1. Introduction

Barium titanate (BaTiO$_3$) has been of practical interest for more than 60 years because of its attractive properties. Firstly, because it is chemically and mechanically very stable, secondly, because it exhibits ferroelectric properties at and above room temperature, and finally because it can be easily prepared and used in the form of ceramic polycrystalline samples [1]. Due to its high dielectric constant and low loss characteristics, barium titanate has been used in applications. The properties of BaTiO$_3$ have been reported in a number of papers. Barium titanate is a member of a large group of compounds which is called the perovskite family. Ceramic materials with a perovskite structure are very significant electronic materials.

1.1. Ferroelectric materials in general

The phenomenon of ferroelectricity was discovered in single-crystal materials of Rochelle salt (sodium tartarate tetrahydrate, NaKC$_4$H$_4$O$_6 \cdot$ 4H$_2$O) in 1921. The two conditions necessary in a material to classify it as a ferroelectric are (1) the existence of spontaneous polarization and (2) a demonstrated reorienting of the polarization [2]. Spontaneously polarized regions, with a single direction of polarization, are called domains. Orientation relationships between domains are governed by the crystal symmetry. The most outstanding feature of a ferroelectric ceramic is its hysteresis loop (i.e. a plot of polarization versus electric field, P-E). Fig.1.1 illustrates a typical hysteresis loop.
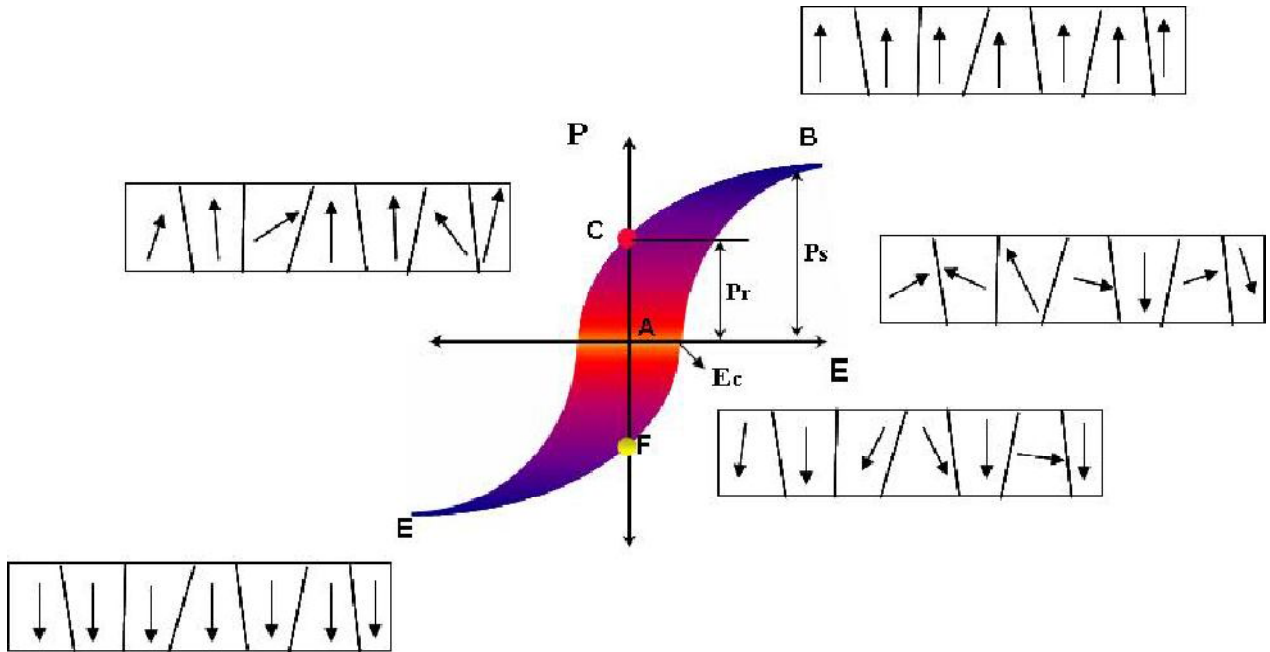
Fig.1.1. A typical P-E hysteresis loop in ferroelectrics [3]

When we apply an electric field, dipoles which are already oriented in the direction of the field will remain so aligned, but those which are oriented in the opposite direction will show a tendency to reverse their orientation, on the hysteresis loop that is a linear relationship between P and E and crystal behaves like a normal dielectric. When the electric field becomes sufficiently high to switch dipoles in crystallites, the polarization changes sharply and reaches saturation at higher fields. When the field is reduced to zero a net permanent polarization remains in the material known as remanent polarization, PR. Extrapolation of the linear part of the curve back of the polarization axis represents the value of the spontaneous polarization, PS. Reversing the electric field, polarization is reduced to zero and then changes sign as the field produced saturation polarization in the opposite direction,

completing the cycle of the hysteresis loop. The value of the field required to reduce P to zero is called the coercive field EC [4-5].

Ferroelectric $BaTiO_3$ has a Curie temperature of 120 ^{o}C, possesses a high dielectric constant at room temperature $\geq$ 1500 and low dielectric loss [6]. Because of these good characteristics barium titanate nowadays has become more and more important in ceramics materials.

1.2. Barium Titanate Structure

1.2.1. Perovskite structure

The perovskite structure is adopted by many oxides that have the chemical formula ABO_3. Barium titanate is a member of this perovskite family. This structure takes it name from the mineral perovskite, $CaTiO_3$. The general crystal structure is a primitive cube, with the A-larger cation in the corner, the B-smaller cation in the middle of the cube, and the anion, commonly oxygen, in the centre of the face edges, where A is a monovalent, divalent or trivalent metal and B a pentavalent, tetravalent or trivalent element, respectively. Geometrical packing of ions in the lattice is a very important factor determining the structure type. The perovskite structure can be considered as a three-dimensional frame work of BO_6 octahedra (Fig.1.2a), but it can also be regarded as a cubic close packed arrangement of A and O ions, with the B ions filling the interstitial positions (Fig.1.2b) [1,7]. The unit cell of the cubic perovskite type lattice is shown on Fig.1. 2.[8]. It can be detected that the coordination number of cation A is 12 and for cation B is 6.
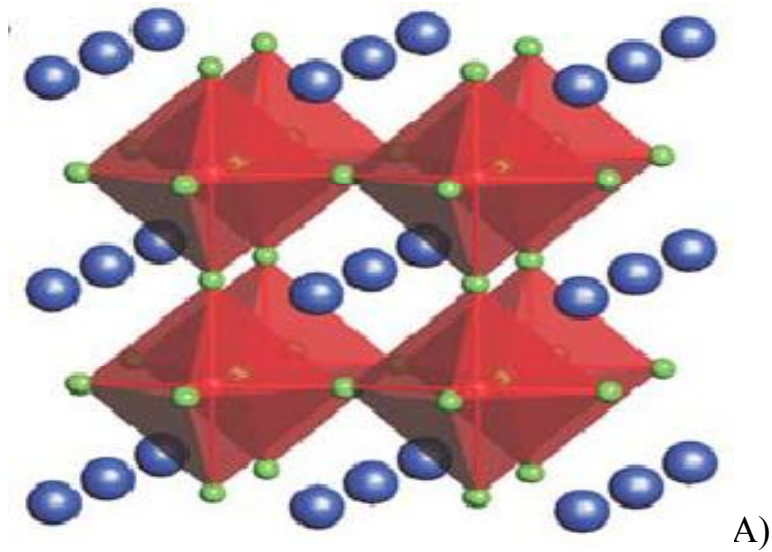
A)
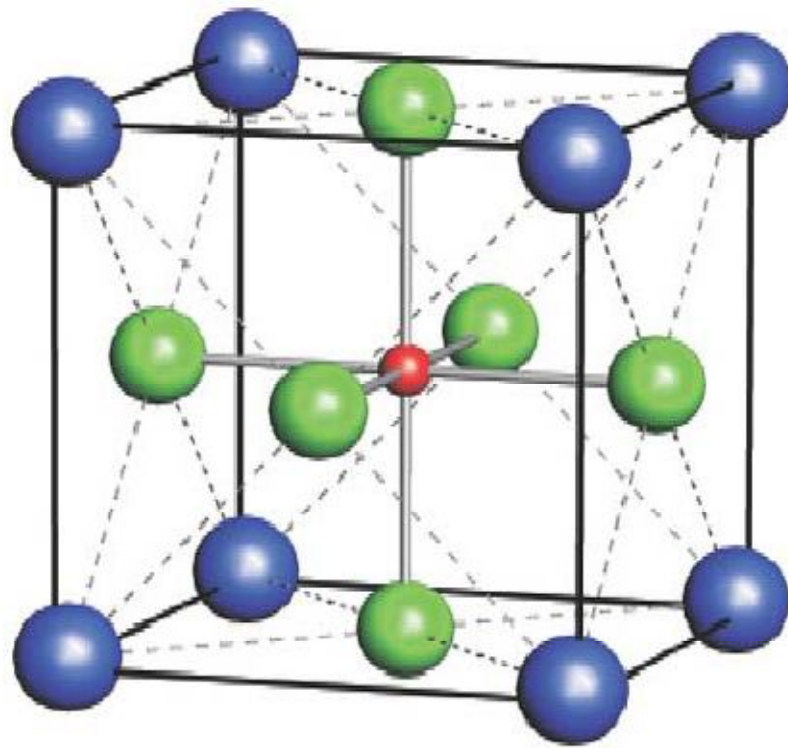
B)

