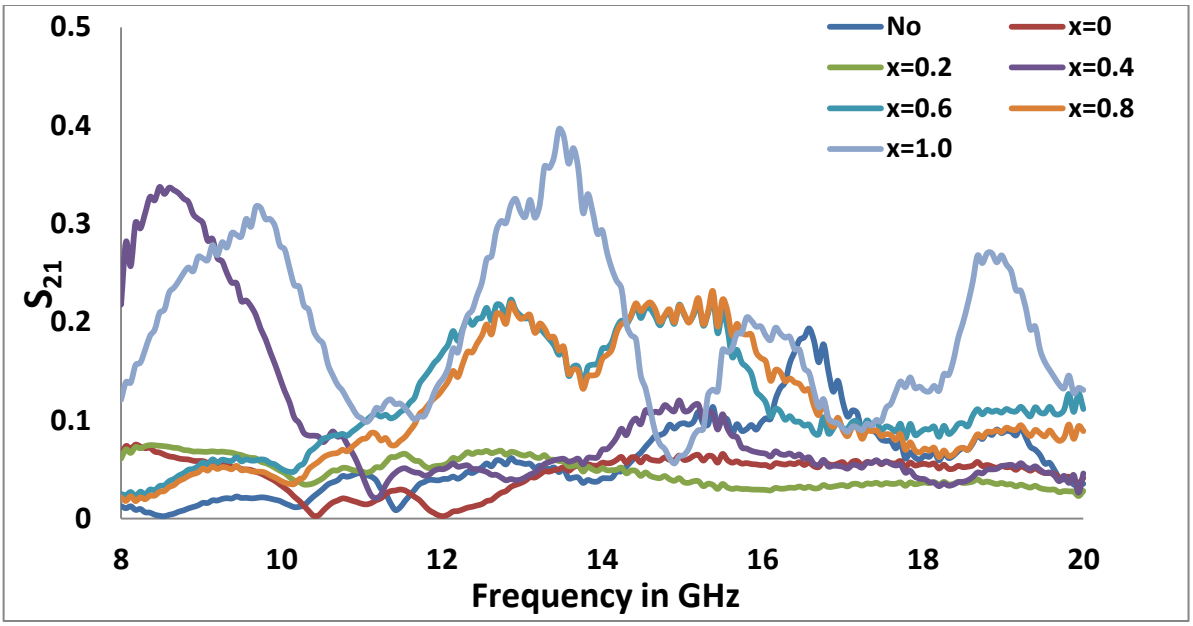

Figure 3.7: Variation of transmission coefficient (S_{21}) of microstrip straight resonator as a function of frequency, with overlay of $Ba_xSr_{1-x}TiO_3$

TABLE I: Resonance frequency and bandwidth of resonator for different overlays		
Composition of overlay	**Resonance frequency (GHz)**	**Bandwidth (GHz)**
No overlay	16.58	0.72
$SrTiO_3$	15.20	6.42
$Ba_{0.2}Sr_{0.8}TiO_3$	12.32	3.72
$Ba_{0.4}Sr_{0.6}TiO_3$	8.4.8	1.44
$Ba_{0.6}Sr_{0.4}TiO_3$	12.68	3.96
$Ba_{0.8}Sr_{0.2}TiO_3$	15.38	1.98
$BaTiO_3$	13.46	1.44

The plot of composition dependent effective dielectric constant of the multilayer structure for the $Ba_xSr_{1-x}TiO_3$ is shown in figure 3.8.

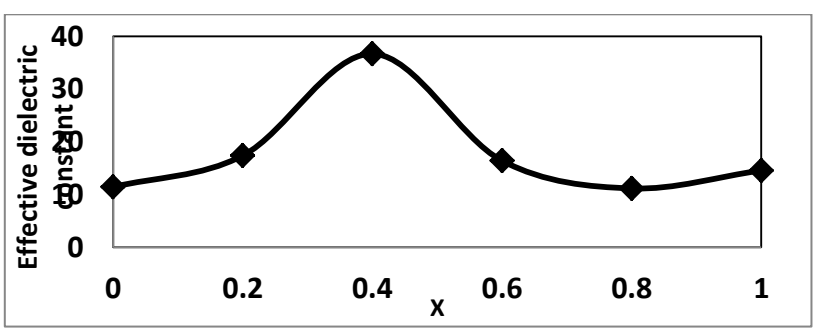

Figure.3.8: Variation of effective dielectric constant with composition of $Ba_xSr_{1-x}TiO_3$, of overlay.

3.4. Discussion:

When the microstrip component is covered by a dielectric material as superstrate or overlay. The overlay will also change the odd and even mode characteristics impedance. The Q value and resonance frequency of such a circuit is affected by perturbation [17, 18]. The perturbation can be brought about by use of overlay. The dielectric constant of the overlay changes the strength of the perturbation which translates into changes in resonance frequency and peak amplitude of the resonator. This cause changes in transmission (S_{21}) the fringing field of the microstrip components interacts with the dielectric, which results in the increased effective dielectric constant of the system, which in turn results in the changes in the characteristics of microstrip component.

The shift in resonance towards the lower frequency side is attributed to the increased effective dielectric constant of the multilayer structure. Due to the dielectric constant of the overlay material the fringing field lines get concentrated, thus increasing the fringing field capacitance which results in decrease in resonance frequency. The broadening of the resonance curve (increase in band width and decrease in Q value) is due to the lossey nature of the overly. The thick film microstrip straight resonator is an open circuit so, apart from dielectric losses, radiation losses are also present, the overlay might be suppressing the radiation loss, resulting in increased S_{21}.

The coupled circuit has odd and even mode propagation. The splitting of resonance curve is due to even and odd modes of coupling. The peak shifting toward lower frequency side is due to even mode of coupling and the peak shifting towards higher frequency side is due to odd mode coupling. The loss value of the odd mode is larger then the even mode. In the coupling sector, the odd mode fields are concentrated and this contributes to the conductor loss. The even mode contributes more to the frequency dependence of ε_{eff} then the odd mode. The broadening of resonance curve is due to lossy nature of the overlaid material.

The straight resonator has four open ends According to Delinger [20] the open ends of the line excite radiation modes. These ends act like discontinuities having reactive modes associated with it, due to which losses increase in the transmission data (S_{21}) of these resonators.

The splitting of resonance curve is due to even and odd modes of coupling. The peak shifting toward lower frequency side is due to even mode of coupling and the peak shifting towards higher frequency side is due to odd mode coupling. The loss value of the odd mode is larger than the even mode. In the coupling sector, the odd mode fields are concentrated and this contributes to the conductor loss. The loss value of the odd mode is larger than the even mode. In the coupling sector, the odd mode fields are concentrated and this contributes to the conductor loss. The even mode contributes more to the frequency dependence of ε_{eff} then the odd mode [21].

The plot of composition dependent effective dielectric constant of the multilayer structure for the $Ba_xSr_{1-x}TiO_3$ is shown in figure 4. From the plot it is clear that composition dependent variation in dielectric constant is observed. It is felt that the overlay technique can be used to predict the dielectric properties of ceramic in the pellet form, using thick film Microstripline components.

3.5. Conclusions

The Ag thick film microstrip straight resonator and ring resonator can be used to predict the dielectric constant of the $Ba_xSr_{1-x}TiO_3$. The composition dependent dielectric constant is observed. The overlay of $Ba_xSr_{1-x}TiO_3$ increases the peak transmission of the microstrip component. The broadening of the resonance peak and increase in the peak transmission is observed. The overlay splits the resonance peak.

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