Fig.1.2. Cubic perovskite-type structure ABO$_3$[8]

The perovskite family includes not only compounds with an ideal cubic perovskite lattice, but also all compounds with structures which can be derived

from the ideal one by way of small lattice distorsions or omission of some atoms. For example a large number of double fluorides, such as $KMgF_3$, $KZnF_3$ and others, also belong to the perovskite family [1].

1.2.2. Structural phase transitions in barium titanate

Barium titanate is the first discovered ferroelectric perovskite. Its ferroelectric properties are connected with a series of three structural phase transitions. The Curie point TC, of barium titanate is 120 ^{o}C. Above 120 ^{o}C the original cubic cell is stable up to 1460 ^{o}C. Above this temperature a hexagonal structure is stable [9]. When the temperature is below the Curie point, crystallographic changes in $BaTiO_3$ occur, first at about 120 ^{o}C a ferroelectric transition between the cubic, paraelectric and ferroelectric phase of tetragonal structure takes place. At 5 ^{o}C, the transition to a phase of the orthorhombic structure goes on and at -90 ^{o}C to the low temperature phase having a trigonal structure. [1,10]. Fig.1.3. illustrates crystallographic changes of $BaTiO_3$[1].

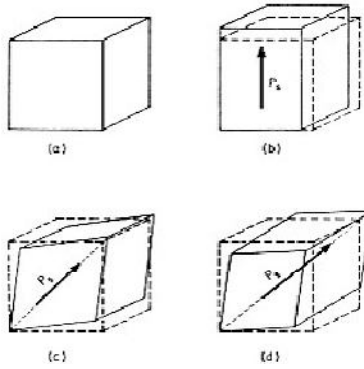**Fig.1.3. Unit cells of the four phases of BaTiO$_3$:**

(a)Cubic, stable above 120 ^{o}C.

(b)Tetragonal, stable between 120 ^{o}C and 5 ^{o}C.

(c)Orthorhombic, stable between 5 ^{o}C and -90 ^{o}C.

(d)Rhombohedral, stable below -90 ^{o}C.The dotted lines in (b), (c) and (d) delineate the original cubic cell[1]

At the Curie point Ti-ions are all in equilibrium positions in the center of their octahedra, but with a decrease of the temperature, Ti-ions jumps between energetically favorable positions out the of octahedron center, as shown in Fig.1.4[3].
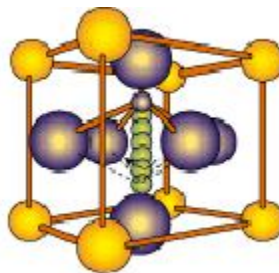

Fig.1.4. Ion positions in tetragonal BaTiO3 [3]

These changes can be related to structural distorsion, lengthening of the bonds or their shortening, so crystallographic dimensions of the barium titanate lattice change with temperature [10]. A lot of papers indicate that the mechanisms responsible for these phase transitions in $BaTiO_3$ are complex.
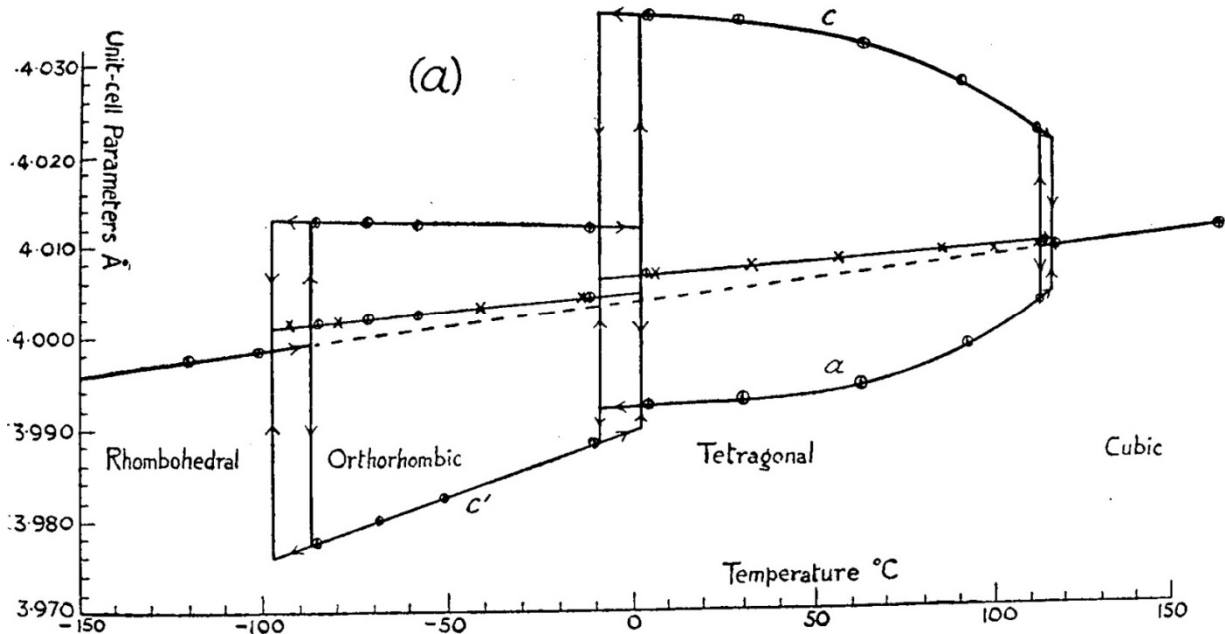
Fig.1.5. Lattice constants of BaTiO3 as function of temperature [1]

The temperature dependence of the lattice constants of $BaTiO_3$ in the four phases is depicted in Fig.1.5 [1].

1.3. Barium titanate synthesis methods

$BaTiO_3$ synthesis techniques were mentioned in a lot of papers. The selected method for barium titanate synthesis depends on cost, but even more important is the end application. The quality of the powders is not only influenced by the synthesis route but also by the starting materials used. As miniaturization of electronic devices continues to demand smaller particle size powders with controlled morphology, the desired characteristics of the starting powder become a critical issue [11]. The successful synthesis of barium titanate powder with their unique dielectric properties largely depends on the purity and crystal structure that greatly influences final properties [2,12]. All techniques have advantages for obtaining $BaTiO_3$ ceramics with required properties.

8

1.3.1. Conventional solid-state reaction

Traditionally, barium titanate is prepared by a solid-state reaction that involves ball milling of $BaCO_3$ or BaO and TiO_2. The mixture has to be calcined at high temperature. In some reports the needed calcination temperature was as high as 800 ^{o}C to 1200 ^{o}C [13-15], and in some other work it was 1300 ^{o}C [16]. Barium titanate powders prepared by a solid-state reaction are highly agglomerated, with a large particle size (2-5 μm) and high impurity contents due to their inherent problems such as high reaction temperature, heterogeneous solid phase reaction, which result in poor electrical properties of the sintered ceramics [17]. To eliminate these problems, many wet chemical synthesis routes are developed to generate high purity, homogeneous, reactive ultrafine barium titanate powders at low temperatures [14].

1.3.2. Chemical methods for barium titanate synthesis

Chemical synthesis has grown up through techniques such as sol – gel, coprecipitation, hydrothermal and polymeric precursor method. The advantage of chemical methods is the quasi – atomic dispersion of constituent components in a liquid precursor, which facilitates synthesis of the crystallized powder with submicron particles and high purity at low temperatures. The properties of the powder may vary as different preparation methods are used [9,18]. Li et al [19] described different synthesis routes for BaTiO3 preparation.

1.3.2.1. Sol-Gel method

Sol-gel is a method for preparing metal oxide glasses and ceramics by hydrolyzing a chemical precursor to form a sol and then a gel, which on drying (evaporation) and pyrolysis gives an amorphous oxide. Upon further heat treatment crystallization can be induced. There are two basic steps involved: (1) partial hydrolysis of metal alkoxide to form reactive monomers; (2) polycondenzation of

these monomers to form colloid-like oligomers (sol); (3) additional hydrolysis to promote polymerization and cross-linking leading to a 3-dimensional matrix (gel). As polymerization and cross-linking progress, the viscosity of the sol gradually increases until the sol-gel transition point, where viscosity abruptly increases and gelatin occurs.

Step 1 – Partial hydrolysis

$M(OR)_n + mH_2O$ --------... $M(OR)_{n-m}(OH)_m + ROH$

Step 2 – Condensation

$2M(OR)_{n-m}(OH)_m$ ------...$(OR)_{n-m}M-O-(OR)_{n-m} + HOH$

or

$(OR)_{n-1}M-(OR) + (HO)-M(OR)_{n-1}$ -------...$(OR)_{n-1}M-O-M(OR)_{n-1} + ROH$
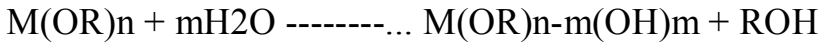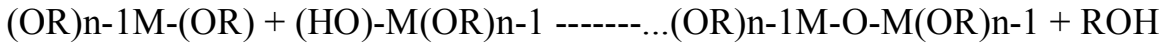
Step 3

3D cross-linking between $(OR)_{n-m}M-O-(OR)_{n-m}$ molecules

In the sol-gel technique, the structural and electrical properties of the final product are strongly dependent on the nature of the precursor solution, deposition conditions and the substrate [5].

Wang et al [17] used two typical wet-chemistry synthesis methods, stearic acid gel and acetic acid gel. In the first method, barium acetate, tetrabutyltitanate, isopropyl alcohol and glacial acetic acid were starting reagents. 0.1 mol of tetrabutyltitanate was dissolved into isopropyl alcohol at room temperature and then 0.3 mol of glacial acetic acid was added and stirred to form a titanylacylate compound. Next, 0.1 mol of barium acetate dissolved in an aqueous solution of acetic acid was dropped into the above compound gradually. The pH value was between 3.0-4.0

using glacial acetic acid. After stirring the mixture for 30 min., a transparent sol was obtained. Aging at 95 ^{o}C was performed until a gel formed, followed by drying at 120 ^{o}C for 12h in an oven, and finally, the dried gel was calcined in air at different temperatures to obtain BaTiO$_3$nanopowders. The average particle size of this powder was from 50-80 nm. For the second method also used by Wang et al [17] starting reagents were barium stearate, tetrabutyltitanate and stearic acid. An appropriate amount of stearic acid was first melted in a beaker at 73 ^{o}C, and then a fixed amount of barium stearate was added to the melted stearic acid and dissolved to form a yellow transparent solution. Next, stoichiometric tetrabutyltitanate was added to the solution, stirring to form a homogeneous brown sol, naturally cooling down to room temperature, and drying for 12h to obtain a gel. The gel was calcined at different temperatures in air to obtain nano-crystallites of BaTiO$_3$ with the size of particles from 25-50 nm. Li at al [19] described the oxalic acid precipitation method which is very similar to the sol-gel acetate method.

But acetic acid was replaced by the oxalic acid, molar ratio between oxalic acid and tetra butyl titanate was 2.2: 1; Ti solution was prepared by dissolving the tetra butyl titan ate into the solution of alcohol and oxalic acid. The particle size prepared by this method was 38.2 nm. Recently, a sol-precipitation process was developed, which is quite similar to the sol-gel method. The advantage of this route is that it requires no further thermal treatment of the product, such as calcination or annealing due to enhance the homogeneity of crystals and crystal growth. However, single-crystal $BaTiO_3$ nanoparticles can be directly obtained at the low temperature of 80 ^{o}C and a strong alkaline condition rather than amorphous gel that often formed in the standard sol-gel process. From TEM examination nanoparticles with an average diameter of about 20 nm were detected [20].

1.3.2.2. Hydrothermal Method

The hydrothermal method is attractive for synthesizing $BaTiO_3$ powder, because the combined effects of solvent, temperature and pressure on the ionic reaction equilibrium can stabilize desirable products while inhibiting formation of undesirable compounds. Hydrothermal synthesis also makes it possible to prepare $BaTiO_3$ powder in a single processing step and does not require elaborate apparatus or expensive reagents [17].

Ciftci et al [21] synthesized $BaTiO_3$ powder by the hydrothermal method at temperatures between ~100-200 ^{o}C by reacting fine TiO_2 particles with a strongly alkaline solution (pH>12) of $Ba(OH)_2$. $TiCl_4$, titanium alkoxide and TiO_2 gels were used as titanium sources at reaction temperatures in the range of 100-400 ^{o}C. Hydrothermal $BaTiO_3$ powders have a fine particle size in the range of 50-400 nm and narrow distribution of sizes making these powders

highly sinterable as well as attractive for the production of thin dielectric layers. Boulos et al [13] synthesized $BaTiO_3$ powders by the hydrothermal method using two different titanium sources $TiCl_3$ and TiO_2. The barium source was $BaCl_2 \cdot 2H_2O$. Synthesis was performed at two temperatures, namely 150 ^{o}C and 250 ^{o}C. SEM micrographs of barium titanate powders show spherical highly crystallized elementary grains with sizes in the range 40-70 nm for samples prepared from $TiCl_3$ at 150 ^{o}C and 80-120 nm at 250 ^{o}C. The average particle size for powders obtained from TiO_2 at 150 ^{o}C or 250 ^{o}C was 40-70 nm.

Recently, a microwave-hydrothermal method was developed and has shown some advantages over the conventional hydrothermal route [22]. Some of the primary reasons for the growing interest in using microwaves for ceramic material synthesis are (1) cost savings due to rapid kinetics (time and energy), (2) rapid intrnal heating and (3) synthesis of new materials [23].

Liu at al [23] attempted to prepare $BaTiO_3$ by the microwave method and evaluate its sinterability, microstructure and dielectric properties in comparison to samples prepared by conventional hydrothermal processing. Starting reagents were $Ba(NO_3)_2$, $TiCl_4$ and KOH. All chemical reactions were conducted in a microwave digestion system. The system is controlled by pressure and the parameters which were varied include pressure (and thus, temperature) and time. Conventional hydrothermal experiments were conducted in teflon-lined par bombs. After synthesis, the solid and solution phases were separated by centrifugation and the solid phases were dried. SEM analysis of powders prepared by both hydrothermal methods yielded a particle size of about 0.2 µm. Thus cubic $BaTiO_3$ powders can be prepared by the microwave method for 0.25 h, but in the case of conventional hydrothermal

route for 2.5 h. Guo et al. [24] also compared these two methods in their work. They detected that the microwave hydrothermal method can obtain the desired BaTiO$_3$ powder in a shorter time and at a lower temperature (30 min, 80 ^{o}C) than the conventional hydrothermal method (5h, 150 ^{o}C).

1.3.2.3. Coprecipitation method

The coprecipitation process is a widely studied technique [14,15,25]. This is a simple and convenient method for achieving chemical homogeneity through mixing of constituent ions on the molecular level under controlled conditions. In the case of coprecipitation by the oxalate route, it is difficult to achieve optimal conditions where precipitation of both Ba and Ti cations occurs simultaneously. This is because titanium is precipitated as titanyl oxalate at pH$\leq$ 2 in the presence of alcohol, and barium precipitation as BaC$_2$O$_4$ needs pH$\geq$ 4. So, in the pH range 2-4 titanium forms soluble anionic species like TiO(C$_2$O$_4$)22- affecting the stoichiometry (Ba:Ti ratio) during simultaneous precipitation. Coprecipitation of barium and titanium in the form of individual oxalates has been rarely

attempted [14]. It is reported to be an innovative way of maneuvering of the chemical conditions such as pH [25], reagent concentration, reaction medium, chelating properties of oxalic acid, complexation with metal ions and their stability, which make it possible to coprecipitate simultaneously and stoichiometrically Ba and Ti in the form of oxalates.

The effect of pH, in the range 2.0-10.0, on the formation of a stoichiometric coprecipitated precursor for BaTiO$_3$ from potassium titanyl oxalate and barium chloride was investigated by Prasadarao et al[25].

1.3.2.4. Polymeric precursor method

The 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 resin. The metal ions can be immobilized in a rigid polyester network, and no segregation of cations was observed during thermal decomposition of the organic material [18]. Scheme.1.1 shows the procedure of BaTiO$_3$ synthesis based on the Pechini method [26].

The Pechini process for obtaining barium titanate (BaTiO$_3$) powder is described below. A titanium citrate solution was prepared by dissolving titanium tetra isopropoxide (Ti[OCH(CH$_3$)$_3$]$_2$)$_4$ in a solution of ethylene glycol (HOCH$_2$CH$_2$OH). This solution was heated at T>60°C with constant stirring for 10 min. Afterwards, the solution of citric acid (HOOCCH$_4$C(OH)(COOH)CH$_2$COOH) was added. The solution of titanium citrate was mixed and heated at 90 ^{o}C. Simultaneously, a barium citrate solution was prepared by dissolving barium acetate in a citric acid solution. This solution was heated at 90 ^{o}C and when it becomes transparent ethylene glycol was added. The molar ratio of citric acid to ethylene glycol was 1:4, for both citrate solutions. The temperature was raised up to 120 - 140 ^{o}C, when the solution becomes solidified into a dark – brown glassy resin. Decomposition of most of the organic part was performed in an oven. The temperature regime was 200 ^{o}C for 1h and then at 300 ^{o}C for 4h, with constant stirring. When the resin incinerated, and became a black solid mass, then the material was pulverized. Calcinations were performed at 500 ^{o}C for 4h, 700 ^{o}C for 3h and 750 ^{o}C for 2h. The heating rate was 2 ^{o}C/min. The agglomerates were smashed by milling. After drying at room temperature and querying
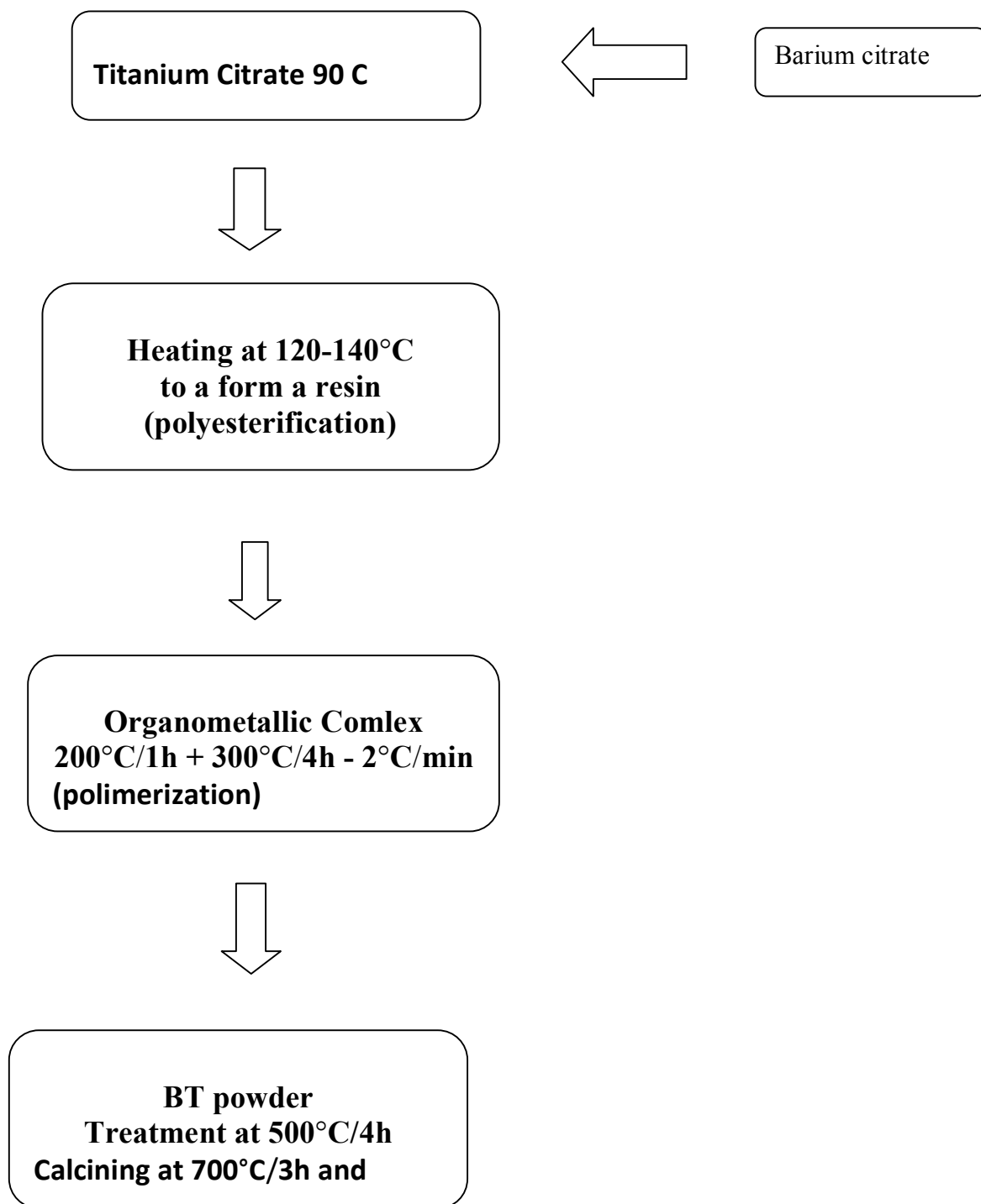
cubic barium titanate powder was obtained. The flow chart for the Pechini process of $BaTiO_3$ is shown in scheme.1.2[26].
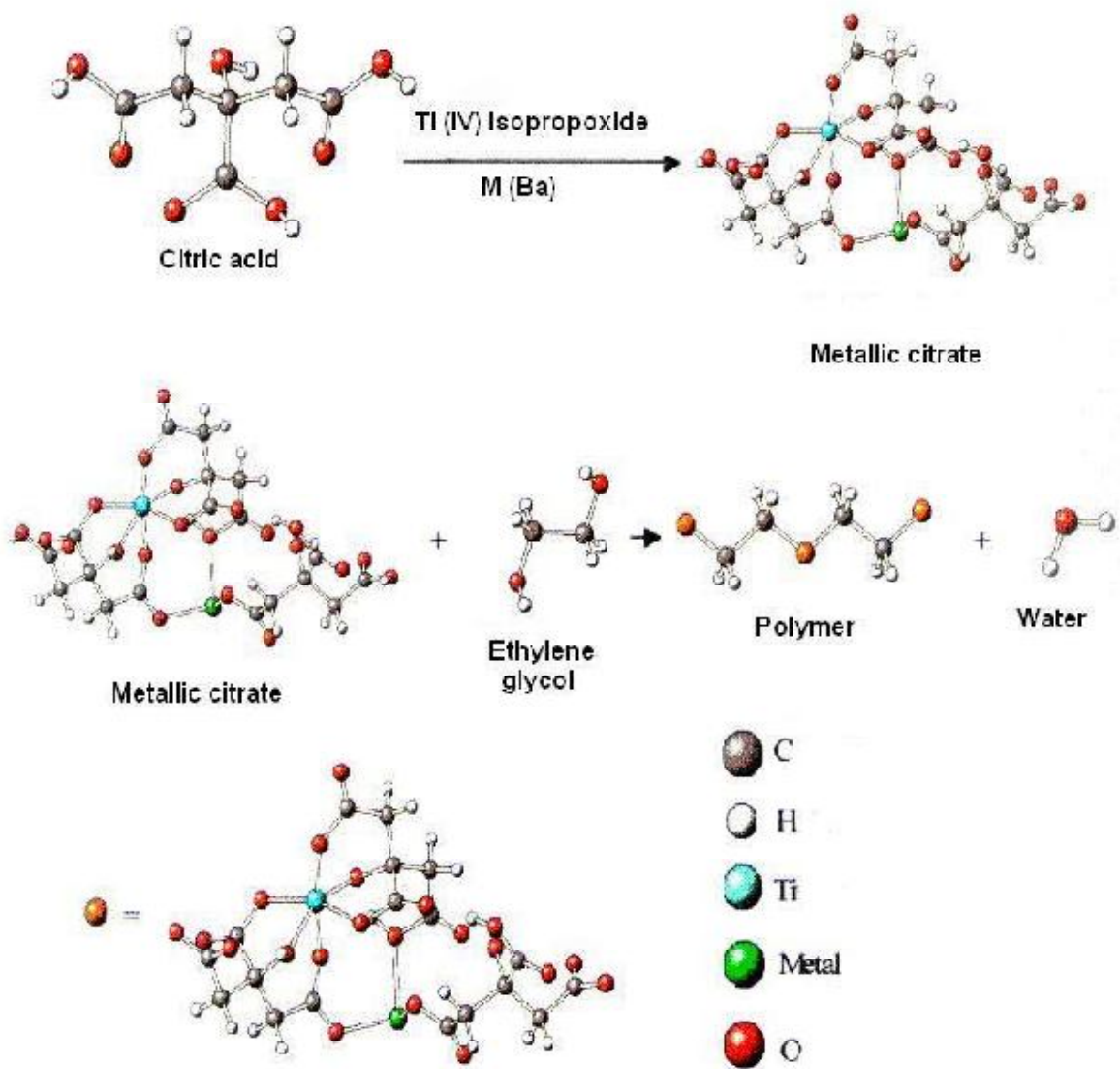
Scheme1.1 Polymeric precursor method-Pechini process [26]

Cho et al. [18] prepared barium titanate by heat-treating polymeric precursors containing barium and titanium in air at 600 °C for 8h. The experimental procedure used is very similar to the one described above. They obtained cubic barium titanate powder with particles of about 20 nm. An increase in the heat-treatment temperature to 900 °C generates grain growth, resulting in $BaTiO_3$ particles several hundreds of nanometers in size. Vinothini et al.

[12]also prepared barium titanate by the Pechini type reaction route and they obtained very similar results.

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┌─────────────────────────────┐        ┌──────────────────┐
│   Titanium Citrate 90 C     │ ⇐════  │  Barium citrate  │
└─────────────────────────────┘        └──────────────────┘
              ⇓
┌─────────────────────────────┐
│   Heating at 120-140°C      │
│   to a form a resin         │
│   (polyesterification)      │
└─────────────────────────────┘
              ⇓
┌─────────────────────────────┐
│   Organometallic Comlex     │
│ 200°C/1h + 300°C/4h - 2°C/min │
│   (polimerization)          │
└─────────────────────────────┘
              ⇓
┌─────────────────────────────┐
│   BT powder                 │
│   Treatment at 500°C/4h     │
│   Calcining at 700°C/3h and │
└─────────────────────────────┘
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Scheme1.2 The flow chart for the Pechini process of BaTiO$_3$ [26]

When coprecipitation and hydrothermal methods were used, stoichiometric deviations and formation of biphasic solid solution were reported. Also the sol-gel method and metallo-organic decomposition require a high temperature for achieving a complete solid solution. The advantage of the polymeric precursor method is based on the fact of its simplicity for obtaining powders of high purity and possibility to hold the initial stoichiometry. This method is not the best choice for industry due to long duration of the preparation process and high amounts of organic materials.

1.3.3. Mechanochemical synthesis

Mechanical activation is a very effective method for obtaining a highly dispersed system as due to mechanical action stress fields form in solids during the milling procedure [27]. During milling, heat is released, new surfaces and different crystal lattice defects are formed and a solid-state reaction is initiated. The accumulated deformation energy is the key of understanding the route of irreversible changes of the crystal structure and consequently microstructure, causing changes of material properties [27-29].

Mechanochemical synthesis of barium titanate has been the subject of research of many authors. Stojanovic et al [29] prepared $BaTiO_3$ starting from barium oxide (BaO) obtained from thermally treated $BaCO_3$, according to the following reaction $BaCO_3 \rightarrow BaO + CO_2$ in air at 900 ^oC/4h, and titanium oxide (TiO_2) in a rutile crystal form. Mechanochemical activation, i.e. mechanochemical synthesis was performed in a planetary ball mill in an air atmosphere for up to 4h, using a zirconium oxide vial and zirconium oxide balls as the milling medium. They identified strong agglomeration of powders after milling for 4h. $BaTiO_3$ powder consisted of particle agglomerates of

varying size and morphology, with grains of a mostly rounded shape in the range of 20-50 nm.

Mechanical treatment of ceramic powders can reduce particle size and make it possible to obtain nano-structured powders, which are very important for preparing nano-sized oxides and

compounds, superconductors, ferroelectric powders etc. [5] This method can be very useful for industrial application.

1.4. Titanium dioxide

Titanium dioxide (TiO_2) is an inorganic solid with a number of excellent physicalproperties, which make it the principal white pigment of commerce. TiO_2 isbecoming increasingly dominant over rival white pigments such as white lead,lithopone and zinc white [30]. Its predominance is mainly attributable to threeimportant physical characteristics, which are a high refractive index, lack ofcolour and chemical inertness.

1.5. Barium carbonate

Barium carbonate is a white solid, does not dissolve in water, it reacts with acids to make barium salts.

It's widely used in the ceramics industry as an ingredient in glazes. It acts as a flux ,a matting and crystallizing agent .

1.6. Applications of Barium Titanate

Barium titanate (BaTiO3) is a very attractive material in the field of electroceramics and microelectronics due to its good characteristics. Its high dielectric constant and low loss characteristics make barium titanate an

excellent choice for many applications, such as capacitors, multilayer capacitors (MLCs) and energy storage devices. Doped barium titanate has found wide application in semiconductors, positive temperature coefficient resistors, ultrasonic transducers, piezoelectric devices, and has become one of the most important ferroelectric ceramics.

1.6.1. Multilayer ceramic capacitors (MLCs)

The largest class of ceramic capacitors produced, in numbers and in value, is the multilayer type. In its simplest form, a capacitor consists of a pair of parallel metal plates separated by free space. When a voltage is applied across the plates, a charge is developed on them that are proportional to the applied voltage. If an insulating material is placed between the plates, the charge on the plates increases by the relative dielectric constant. High values of dielectric constant make $BaTiO_3$ ceramic a popular choice for use in capacitors. The first multilayer capacitors made from polycrystalline $BaTiO_3$ ceramics were produced in the early 1950s. A multilayer ceramic capacitor is depicted on Fig. 1.6.[31].
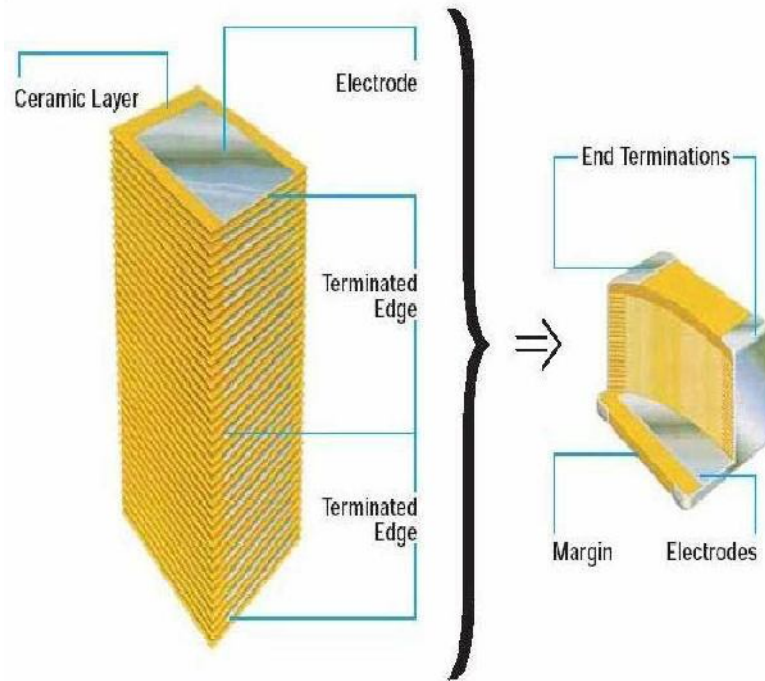
Fig.1.6. Multilayer ceramic capacitor[31]

Due to growing requirements for miniaturization, the multilayer capacitor structure enables the maximum capacitance available from a thin dielectric to be packed into the minimum space in amechanically robust form [32]. Multilayer ceramic capacitors possessing high capacitance of 1-100 μF can be engineered into passive components in circuits for LSI, replacing the widely used tantalum capacitors and aluminum electrolytic capacitors. Recently, nickel (Ni) and copper (Cu) internal electrodes have been increasingly produced to replace the expensive Ag-Pd electrodes, due to demand of cost reduction. The so-called base metal-electrode process requires a nonreducible $BaTiO_3$ dielectric that can be fired in a reducing atmosphere to prevent the electrodes from oxidation. In recent years, MLCs with Ni internal electrodes, which are composed of 500 or more laminated thin dielectric layers of ~2 μm, have been produced [33].

1.6.2. Positive temperature coefficient (PTC) thermistors

Elements based on materials with a positive temperature coefficient of resistance (PTCR) have found wide application practically in all branches of engineering [34]. The positive temperature coefficient of resistance (PTCR) effect was found in doped semiconducting barium titanate and

it is a grain boundary controlled phenomenon. Barium titanate is normally an insulator but after doping with trivalent donors (e.g. La, Sb, Y) that substitute for Ba^{2+} or with pentavalent donors (e.g. Nb, Ta) that substitute for Ti^{4+}, $BaTiO_3$ becomes semiconductive [32]. It has been established that the PTCR effect is a grain boundary resistance effect, even at temperatures well below the Curie point. The grain-boundary model given by Heywang treats the grain boundary as a n-type Shottky barrier with deep acceptor states at grain boundaries. The resistance anomaly behavior of doped $BaTiO_3$ is shown in Fig.1.7[35].
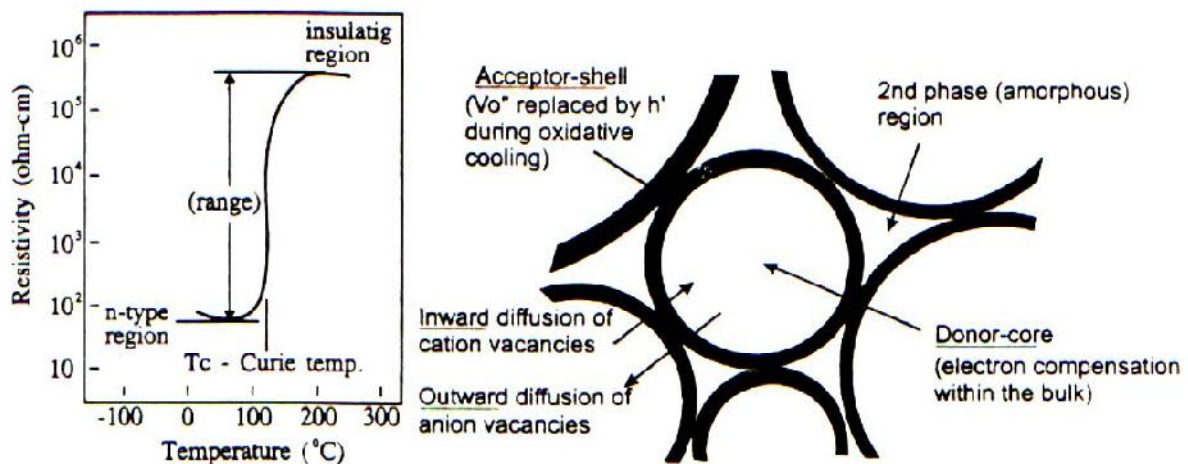

Fig.1.7. Electrical resistivity for typical PTCR device and schematic presentation of defect chemistry responsible for PTCR effect [35]

These PTC materials prepared from doped semiconducting $BaTiO_3$ ceramics can be used in various kinds of electronic circuitry as a switching device or as a constant temperature heater. Other important application of a PTC thermistor is the measurement/detection/control of temperature or parameters related to temperature. These PTC materials are known to have the highest temperature coefficient of resistance among all sensor materials available [36].
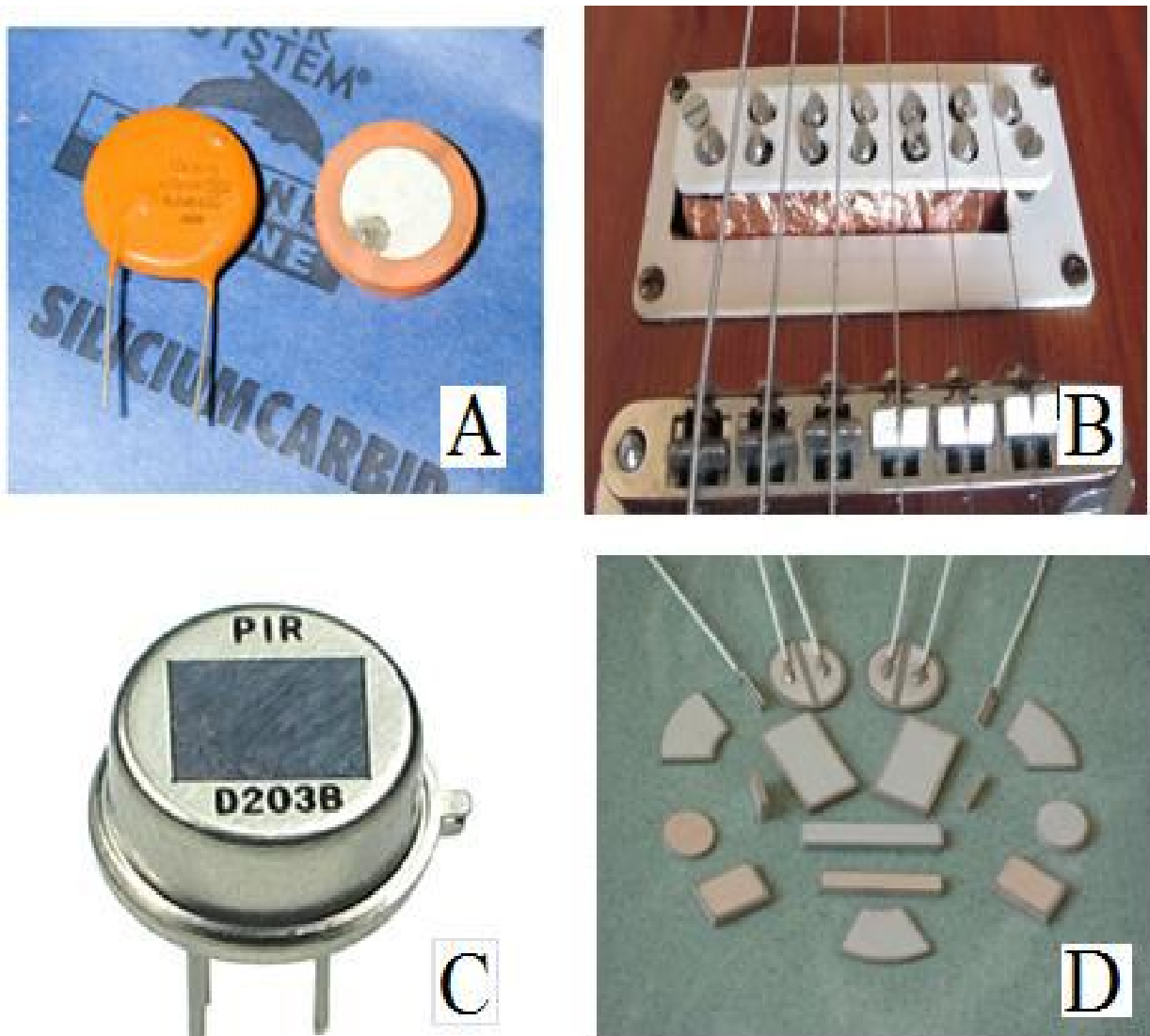

Figure 1.8. A. Ferroelectric capacitor [37] B. Piezoelectric electronic guitar pick up [38],C.Passive infrared sensor[39] and D. PTC elements[40].

1.7.Objectives

To preparation and characterization of barium titanate

2. Experiments and Instruments

2.1. Chemicals

-Barium carbonate(Central Drug House India, 98% purity)

-Titanium dioxide(Central Drug House India, 99%purity)

2.2. Instruments

-Mortar

-Sensitive balance

-Furnace(Serial No 20-803495)

-X-ray diffractometer(2-8DOC-M88-E02054)

-Infraredspectrometry (A21014200950-LP)

2.3. Method

Barium titanate was synthesized from commercially available barium carbonate(98%) and titanium dioxide (99%) ,0.99g of $BaCO_3$ and0.4 g of TiO_2 powder were milled in a mortar for 2 hours, the powder mixture was pressed in the form of disc. The pressed mixture wascalcinatedin furnace for 2hours at $600\,^{o}C$.

The above step was repeated at800 ^{o}C and 900 ^{o}C.All experiments were done in air.

X-ray diffraction and Infrared spectroscopic techniques were used for analysis.

3. Results and Discussion

3.1. Results

3.1.1. Infrared Spectrum:

table 3.1: Main peaks of IR spectra for $BaTiO_3$ at 900^oC

1043.42 cm^{-1}	862.12 cm^{-1}	588.25 cm^{-1}

Table 3.1 IR results
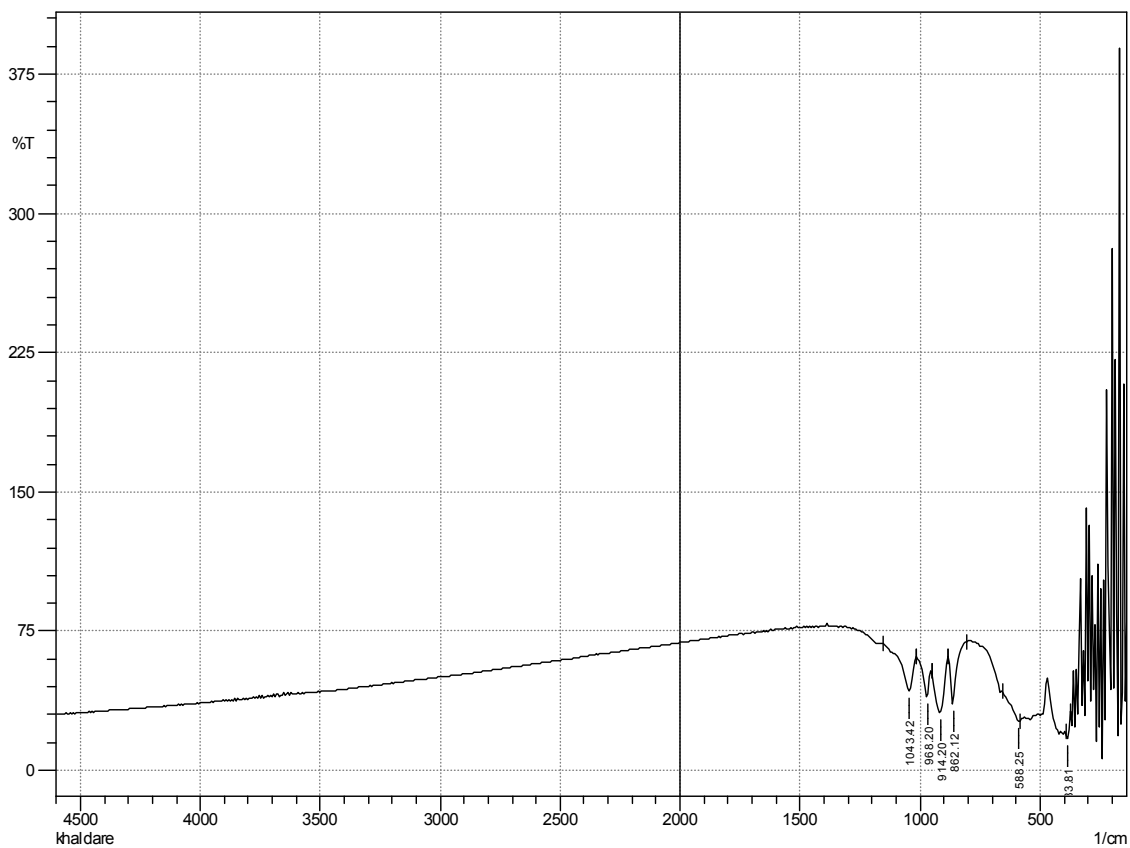
Fig 3.1. IR spectra for $BaTiO_3$ at $900\ ^oC$

3.1.2X-ray Diffraction
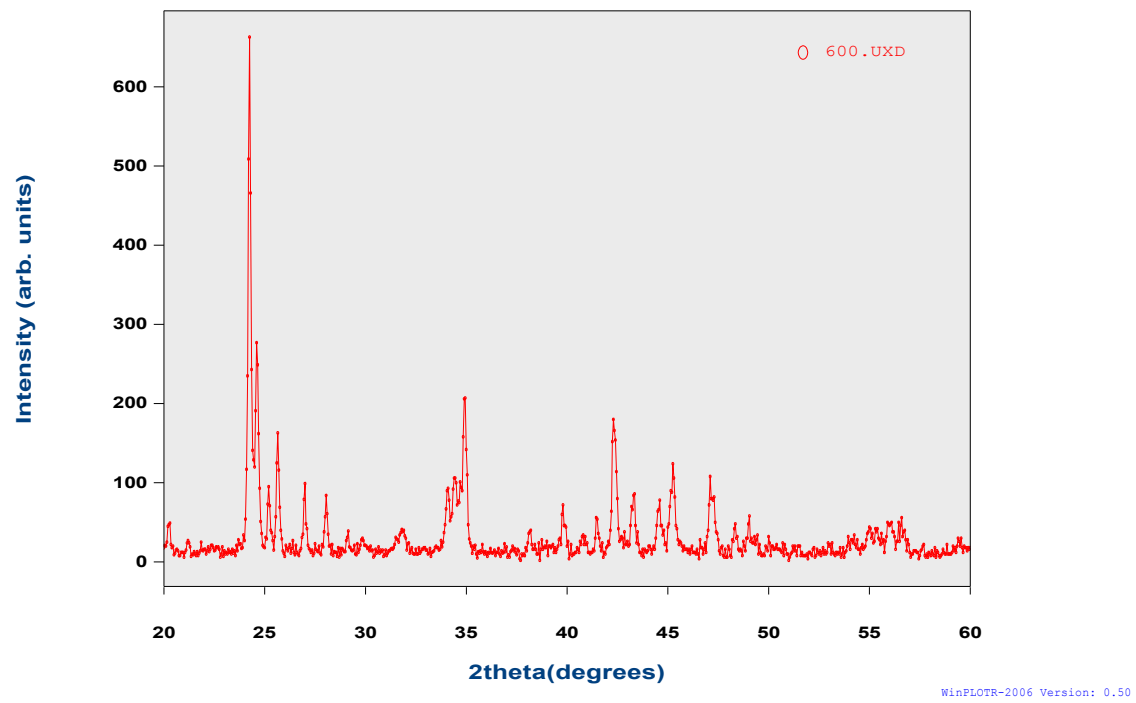
Fig 3.2. XRD spectrum of BaTiO$_3$ at 600 ^{o}C
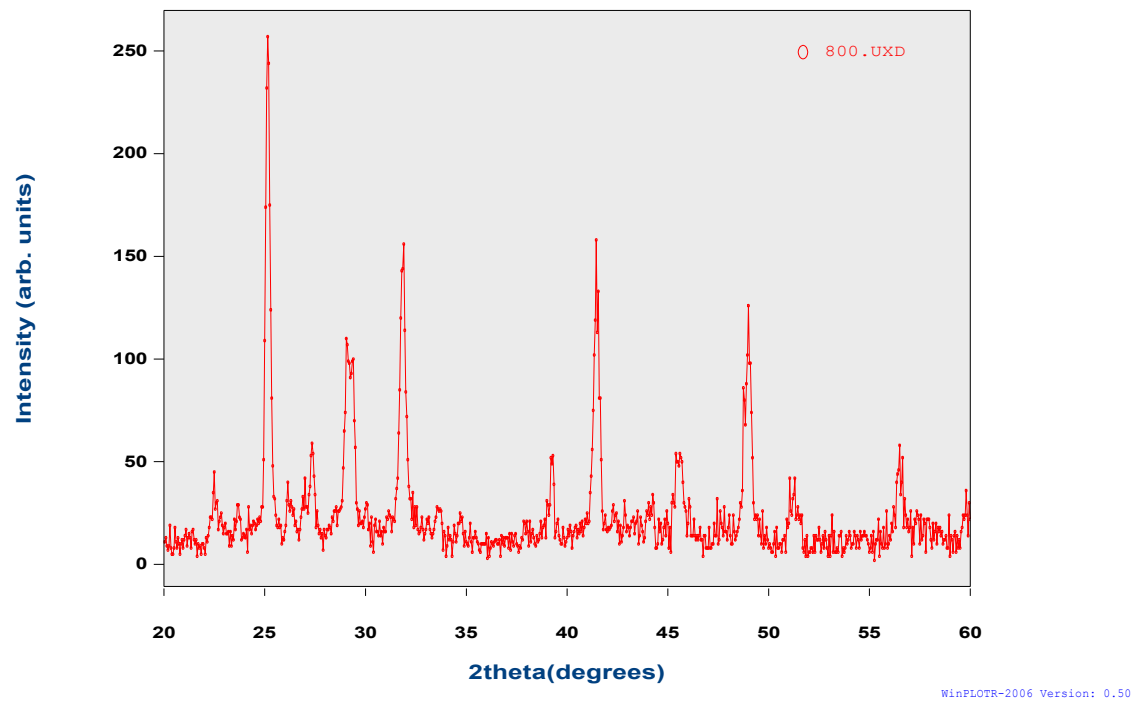
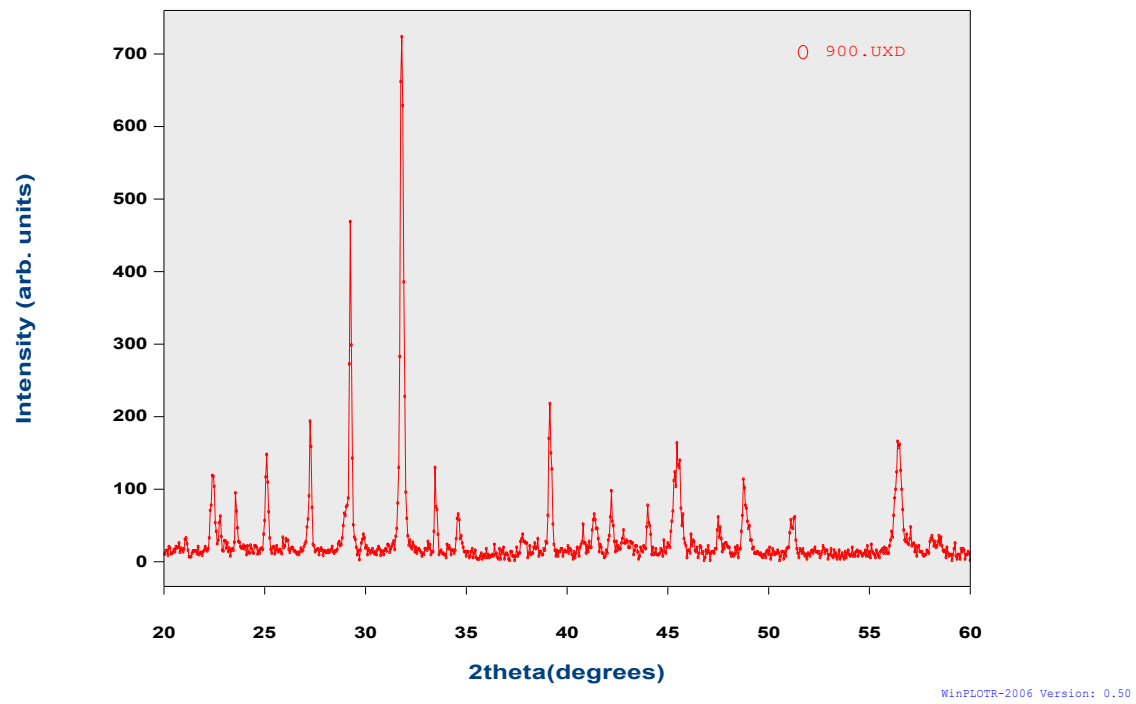**Fig 3.3 XRD spectrum of BaTiO$_3$ at 800°C**

Fig 3.4 XRD spectrum of BaTiO$_3$ at 900 ^{o}C

3.2.Discussion:

3.2.1.IR spectrum

Fromfigure 3.1Peak appears at1043.42 cm^{-1} may be due to C-O bond, peak at 862.12cm^{-1} is due to metal-oxygen ion streatching vibration and peak at 588.25 cm^{-1} is due to pure tetragonal phase.

3.2.2.XRD spectrum

Figures (3.2_3.4) show the XRD results of three different powder mixtures calcinated at 600°**C**, 800°**C** and 900°**C**, after calcination of powder mixtures at 600°**C** the XRD shows peaks , and these peaks may be due to $BaCO_3$ and TiO_2 precursors, in addition to apperance of small peak around 31 which due to traces of $BaTiO_3$ in the mixture. With the increase in calcination temperature, the amount of $BaTiO_3$ phase increases, and sothe intensity of barium titanate increase with increasing temperature.

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